CONFERENCE ON DISARMAMENT

CHEMICAL WEAPONS

WORKING PAPERS OF THE

Ad Hoc COMMITTEE ON CHEMICAL WEAPONS 1991

CD\CW\WP

COMPILED AND EDITED BY:

ARMS CONTROL AND DISARMAMENT DIVISION OF

EXTERNAL AFFAIRS AND INTERNATIONAL TRADE CANADA

OTTAWA, CANADA

FEBRUARY 1992

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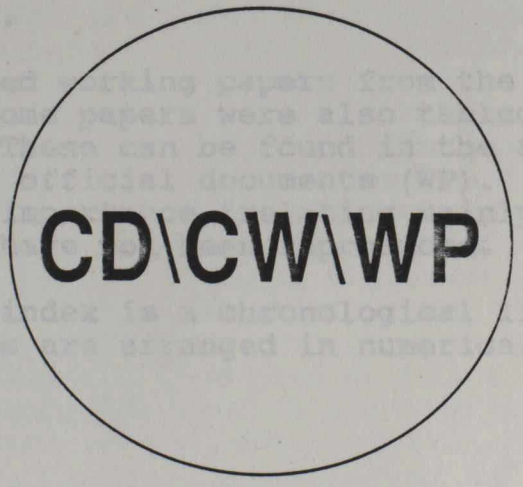

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PREFACE

CD/CW/WP

This volume covers working papers tabled in the Ad Hoc Committee on Chemical Weapons (AHCCW) during its 1991 sessions from 28 January 1991 to 23 August 1991. Also included are working papers from the intersessional meetings of the AHCCW which took place from October 1991 to December 1991. The volume is compiled to facilitate discussions and research on the issue of Chemical Weapons.

Not all numbered working papers from the AHCCW have been reproduced here. Some papers were also tabled in plenary and given a CD/number. These can be found in the appropriate annual volumes for plenary official documents (WP). Other papers were of such transitory importance (relating mainly to procedural matters) that they have not been reproduced.

Note that the index is a chronological listing while the documents themselves are arranged in numerical order by CD/CW/WP number.

Chemical Weapons Working Papers
Submitted to AHCCW of the CD 1991
Chronological Index

1991

Serial	Reference	Country	Description	Date
516	CD/1048	Czech and Slovak Federal Republic	Data relevant to the Chemical Weapons Convention (also issued as CD/CW/WP.326)	24.1.91
517	CD/1052	Canada/ Netherlands	Report on a joint chemical weapons trial challenge inspection (also issued as CD/CW/WP.327)	28.1.91
517.1	CD/CW/ WP.328	Netherlands	How to create an instrumental data base for verification?	28.1.91
519	CD/1055	Australia	Strategy for preparing for the implementation of the Chemical Weapons Convention in Australia (also issued as CD/CW/WP.329)	5.2.91
520	CD/1056 and Corr.1	Germany/UK	Report on two joint chemical weapons practice challenge inspections (also issued as CD/CW/WP.330 and Corr.1)	8.2.91
521	CD/1057	New Zealand	Report of a national trial inspection (also issued as CD/CW/WP.331)	13.2.91
523	CD/1061	Hungary	Provision of data relevantto the Chemical Weapons Convention (also issued as CD/CW/WP.332)	18.2.91

Serial	Reference	Country	Description	Date
523.1	CD/CW/ WP.333	AHCCW Chair-man	Working paper presented by the Chairman of the Ad Hoc Committee: "Organiz-ation of work for the 1991 session" (Not Reproduced)	20.2.91
524	CD/1062	Austria	Letter dated 19 February 1991 from the Permanent Representative of Austria addressed to the Secre-tary-General of the Con-ference on Disarmament transmitting three studies related to the verifica-tion of chemical weapons (also issued as CD/CW/WP.334)	21.2.91
525	CD/1063	France	Second trial request inspection (also issued as CD/CW/WP.335)	21.2.91
527	CD/1074	USA	A report on the destruc-tion of 3-Quinuclidinyl benzilate (BZ) (also issued as CD/CW/WP.336)	20.3.91
528	CD/1075	Peru	Working paper on chal-lenge inspection/ inspections on request (also issued as CD/CW/WP.337)	14.5.91
529	CD/1076	Austria	Letter dated 16 May 1991 from the Deputy Permanent Representative of Austria to the Secretary-General of the Conference on Dis-armament transmitting a study entitled "Detection of inhibitors of the enzyme acetylocholine esterase over long dis-tances using optic fibres" (also issued as CD/CW/WP.338)	21.5.91

Serial	Reference	Country	Description	Date
530.1	CD/CW/ WP.339	Belgium	National Registers and "Definition of Capable Facilities"	29.5.91
531	CD/1078	Norway	Letter dated 30 May 1991 from the Deputy Permanent Representative of Norway to the Deputy Secretary-General of the Conference on Disarmament transmitting a document entitled, "Verification of alleged use of chemical warfare agents: application of procedures after a simulated chemical attack on an air base" (also issued as CD/CW/WP.340)	30.5.91
533	CD/1080	UK	Verification of the Chemical Weapons Convention: practice challenge inspections at civil chemical plants (also issued as CD/CW/WP.341)	5.6.91
533.1	CD/CW/ WP.342	Finland, Netherlands	The network of laboratories under the Chemical Weapons Convention: possible structure and functions	6.6.91
533.2	CD/CW/ WP.343	AHCCW	Recommendation by the Ad Hoc Committee on Chemical Weapons (Not Reproduced)	12.6.91
534	CD/1082	Spain	Report on a national trial inspection in the civil chemical industry (also issued as CD/CW/WP.344)	12.6.91
537.1	CD/CW/ WP.345	USA	Analytical database discussion paper	25.6.91

Serial	Reference	Country	Description	Date
537.2	CD/CW/ WP.346	USA	Information processing for CW monitoring	25.6.91
537.3	CD/CW/ WP.347	USA	Quality assurance of verification analytical laboratories	25.6.91
537.4	CD/CW/ WP.348 and Corr.1	Egypt, Ethiopia, Indonesia, Iran, Kenya, Nigeria, Pakistan, Yugoslavia,	Verification of the chemical industry under Article Vi and its Annexes	27.6.91
538.1	CD/CW/ WP.349	AHCCW	Report of the Technical Group on Analytical Data Base and Laboratories	12.7.91
538.2	CD/CW WP.350	Australia, Canada, China, Czech and Slovak Federal Republic, Finland, France, Germany, India, Netherlands, Norway, Sweden, Switzerland, USSR, UK, USA	Second International Inter-laboratory Comparison (Round Robin) Test	12.7.91

Serial	Reference	Country	Description	Date
538.3	CD/CW/ WP.351	France	Report on a trial inspection to verify an industrial facility	15.7.91
538.4	CD/CW/ WP.352	Austra-lia, Japan, UK, USA	Recommended text for Article IV - challenge inspection	15.7.91
538.5	CD/CW/ WP.353	Austra-lia	On-site chemical analysis for verification of non-production of families of scheduled chemicals	15.7.91
540	CD/1093	Poland/ USSR	Joint report on a trial inspection on request (also issued as CD/CW/WP.354)	6.8.91
540.1	CD/CW/ WP.355	Nether-lands	The use of thermospray-liquid chromatography mass spectrometry for the veri-fication of chemical war-fare agents	6.8.91
540.2	CD/CW/ WP.356	USA	Challenge inspection pro-cedures for declared faci-lities	6.8.91
540.3	CD/CW/ WP.357	USA	Measures to ensure univer-sality	8.8.91
541.1	CD/CW/ WP.358	UK	Proposals for establish-ing thresholds in the Chemical Weapons Conven-tion: Schedule 2.B	13.8.91
542	CD/1100	USA	Report on the third United States trial inspection exercize (also issued as CD/CW/WP.359)	14.8.91
543	CD/1101	Germany	Report on a trial chal-lenge inspection at a large chemical plant site (also issued as CD/CW/WP.360)	15.8.91

Serial	Reference	Country	Description	Date
544	CD/1102	Germany	Report on an international trial challenge inspection (also issued as CD/CW/WP.361)	15.8.91
545.1	CD/CW/WP.362	Friend of the Chair on Technical Matters (Mr. Arend Meerburg)	Discussion Paper on Schedules and Guidelines	19.8.91
545.2	CD/CW/WP.363	AHCCW	Draft Report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament (Not Reproduced)	21.8.91
545.3	CD/CW/WP.364	USA	A Chemical Weapons Convention: staffing and cost estimates for a Technical Secretariat	21.8.91
545.4	CD/CW/WP.365	Romania	Information regarding Romanian export controls of precursors, equipment, plants or components thereof which could be used in the manufacturing of chemical weapons	23.8.91
546	CD/1107/Rev.1	USA	Report on the fourth United States trial inspection exercize (also issued as CD/CW/WP.366/Rev.1)	23.8.91
548.1	CD/CW/WP.367	USSR	Main technological aspects of the destruction of chemical weapons	7.10.91

Serial	Reference	Country	Description	Date
348.2	CD/CW/ WP.368	USSR	Environmental aspects of the destruction of chemical weapons	7.10.91
548.3	CD/CW/ WP.369	USSR	Complex for the destruction of faulty chemical munitions (KUASI)	8.10.91
549.1	CD/CW/ WP.370	Germany	Verification in chemical industry under Article VI	9.10.91
549.2	CD/CW/ WP.371	AHCCW	Article IX: Procedure for challenge inspections	11.10.91
549.3	CD/CW/ WP.372	Switzerland	Report on the second Swiss trial inspection	11.10.91
549.4	CD/CW/ WP.373	UK	Destruction of CW stocks, weapons and associated plant	21.10.91
549.5	CD/CW/ WP.374	Germany	"Old Chemical Weapons" disposal	31.10.91
549.6	CD/CW/ WP.375	Italy	Italian experience of the destruction of old and obsolete chemical weapons	20.11.91
550.1	CD/CW/ WP.376	Netherlands	Verification of alleged use of chemical warfare agents: retrospective immunochemical detection of exposure to mustard gas	6.12.91
550.2	CD/CW/ WP.377	Friend of the Chair on Technical Matters (P. Canonne)	Report of experts' meeting on the destruction of chemical weapons	9.12.91

Serial	Reference	Country	Description	Date
550.3	CD/CW/ WP.378	Finland, Nether-lands	Accreditation of Verifi-cation Laboratories	16.12.91

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The following documents of the AHCCW, which do not contain any substantive material or are draft reports, are not reproduced but are listed here for identification:

Serial	Reference	Country	Description	Date
523.1	CD/CW/ WP.333	AHCCW Chair-man	Working paper presented by the Chairman of the Ad Hoc Committee: "Organization of work for the 1991 session" (Not Reproduced)	20.2.91
533.2	CD/CW/ WP.343	AHCCW	Recommendation by the Ad Hoc Committee on Chemical Weapons (Not Reproduced)	12.6.91
545.2	CD/CW/ WP.363	AHCCW	Draft Report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament (Not Reproduced)	21.8.91

Czech and Slovak Federal Republic	CD/CW/WP.326	Data relevant to the Chemical Weapons Convention NOT REPRODUCED (see WP volume)	Also issued as CD/1048 24.1.91

* * *

Canada, Netherlands	CD/CW/WP.327	Report on a joint chemical weapons trial challenge inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1052 28.1.91

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CONFERENCE ON DISARMAMENT

CD/CW/WP.328
28 January 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

THE NETHERLANDS

HOW TO CREATE AN INSTRUMENTAL DATA BASE FOR VERIFICATION ?
Working Paper

1. Introduction

The necessity of creating a computerized data base containing information about chemical warfare (CW) agents and related compounds was expressed by the Technical Group on Instrumentation during their meetings in Geneva in 1989 and 1990 (1,2). In addition to more general information about the compiled compounds, this data base should contain the spectrometric and chromatographic data which may serve as references in order to identify unequivocally a compound of CW interest.
Basically, there are two ways to create such an instrumental data base :

- Existing data from various laboratories with experience in the field of identification of CW agents could be taken on, provided that the data meet certain quality requirements.

- The data could be recorded anew, using generally accepted operational procedures. In this case the compounds of interest have to be collected. The task of recording the data could be carried out by one or more accredited laboratories.

The Netherlands is in favour of the first option. The reasons for this choice will be presented in this working paper. Furthermore, some remarks will be made about which data should be collected as well as about the way the data should be distributed to laboratories operating under the CW Convention. Finally, as a possible contribution to an instrumental data base for verification, a survey of data digitally available in The Netherlands will be given.

2. The creation of an instrumental data base from existing data

The basic idea of creating an instrumental data base from new recorded spectrometric and chromatographic data according to standard operational procedures using high quality analytical equipment is attractive. It might appeal to a general human quality to make a fresh start every now and then. However, the following arguments may be raised against this approach :

- The workload will be enormous. This will especially be caused by collecting and preparing the compounds. It may take weeks to prepare a few compounds in a sufficiently pure form.

- The laboratory or laboratories providing the data bear(s) a great responsibility on the usability of these, whereas in the case of compiling existing data from various laboratories a comparison can be made. Possible differences can be discussed by experts, thus leading to more reliable or better applicable data.

- Spectrometric data depend on the instrumentation as well as on the physical state of the compounds and the operating procedures. For instance, it is accepted in mass spectrometry that, with the present state of the art, it is not feasible to obtain spectra that are independent of the instrumentation (3). A data base containing spectra from different sources will therefore be more realistic, especially if the accredited laboratories use different types of instruments.

- As spectrometric data depend on the instrumentation one may have to consider to start all over again whenever analytical equipment is replaced.

The major disadvantage of creating an instrumental data base consisting of data obtained from different laboratories seems to be the fact that it will lead to non-uniformity. For certain types of spectrometry this will certainly be the case, but the question is whether that really matters. If a spectrum varies essentially when it is obtained with different instruments, this spectrum can never be the basis for a sound identification. Moreover, identification has to be considered as a subject for specialists which should be capable to interpret differences occurring eventually between the recorded spectra and the ones in the data base. They should also be aware of the fact that blind acceptance of reference data may lead to misinterpretation as was recently demonstrated (4).

Thus far, only Finland has systematically presented its collected spectrometric and chromatographic reference data. A series of Blue Books (5) was published prior to the start of the Verification Data Base (6) which will include all Finish recorded data. The Netherlands has also collected reference data of compounds of CW interest. These data were obtained mainly from compounds which were prepared in the PML-TNO laboratory in Rijswijk. This collection started some twenty years ago, first in the form of printed spectra. After the introduction of spectrometric data systems the collection was converted into a computerized data base. Data can be made available in several formats. Special parts of the compiled mass spectral data were occasionally publised (7-10). Other nations have indicated that they possess useful data. For instance, D'Agostino and co-workers from the Canadian DRES laboratory in Suffield have published several papers containing gas chromatographic data (retention indices) as well as electron impact (EI) and chemical ionization (CI) mass spectra of CW agents and other compounds of CW interest (11-18).

If the states represented in the Technical Group on Instrumentation would agree with the Netherlands view to create an instrumental data base from existing data, an inquiry might be held in which the states indicate the kind of data they are willing to provide. The data base will then be constructed in more or less the same way as the reference data collection for mass spectra in the NIST/EPA/MSDC data base (19). The major concern of such a file of 40.000 mass spectra is the quality of the spectra it contains. It is almost impossible to check manually each spectrum in such a large compilation. Therefore each spectrum in this file was evaluated by calculating a set of quality factors using a computer programme (20,21). It is obvious that these quality factors can be calculated for the mass spectral data of compounds of CW interest. It should be investigated whether similar quality factors could be deduced for other spectrometric as well as the chromatographic techniques. The number of compounds in the instrumental data base for verification will be limited and possibly not exceed 500. A selected group of experts might form an editorial board and give their opinion on each entry.

3. Instrumental data to be collected

A survey of the instrumental data which should be incorporated in a data base is presented in a report of the Technical Group on Instrumentation (2). Although in principle each piece of information might have its value, some data are more important than others. The order of importance can be deduced from the criteria for confirmation of CW agents identification, which have been proposed by The Netherlands (22). The following order is proposed :

- Electron Impact (EI) mass spectra and infrared (IR) spectra (different phases). These types of spectra are frequently used in computer search systems where a direct comparison between the recorded spectrum and the library spectra is made.

- Gas chromatographic (GC) retention indices and nuclear magnetic resonance (NMR) shifts (31P,19F). These data are generally single numbers, which could easily be searched.

- 1H NMR (different field strengths, different solvents) and Chemical Ionization (CI) mass spectra (different reaction gases). These types of spectra are generally not used in computer search systems. NMR spectra are normally interpreted using chemical shift correlation tables and coupling constants. CI mass spectra depend strongly on the type of instrumentation and recording conditions making them less suitable than EI mass spectra for retrieval purposes.

- Standardized thin-layer chromatographic (TLC) RF values. These data are similar in nature as GC retention indices.

- Spectral data such as high resolution mass spectral data, collision activated dissociation (CAD) mass spectra and liquid chromatography-mass spectral data which are produced by instruments which are not generally available.

- 13C NMR data. Although very useful for structure elucidation the use of 13C NMR spectrometry is limited due to its low sensitivity.

- Spectral data which provide limited additional information, such as originating from ultraviolet (UV), Raman and near-infrared (NIR) spectrometry.

It is not advisable to create a compilation of standardized liquid chromatographic (LC) data in the same way as GC retention indices. Such a system could possibly be effective in one laboratory for a restricted class of compounds. However, due to the variations in column packings and eluents as well as the large variety in nature of the CW agents and their related compounds, a reliable retention index system for LC would be very difficult to achieve at the moment. Although TLC tends to become a forgotten technique, it is in some areas still the method of choice for the screening of polar compounds and could be considered as an alternative for LC data (23). By using coloured or UV-absorbing reference compounds the measured RF values can be converted into standardized data.

4. Distribution of the data

Creating an instrumental data base from existing data obtained from various sources can only be carried out by assembling the data in a central computer system, situated either in the Technical Secretariat or in its central laboratory. However, the best places to actually use the compiled data are the designated laboratories where the analyses are performed. Instead of creating a system such as the Chemical Information System (CIS) (24) where the data are only accessible by a computer network, the relevant verification data should rather be incorporated in the computers used by the laboratories. For this purpose the search/library facilities provided by instrument manufacturers could be used. Nowadays almost every spectrometer manufacturer offers a library search system as a part of their data systems, including a library obtained from NIST (mass spectra) or Sadtler (IR, NMR spectra). Relevant data of the compounds of CW interest from the central computer should be converted into the formats of the various data systems of the designated laboratories using information obtained from the manufacturers about the data format applied. Additional data should be supplied to these laboratories on a regular basis. Finally, despite all these computer systems chemical analysts would still like to have the data in a printed form as well. Therefore the data should also be distributed in the form of atlasses.

5. Data available in The Netherlands

The Netherlands is willing to provide a first set of data. Based on a number of recent publications (25-28) in which CW agents are reviewed, a list of 35 compounds (See Annex 1) has been compiled consisting of nerve agents, vesicants, suffocating agents, paralysants, a psychochemical and military irritants. Starting from these 35 CW agents the related compounds were also compiled in separate classes of compounds. These consist of decomposition products, precursors, impurities, artifacts as well as analytical derivatives. The deviation between the various classes of related compounds is not always clear-cut. For instance, a compound such as thiodiglycol is a decomposition product of mustard, but also its main precursor. Both decomposition products and precursors may be present in a chemical weapon as impurities. The class of precursors also contains additives and side products. A special class is formed by artifacts and analytical derivatives. Artifacts are formed by accident, when for instance the CW agent reacts with solvents (especially alcohols) during the extraction of the samples. Analytical derivatives of a number of less volatile polar compounds were prepared in order to be able to analyse with gas chromatography. Compounds with alcoholic hydroxy groups were derivatized with trifluoracetylation and trimethylsilylation reagents, whereas acids were converted into alkyl (methyl, occasionally ethyl or isopropyl) and dimethyl-t-butylsilyl esters (29).

In Annex 1 a survey of the data, which are available digitally at the moment, is presented. The list contains several gaps. However, an EI mass spectrum is available for almost all compounds. Some IR and NMR data, which were recorded on spectrometers not connected with a data system in the past, are difficult to digitise. Therefore these spectra only exist in a printed form. However, data are continuously updated in order to fill gaps and to replace older data.

6. References

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List of spectral data (available in digital form)

Explanation of the symbols:

M = digitised EI mass spectrum
I = digitised infrared spectrum
R = digitised raman spectrum
H = digitised 1H NMR spectrum
P = digitised 31P NMR spectrum
C = digitised 13C NMR spectrum
G = gas chromatographic data
T = thin-layer chromatographic data

Chemical warfare agents

Nerve agents

MIRHP	GT	isopropyl methylphosphonofluoridate (sarin)
MIR	GT	cyclohexyl methylphosphonofluoridate (cyclohexylsarin)
MIRHP	GT	1,2,2-trimethylpropyl methylphosphonofluoridate (soman)
MIRHP	GT	ethyl N,N-dimethylphosphoramidocyanidate (tabun)
MI HP	GT	ethyl S-2-diisopropylaminoethyl methylphosphonothioate (VX)

Vesicants

MIRH	CGT	2,2'-dichlorodiethyl sulphide (mustard)
MI	GT	1,2-bis(2-chloroethylthio)ethane (sesquimustard)
M RH	G	bis(2-(2-chloroethylthio)ethyl)ether (dimustard ether)
MIR	GT	tris(2-chloroethyl)amine (N-mustard, HN-1)
IR	GT	tris(2-chloroethyl)amine hydrochloride
MIR	GT	bis(2-chloroethyl)methylamine (N-mustard, HN-2)
I	GT	bis(2-chloroethyl)methylamine hydrochloride
MIR	GT	bis(2-chloroethyl)ethylamine (N-mustard, HN-3)
I	GT	bis(2-chloroethyl)ethylamine hydrochloride
MI	GT	2-chlorovinyldichloroarsine (lewisite I)
MIR	GT	bis(2-chlorovinyl)chloroarsine (lewisite II)
MI	GT	tris(2-chlorovinyl)arsine (lewisite III)
MIR		dichloroformaldoxime (phosgene oxime)

Suffocating agents

MI		carbonyl dichloride (phosgene)
MI		trichloromethyl chloroformate (diphosgene)
MI	G	trichloronitromethane (chloropicrin)

Paralysants

MI	G	hydrogen cyanide
MI		cyanogen chloride
MIR		cyanogen bromide

Psychochemicals

MI	GT	3-quinuclidinyl benzilate (BZ)

Irritants

MIR	GT	omega-chloroacetophenone (CN)
MIR	GT	alpha-bromobenzyl cyanide (CA)
MIRH	GT	2-chlorobenzylidenemalononitrile (CS)
MI	GT	dibenzoxazepine (CR)
MI	T	methyldichloroarsine
MI	T	ethyldichloroarsine
MI	T	phenyldichloroarsine
MI	T	10-chloro-5,10-dihydrophenarsazine (adamsite)
MI	T	diphenylchloroarsine
MI	T	diphenylcyanoarsine

Decomposition products

Decomposition products of sarin, cyclohexyl sarin and soman

MI HP T	isopropyl hydrogen methylphosphonate
MI	methylphosphonic acid
MI GT	diisopropyl dimethylpyrophosphonate
MI P	methylphosphonofluoridic acid
MIRH	methylphosphonic difluoride
MIR G	propanol-2
MI HP GT	diisopropyl methylphosphonate
MI	methylphosphonofluoridic anhydride
MI T	cyclohexyl hydrogen methylphosphonate
MI T	dicyclohexyl dimethylpyrophosphonate
MI T	dicyclohexyl methylphosphonate
MI G	cyclohexene
MI G	cyclohexanol
MI HPC T	hydrogen 1,2,2-trimethylpropyl methylphosphonate
MI T	bis(1,2,2-trimethylpropyl) dimethylpyrophosphonate
MIRHP GT	bis(1,2,2-trimethylpropyl) methylphosphonate
MI G	3,3-dimethylbutene
MI G	2,3-dimethylbutene
MI G	2,3-dimethyl-2-butene
MIRH CG	3,3-dimethyl-2-butanol (pinacolyl alcohol)

Decomposition products of tabun

M R	hydroxylamine hydrochloride
M	phosphoric acid
MIRH CG	ethanol
MIRHP	diethyl N,N-dimethylphosphoramidate
MIRHP	bis(dimethylamido) ethyl phosphate
M	N,N,N',N'-tetramethylphosphorodiamidocyanidate

Decomposition products of VX

MIR	ethyl hydrogen methylphosphonate
MI CG	diethyl methylphosphonate
MI H	diethyl dimethylpyrophosphonate
MIR G	2-diisopropylaminoethanethiol
MIRH GT	bis(2-diisopropylaminoethyl)sulphide
MIR G	bis(2-diisopropylaminoethyl)disulphide
MI	bis(2-diisopropylaminoethyl) methylphosphonodithioate
MI HP	hydrogen S-2-diisopropylaminoethyl methylphosphonothioate

Decomposition products of vesicants

M RH GT	2-chloro-2'-hydroxydiethyl sulphide
M RH CGT	2,2'-thiodiethanol (thiodiglycol)
MIRH	1,4-thioxane
MIRH	1,4-dithiane
M	vinyl-2-chloroethyl sulphide
M	divinyl sulphide
M H T	2,2'-dichlorodiethyl sulphoxide
MIRH T	2,2'-dichlorodiethyl sulphone
M RH	2,2'-dihydroxydiethyl sulphoxide
M RH	2,2'-dihydroxydiethyl sulphone
M	1,2-bis(2-hydroxyethylthio)ethane
MI H	bis(2-(2-hydroxyethylthio)ethyl)ether
M RH	tris(2-hydroxyethyl)amine
M H C	N,N,N',N'-tetrakis(2-chloroethyl)piperazinium dichloride
M P C	methyl-bis(2-hydroxyethyl)amine
M H C	N,N'-dimethyl-N,N'-bis(2-chloroethyl)piperazinium dichloride
	ethyl-bis(2-hydroxyethyl)amine
M H C	N,N'-diethyl-N,N'-bis(2-chloroethyl)piperazinium dichloride
AT T	2-chlorovinylarsine oxide

Decomposition products of paralysants

M H G	methanoic acid
MI	cyanogen
MI H	oxamide
M RH	cyanuric acid
MI G	cyanuric chloride

Decomposition products of BZ

M R T	2-hydroxy-2,2-diphenylethanoic acid
M R G	3-quinuclidinol
M R GT	diphenyl ketone
M R GT	alpha-phenylbenzenemethanol

Decomposition products of CS

M RH GT	2-chlorobenzaldehyde
M RH G	dicyanomethane
MIRH	1,1-dicyano-2-(2-chlorophenyl)ethane
M	1,1-dicyano-2-(2-chlorophenyl)pentanone-4
MIRH G	1-cyano-1-carboxamido-2-(2-chlorophenyl)ethene
M	1-cyano-2-(2-chlorophenyl)ethene
MI H	1-cyano-1-carboxamido-2-(2-chlorophenyl)ethane
M G	1-cyano-2-(2-chlorophenyl)ethane
MIRH	1,1-dicyano-2-(2-chlorophenyl)oxirane
M	1-cyano-1-carboxamido-2-(2-chlorophenyl)oxirane
MIRH	2-chlorobenzoic acid
MI H G	2-chlorobenzenemethanol
MI H G	1-(2-chlorophenyl)-1-butene-3-one

Decomposition products of irritants

M RH GT	1,2-diphenyl-1,2-dicyano-ethene
RH	methylarsine oxide
R	ethylarsine oxide
I T	phenylarsine oxide
M H	bis(5,10-dihydrophenarsazine)oxide
M H T	diphenylarsenious oxide

Precursors and impurities

Sarin and soman precursors

MIRH	chloromethane
MIRHP	methylphosphonous dichloride
MI	phosphorus trichloride
M	methylphosphonic chloride fluoride
MIRHP	methylphosphonic dichloride
MIRHPC	methyl methylphosphinate
MIRHP	dimethyl phosphite
M	trimethyl phosphite
MIRHP G	dimethyl methylphosphonate
M	hydrogen methyl methylphosphonate
M G	diisopropyl phosphite
M P G	triisopropyl phosphite
M	isopropyl methylphosphonochloridate
MI HPC	1,2,2-trimethylpropyl methylphosphonochloridate
MI H G	3,3-dimethylbutanone-2

VX precursors

MIRH	diisopropylamine
MIRH C	2-diisopropylaminoethanol
M	2-diisopropylaminoethylchloride
IRH	2-diisopropylaminoethylchloride hydrochloride
MIRHP	methylthiophosphonic dichloride
M	O,O-diethyl methylthiophosphonate
MIRHP	ethyl hydrogen methylphosphinate
MI HP	ethyl methylphosphonochloridate
MIRHPCG	2-diisopropylaminoethyl ethyl methylphosphonite
MIRHP	diethyl methylphosphonite
M	phosphorus pentasulphide
MI	sulphur

Tabun precursors

MIRH	dimethylamine hydrochloride
MIR P	phosphoroxychloride
M	dimethylaminophosphoryl dichloride
M	ethyl N,N-dimethylphosphoramidochloridate
M	ethyl phosphorodichloridate

Mustard precursors

M	thionyl chloride
MIR	sulphur dichloride
MIRH	oxirane
M	2-chloroethanol
M	chloroethene
MIRH	ethene

Lewisite precursors

MI	arsenic trichloride
M	acetylene

VX impurities

M HP	2-diisopropylaminoethyl hydrogen methylphosphinate
MI H G	2-diisopropylaminoethyl ethyl methylphosphonate
M	diethyl S-2-diisopropylaminoethyl phosphorothioate
MI G	O,S-diethyl methylphosphonothioate
MI G	O-ethyl-O-hydrogen methylphosphonothioate
MI HP	diethyl phosphoric acid
MI G	triethyl thiophosphate
MI H	ethylene sulphide
MI H G	isopropylamine
MIRH	dicyclohexylcarbodiimide
M	N,N'-dicyclohexylurea

Tabun impurities

M HPCG	triethyl phosphate
M	N,N-dimethyl ethyl isopropyl phosphoramidate

Mustard impurities

MI H G	2,2'-dichlorodiethyl disulphide
M G	2-chloroethyl-2-chloropropyl sulphide
M G	2,2'-dichlorodipropyl sulphide
M G	2-chloroethyl-2-chloropropyl disulphide
M G	1-(2-chloroethylthio)-2-(2-chloropropylthio)ethane

CS impurities

MI	2,2-dicyano-2-(2,4-dichlorophenyl)ethene
M	2,2-dicyano-2-phenylethene

Derivatives and analytical artifacts

Derivatives of decomposition products of nerve agents

M	G	ethyl methyl methylphosphonate
M	H G	isopropyl methyl methylphosphonate
MIRHP	G	methyl 1,2,2-trimethylpropyl methylphosphonate
M		cyclohexyl methyl methylphosphonate
M		O-methyl-S-2-diisopropylaminoethyl methylphosphonothioate
M	HP G	trimethyl phosphate
M		ethyl isopropyl methylphosphonate
MIRHP		ethyl 1,2,2-trimethylpropyl methylphosphonate
MIRHPC		isopropyl 1,2,2-trimethylpropyl methylphosphonate
M		bis(t-butyldimethylsilyl) methylphosphonate
M		ethyl t-butyldimethylsilyl methylphosphonate
M		isopropyl t-butyldimethylsilyl methylphosphonate
M		t-butyldimethylsilyl 1,2,2-trimethylpropyl methylphosphonate
M		cyclohexyl t-butyldimethylsilyl methylphosphonate
M		tris(t-butyldimethylsilyl) phosphate
M	HP	S-2-diisopropylaminoethyl t-butyldimethylsilyl methylphosphonothioate

Derivatives of decomposition products of vesicants

M	G	bis(trimethylsilyl)ether of 2,2'-dihydroxydiethyl sulphide
M	G	bis(trifluoroacetyl)ester of 2,2'-dihydroxydiethyl sulphide
M	G	bis(trimethylsilyl)ether of 2,2'-dihydroxydiethyl sulphoxide
M	G	bis(trifluoroacetyl)ester of 2,2'-dihydroxydiethyl sulphoxide
M		bis(trimethylsilyl)ether of 2,2'-dihydroxydiethyl sulphone
M		bis(trifluoroacetyl)ester of 2,2'-dihydroxydiethyl sulphone
M	G	tris(trimethylsilyl)ether of tris(2-hydroxyethyl)amine
M	G	tris(trifluoroacetyl)ester of tris(2-hydroxyethyl)amine
M	G	bis(trimethylsilyl)ether of methyl bis(2-hydroxyethyl)amine
M	G	bis(trifluoroacetyl)ester of methyl bis(2-hydroxyethyl)amine
M	G	bis(trimethylsilyl)ether of ethyl bis(2-hydroxyethyl)amine
M	G	bis(trifluoroacetyl)ester of ethyl bis(2-hydroxyethyl)amine
M		bis(trimethylsilyl)ether of bis(2-(2-hydroxyethylthio)ethyl)ether
M		bis(trifluoroacetyl)ester of bis(2-(2-hydroxyethylthio)ethyl)ether

Derivatives of decomposition products of CS and BZ

M	RH	methyl ester of 2-chlorobenzoic acid
M		ethyl ester of 2-chlorobenzoic acid
M		methyl ester of 2-hydroxy-2,2-diphenylacetic acid
M		methyl ester of hydroxyacetic acid
M		t-butyldimethylsilyl ester of 2-hydroxy-2,2-diphenylethanoic acid
M		bis(t-butyldimethylsilyl)ester of 2-hydroxy-2,2-diphenylethanoic acid

Artifacts of CS and its decomposition products

M	1,1-dicyano-2-(2-chlorophenyl)cyclopropane
M	dimethylacetal of 2-chlorobenzaldehyde
M	diethylacetal of 2-chlorobenzaldehyde
M	2-chloroacetophenone
M	2-chlorophenyl oxirane

Artifacts of vesicants

M		2-hydroxyethyl-2'-methoxyethyl sulphide
M		2-chloroethyl-2'-methoxyethyl sulphide
M	H	2,2'-dimethoxydiethyl sulphide
M		2-chloroethyl-2'-isopropoxyethyl sulphide
M		2,2'-diisopropoxydiethyl sulphide
M		diethoxy-2-chlorovinylarsine

Australia	CD/CW/WP.329	Strategy for preparing for the implementation of the Chemical Weapons Convention in Australia NOT REPRODUCED (see WP volume)	Also issued as CD/1055 5.2.91

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Germany, UK	CD/CW/WP.330 and Corr.1	Report on two joint chemical weapons practice challenge inspections NOT REPRODUCED (see WP volume)	Also issued as CD/1056 and Corr.1 8.2.91

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New Zealand	CD/CW/WP.331	Report of a national trial inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1057 13.2.91

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Hungary	CD/CW/WP.332	Provision of data relevant to the Chemical Weapons Convention NOT REPRODUCED (see WP volume)	Also issued as CD/1061 18.2.91

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AHCCW Chairman	CD/CW/WP.333	Working paper presented by the Chairman of the Ad Hoc Committee: "Organization of work for the 1991 session" NOT REPRODUCED	20.2.91

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Austria	CD/CW/WP.334	Letter dated 19 February 1991 from the Permanent Representative of Austria addressed to the Secretary-General of the Conference on Disarmament transmitting three studies related to the verification of chemical weapons NOT REPRODUCED (see WP volume)	Also issued as CD/1062 21.2.91
		* * *	
France	CD/CW/WP.335	Second trial request inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1063 21.2.91
		* * *	
USA	CD/CW/WP.336	A report on the destruction of 3-Quinuclidinyl benzilate (BZ) NOT REPRODUCED (see WP volume)	Also issued as CD/1074 20.3.91
		* * *	
Peru	CD/CW/WP.337	Working paper on challenge inspection/inspections on request NOT REPRODUCED (see WP volume)	Also issued as CD/1075 14.5.91
Austria	CD/CW/WP.338	Letter dated 16 May 1991 from the Deputy Permanent Representative of Austria to the Secretary-General of the Conference on Disarmament transmitting a study entitled "Detection of inhibitors of the enzyme acetylocholine esterase over long distances using optic fibres" NOT REPRODUCED (see WP volume)	Also issued as CD/1076 21.5.91

CONFERENCE ON DISARMAMENT

CD/CW/WP.339
29 May 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

BELGIUM

National Registers and "Definition of Capable Facilities"

1. National Registers

National Registers would include all facilities in the chemical industry on the basis of "The International Standard Industrial Classification of All Economic Activities" of the United Nations (ISIC).

2. Definition of "capable facilities"

On the basis of National Registers, the following definition of "capable facilities", eligible for verification, is suggested:

(a) A chemical production facility means any combination of equipment designed for carrying out large scale chemical synthesis (reaction). Facilities or parts of facilities (plants, units) designed for product formulation processes or mechanical processing of chemicals would not be included in the definition.

(b) A chemical synthesis (reaction) means "any transformation of chemicals where rearrangements of atoms, groups of atoms, ions or radicals of one or more substances (called basic or raw materials) result in the formation of new substances".

Not included are physical or physicochemical processes, such as distillation, vulcanization, tanning, product formulation and mechanical processing.

Norway	CD/CW/WP.340	Letter dated 30 May 1991 from the Deputy Permanent Representative of Norway to the Deputy Secretary-General of the Conference on Disarmament transmitting a document entitled, "Verification of alleged use of chemical warfare agents: application of procedures after a simulated chemical attack on an air base" NOT REPRODUCED (see WP volume)	Also issued as CD/1078 30.5.91

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UK	CD/CW/WP.341	Verification of the Chemical Weapons Convention: practice challenge inspections at civil chemical plants NOT REPRODUCED (see WP volume)	Also issued as CD/1080 5.6.91

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CONFERENCE ON DISARMAMENT

CD/CW/WP.342
6 June 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

Finland and the Netherlands

Working Paper

THE NETWORK OF LABORATORIES UNDER THE CHEMICAL WEAPONS CONVENTION

Possible structure and functions

Summary

Under the Chemical Weapons Convention several analytical and related tasks have to be fulfilled, such as the analyses of samples taken by inspectors, the compilation and updating of the analytical chemical data base, the development and testing of verification instruments, the training of inspectors to use these instruments, the handling and transport of samples, quality control of participating "Accredited Laboratories", etc.

In this paper a short analysis is made of the different functions which various laboratories can have under the Chemical Weapons Convention, in particular the "Laboratory of the Technical Secretariat" and the "Accredited Laboratories" in States Parties certified by the Organization to perform specific analytical and other tasks.

There are two main options:

1. A centralized structure: a substantial central laboratory performing most of the required tasks. A limited number of tasks would be left for "Accredited Laboratories".

2. A decentralized structure: most of the tasks would be performed by "Accredited Laboratories" on request. The "Laboratory of the Technical Secretariat" would be modest, would only carry out those tasks which cannot be done elsewhere, bit it would play a stimulating and coordinating role.

It is concluded that the second option is preferable.

GE.91-61137

1. Functions of laboratories under the Convention

In this section, a description is given of the various analytical and other technical tasks which have to be carried out in laboratories under the CW Convention. Three types of laboratories are being considered: the Laboratory of the Technical Secretariat, Accredited Laboratories and National Laboratories.

1.1 Verification analysis

During inspections the inspection team will utilize agreed instrumentation for performing chemical analyses on site. In many cases it is envisaged that this instrumentation will be sufficient to fulfill the main purpose of the inspection (e.g. verification of the declarations concerning the storages of CW and of production in the chemical industry). In some other cases this instrumentation is from the outset regarded only as a tool for selection of the best samples, which will later on be analysed in fully equipped off-site laboratories (e.g. in cases of alleged use and challenge inspections). During all inspections the inspectors may, however, encounter situations when the available instrumentation is not sufficient to give a clear proof of compliance, and further chemical analysis in off-site laboratories is needed. This is especially obvious in situations when the on-site analyses would indicate a possibility of non-compliance with the Convention.

The transport of samples for analysis to off-site laboratories must thus be possible. The draft Convention (CD/1046) furthermore requires in cases of off-site analyses independent results from at least two laboratories. If their results are not in agreement, a third laboratory should be called in.
Thus the minimum number of laboratories performing verification analysis is three. In order to secure prompt results, a network of laboratories in different parts of the world would provide a better basis for this task.

The analytical work that these off-site laboratories would be required to do can be summarized as follows:

- Unambiguous identification of scheduled compounds
- Structure elucidation of possible novel agents
- Semiquantification

1.2 Analytical chemical methods development

The selection of the recommended analytical methods used for the Organization should be based on international cooperation for method development, including international interlaboratory comparisons and collaborative tests. In order to ensure that the Organization has at all times at its disposal the best available means for verification, it is an absolute necessity to develop these analytical methods continuously. It is evident that all the verification laboratories will on their own improve the existing methods and develop new ones in parallel. The evaluation of these methods may be carried out by the Technical Secretariat and/or by one or more Accredited Laboratories. The Laboratory of the Technical Secretariat is responsible for the coordination in this field.

The methods development involves extensive testing in order to ensure the validity and wide applicability of new improved methods before they can be recommended for verification purposes. Despite this requirement, the validation process of new methods should be efficient and prompt enough to guarantee that up-to-date methods are used.

1.3 Synthesis of reference compounds

The verification laboratories and the inspectors will need several types of reference compounds for calibration of instruments and comparison of analytical data for identification purposes. As far as scheduled compounds are concerned, these reference chemicals can be obtained either by synthesizing them in the Accredited Laboratories or by purchasing them from single small-scale facilities or other declared laboratories. Other chemicals could be obtained from industry, etc. In all cases the reference compounds have to be validated for purity and authenticity (cf. CD/CW/WP.272).

In addition to the synthesis of reference compounds for the already agreed scheduled chemicals, synthesis may also be needed when new chemicals are added to the schedules.

1.4 Compiling and updating the analytical database

An analytical database containing identification data of as many scheduled compounds as possible is extremely important to allow on-site analysis and facilitate analyses in verification laboratories.

The database will be very extensive as it will eventually include data on the scheduled chemicals, their precursors, degradation products, and characteristic impurities recorded with all recommended analytical techniques. In order to ensure that there is an adequate

database available when the Convention enters into force, it seems necessary to accept initially data from several sources (other databases, verification laboratories, etc.) and to proceed to record fresh data in due course. The workload connected with the recording of fresh data obtained by using approved operating procedures could be distributed among Accredited Laboratories. Another option could be that, for standardisation purposes, in a later stage only one laboratory would record the data with analytical instruments and insert these into the central database.

Once the database contains the relevant data on scheduled compounds, the addition of data of possible new chemicals which are added to the Schedules will pose no difficulties.

For quality control reasons, only the Technical Secretariat should be allowed to add data to the central database. This database would be provided to all laboratories performing verification analyses. They could use the data base directly ("on line") and/or incorporate relevant data in the computers of their own instruments. This requires compatible instrumentation and frequent updates of the database.

1.5 Training of inspectors in analytical chemical tasks

Training has to follow a programme approved by the Organization. A large part of the training can be assigned to different laboratories according to the required volume of trainees at a certain time. Training programmes are probably needed on a continuous basis, but in varying numbers and on different levels.

1.6 Handling of authentic samples

The samples which the inspection team decides to dispatch for chemical analysis in off-site laboratories have to follow a deliberate procedure with which their identity is concealed from the laboratories which analyse them (cf. CD/CW/WP.253). At the same time the integrity of the samples has to be secured. This involves at least the following stages:

- Transport from the site to the Technical Secretariat utilizing approved means.
- Division of the samples into at least three identical lots.
- Preparation of necessary control samples.
- Record keeping of all samples which are being analysed.
- Transport of samples from the Technical Secretariat to two or three Accredited Laboratories.
- Decoding of laboratory results and putting the results together.

- Informing the relevant section of the Technical Secretariat and/or the inspection team involved.

1.7 Quality assurance

The credibility of the verification system is to a large extent dependent on the confidence that the analyses of the samples can give definite proof of the contents of the samples and thus serve as a basis for conclusions with respect to compliance or non-compliance with the Convention. Furthermore, the confidence in the validity of the results should be equal for all the Accredited Laboratories. This emphasizes the necessity to monitor the performance and capabilities of the Accredited Laboratories by the Organization on a regular basis. Within the Organization this may be best achieved by establishing a specific group of experts on quality assurance.

The accreditation process will involve an inspection of the laboratories during which a team of experts examines the facilities, equipment as well as written technical and operating procedures.

The performance of new laboratories seeking accreditation should be compared with that of the existing Accredited Laboratories by several tests. As these tests are best combined with the quality assurance monitoring, the same unit within the Organization should be assigned to both tasks.

The principal method for quality assurance monitoring would entail tests which would be indistinguishable from authentic analytical tasks assigned to the Accredited Laboratories. The utilization of good laboratory practices (GLP) should also be monitored by frequent visits to the laboratories. The attainment of the status of Accredited Laboratory and its prolongation should be contingent upon the performance shown in the quality assurance tests. The Director General should annually report on the quality control activities of the Organization.

It is envisaged to discuss the different aspects of quality assurance in a separate working paper.

1.8 Technical support for the Organization

The entry into force of the Convention cannot mean the freezing of the technical knowledge available and applied within the Organization and its Technical Secretariat. Various Accredited Laboratories may be involved in developing new equipment. Some equipment may also be commercially developed or under contracts financed by the Organization or by States Parties ("national support programmes").

Besides sophisticated mobile analytical equipment, the Technical Secretariat will also need other means to detect chemical warfare agents, protection gear, seals and markers, equipment to take samples and transport these, etc.

It would be up to the Technical Secretariat to evaluate the quality of existing and new equipment and to decide whether these should be utilized by the Organization.

1.9 Maintenance of equipment for on-site inspections

Of course, the equipment which is being used by the Technical Secretariat needs to be kept up. In particular, the sophisticated analytical verification equipment used during inspections needs to be calibrated, repaired, etc.

2. Structure of the laboratory network

2.1 General approach

2.1.1 Certain tasks for the implementation of the Convention have to be performed by the international staff of the Technical Secretariat at or near the Headquarters of the Organization. Other work can be done by Accredited Laboratories in States Parties, on request or under contract by other laboratories and scientific/technical or sometimes even commercial institutes.

2.1.2 Two different alternatives could be pursued in structuring the laboratory network:

- The first approach would be to try to perform as many tasks as possible in a kind of "central laboratory". The analytical chemical work entrusted to off-site laboratories would be assigned to Accredited Laboratories in States Parties. In this option, such a central laboratory would be a fully equipped analytical laboratory, would have possibilities to synthesize chemical warfare agents, would have the capability to develop and test new analytical methods and equipment, calibrate mobile analytical instruments, etc..

- In the second approach, facilities and knowledge in States Parties would be optimally utilized on request or under contract of the Technical Secretariat. In this option analytical and other related tasks would be performed in Accredited Laboratories to the maximum extent possible. The corresponding unit of the Technical Secretariat would not comprise an analytical laboratory. The technical workload of the Secretariat would also

be extensively reduced by maximalizing input by States Parties through "national support programmes", contract research etc..

Of course, all kinds of intermediate solutions between these two main approaches are possible.

2.1.3 The first option would be costly, in particular if one tries to build up all the necessary facilities, skills and knowledge within a central laboratory which subsequently could be underutilized. It seems much more cost-effective to make, as far as possible, use of the facilities and knowledge available in States Parties in harmony with the requirements of the Organization. The authors of this paper therefore have a preference for a modified second approach, realizing that a number of technical activities need to be done at or near Headquarters in what we call hereafter the Laboratory of the Technical Secretariat but which in practice also has many characteristics of a "technical workshop".

In the following section, a description is given of the possible tasks of the different laboratories as seen by the authors of this paper.

2.2 Headquarters/Laboratory of the Technical Secretariat

2.2.1 The Technical Secretariat has many organisational and coordinating tasks with respect to the issues mentioned above. Part of the work can thus be done at the main office building of the Technical Secretariat, while other work may have to be done in a laboratory/workshop in the neighbourhood of the main building depending on local circumstances. This laboratory/workshop forms part of the Technical Secretariat. It is up to the Director General how best to organize the division of work between the different sections of the Technical Secretariat. For the time being, we call this ensemble the Laboratory of the Technical Secretariat.

It is conceivable that some of the operational tasks mentioned hereunder are also done at regional branches of the Technical Secretariat in various parts of the world if these are established.

2.2.2 The Laboratory of the Technical Secretariat could be assigned with, inter alia, the following tasks:

Operational tasks:

- maintenance and storage of verification equipment and other tools used by inspectors (communication, protection, etc.);
- calibration of mobile analytical equipment (if this cannot be done elsewhere);

- splitting samples and packaging these for transport to Accredited Laboratories for analysis; preparation of control samples; record keeping;
- maintenance and storage of protective means (if so decided under Article X of the Convention).

Quality control with respect to Accredited Laboratories:
- organizing "Round Robin" tests;
- preparing samples for these (if this cannot be done elsewhere);
- organizing laboratory audits by relevant experts.

Evaluation:
- (organizing the) evaluation of new inspection tools to be used by inspectors;
- organizing the evaluation of new analytical methods to be used by accredited laboratories.

Training:
- training of inspectors in using their inspection tools (if this cannot be done elsewhere, for example in Accredited Laboratories).

Data base:
- organizing the addition of data to the central analytical data base and its quality control.

2.3 Accredited Laboratories

Accredited Laboratories are laboratories located in States Parties which are certified by the Technical Secretariat to perform specific tasks, in particular the chemical analyses of samples for verification purposes. These laboratories can also assist in various other ways. They can, for example, provide scheduled chemicals for calibrations and "round robin" tests, improve equipment and analytical methods etc., including under contract with the Technical Secretariat. It is to be expected that some of the Accredited Laboratories will specialize in providing particular knowledge to the Organization and be commissioned special responsibilities in this regard.

Regular tasks:
- verification analysis;
- analytical chemical methods development;
- training of inspectors in analytical chemical tasks;
- synthesis of reference compounds;
- recording analytical data for the database;
- technical support to the Organization.

2.4 National Laboratories

National Laboratories of States Parties perform tasks assigned to them by their National Authority. They do not automatically form part of the laboratory network for verification analyses (but could, of course, also become Accredited Laboratories). It is conceivable that such laboratories - either under contract or under "national support programmes" - could develop analytical methods and instruments which could be useful for the Technical Secretariat. (The same holds for other scientific or commercial institutions.)

It could also be envisaged that States Parties which do not consider it necessary to build a National Laboratory of their own, could cooperate with other countries in the same region in establishing a joint laboratory that would act as a National Laboratory for several participating countries.

In order to contribute to standardization, the National Laboratories would be provided with the central analytical database and the recommended operating procedures of the Organization.

3. Conclusions

In good cooperation between the Technical Secretariat and States Parties, it seems possible to set up a cost-effective laboratory network meeting all the needs of the Technical Secretariat under the Chemical Weapons Convention. The system would optimally utilize knowledge and equipment already available or to be developed by States Parties. This approach will require a reliable quality assurance system.

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AHCCW	CD/CW/WP.343	Recommendation by the Ad Hoc Committee on Chemical Weapons NOT REPRODUCED	12.6.91

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Spain	CD/CW/WP.344	Report on a national trial inspection in the civil chemical industry NOT REPRODUCED (see WP volume)	Also issued as CD/1082 12.6.91

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CONFERENCE ON DISARMAMENT

CD/CW/WP.345
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Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

UNITED STATES OF AMERICA

Analytical Database Discussion Paper

Introduction:

It must be recognized that the analytical database will be only one segment of the larger database that will be required for verification of the CWC. The complete database available to the Technical Secretariat will encompass many functions which, though not required for analytical purposes, will be needed for reporting, auditing, accounting and similar administrative functions. We intend, in this paper, to consider only the requirements for chemical analysis to support verification; i.e., basic reference data, analytical methods, and the treatment of analytical results. We anticipate that even this segment of the total database will be complex since each of these functions will require a separate computer program, each with a different purpose, and to some extent different users and different sources of data. Many of the data handling tasks will have commonalities but the exact computer program for each function will probably be unique. User manuals and training programs for each functional area will be a necessity.

Types of Data

The types of data to be entered into the database will be of three general types: compound analytical reference data, analytical procedures, and the results of the analyses performed during inspections. Probably of most interest to date is the analytical reference data where several working papers have already been produced. The compounds to be entered into the database can be an almost infinite number. Therefore, we believe that criteria should be developed and agreed to place reasonable limits on this number. One approach would be to include only Schedule 1 compounds together with their precursors, degradation products and related derivatives. The data to be included for even this limited list become quite large and more stringent criteria might be necessary. Each compound should be precisely identified by CAS number. For at least the primary compounds, physical and chemical properties should be included and where available toxicity data by a standard method should be added.

As for the analytical reference data itself, of primary importance to the analytical community, is that both spectrometric and chromatographic data for all compounds in the database must be included. This leads to the question of how will these data be obtained. We generally support the concept that the chromatograph and spectra should be solicited from the laboratories which have been active in the field and which have experience in the identification of chemical agents. Other data especially spectra of precursors and degradation products, may be available in commercial databases and the chemical literature. Other sources of standard spectra of organic compounds in the U.S. are the National Institute of Standards and Technology, (formerly the National Bureau of Standards), the Environmental Protection Agency and the National Institutes of Health. Similar sources are available in other countries. There will, in all probability, be multiple sets of data available for each material. Rather than including all data in the database, quality tests must be used to evaluate the available data and select only one set for inclusion in the database. Such tests have been developed by the U.S. Environmental Protection Agency and others. These techniques are available to ensure that only the highest quality data is included.

The selection of the types of spectra to be included in the database is a critical issue that depends to some extent on whether the analyses are to be performed on-site during an inspection or at a fixed laboratory off-site. The types of instruments which can be used on-site or in a mobile laboratory will limit the spectra to be included in the database. For on-site use, we propose that gas chromatography (GC) retention times, electron impact (EI) mass spectra would certainly be included. Infrared (IR) spectra would also be desirable. The instrument operational conditions under which the data were obtained would be specified in agreement with the prescribed analytical standard operating procedure; i.e., GC temperature/flow rate conditions, GC column type, etc., mass spectrometer ionizing voltage, etc., additional data for use in off-site laboratories, where more sophisticated instruments will be available should also be included for referee or reference laboratory use. These would include nuclear magnetic resonance (NMR) spectra and possible others which would be needed for structure elucidation if that is desired.

A separate database section should be developed comprising the reference analytical procedures, i.e., standard operating procedures for the instruments selected to be used in the verification process. A necessary part of this procedural database would be standard sampling procedures for all types of samples that may be encountered during an inspection, i.e., process samples, environmental samples, etc., and standard packaging,

storage and transportation procedures. An earlier paper by the U.S., CD/CW/WP.266 discussed this area in some detail and may serve as a base for development of procedures. Standard sample preparation procedures for the instruments selected would be included in this database section. Of critical importance is the development of instrument calibration procedures and the use of known analytical standards for both pre and post analysis calibration and periodic sample blanks during the analytical process.

A third portion of the analytical database comprises the methodology for handling the analytical results arising from inspections, via the chromatograms, spectra, field measurements etc., accumulated at the inspection site. Special concern has been expressed here to protect commercial chemical information. This concern may be dealt with by restricting the compounds included in the database as described earlier or by limiting the analytical methods which may be used to analyze samples or both. The analytical results should be capable of being entered into the database in a variety of formats, i.e., textural, tabular, graphic, structural, etc., or show reaction data. The database should also permit cross reference or analysis of data in a variety of forms and formats.

Sources of Data

The sources of data for the various elements of the analytical database will be varied and numerous as a function of its purpose and intended use. Initial declarations of compounds included in the inventories of parties will identify the primary compounds to be included in the database. From these, precursor and degradation compounds and their derivatives can be determined. Analytical data gained by the inspectors from on-site analyses will be entered to confirm those declarations. Should anomalies arise, additional, more detailed analyses will be performed at off-site laboratories.

Data may be developed during the laboratory certification process which must be retained in the database for future comparison. Likewise, analytical methods development data will be a continuous process since new, more compact, and more accurate instruments are being developed every day. The database must provide comparison information between established methods and any new methods developed to allow correlation of analytical results over time. Additional inter-laboratory comparisons may be necessary to ensure that uniform results are being obtained by the various parties involved in the inspection process. Further, a quality assurance (QA) program for all laboratories involved must be maintained in the database. The QA program can encompass both quality control aspects, i.e., instrument calibration and

maintenance records as well as quality assessment features such as audits surveillance, control charts and duplicate sample analyses. Historical or bibliographic data will also be maintained in the database as a ready reference for all users. While all of these features may not be available in the on-site or mobile laboratory, they will certainly be assessable at the off-site facility.

Data Handling Tasks

The database should be adaptable to a variety of tasks ranging from data collection, not only from earlier mentioned inspection results, but from other inspection activities such as process monitoring instrumentation input which might be automatically entered, to sensor and seal data which could be either automatically or manually entered. The inspectors would be expected to be able to retrieve certain data while at the inspection site in order to collaborate on-site analytical results. Similarly, the Technical Secretariat or the National Authority involved may need to recover analytical results or perform statistical manipulation of data contained in the database either during an inspection, as part of Quality Control or as part of their oversight function. In this regard, protection of data is again a concern, whether to protect proprietary or business confidential information or to prevent unauthorized disclosure of any results contained in the database.

Database Operations and Users

The analytical data base will be used in a variety of ways. At the analyst level, comparison to stored reference spectra constitutes one use. In one possible operational mode, infrared and mass spectra would be sorted in separate arrays. The GC retention time would serve as a trigger to search segments of a spectral array for a match to experimental data. Some boundary conditions on retention time would be preset; e.g., =/- 20 seconds, and the spectral arrays would only be searched for compounds falling within those boundaries. Likewise, a floor on GC peak amplitude would also be preset such that only peaks whose amplitude exceeded the floor would trigger a search. The floor would correspond to a predetermined concentration level that was deemed to be significant. The best spectral match would then be displayed along with the experimental spectra for visual comparison by the analyst or inspector. Other information in the database could also be displayed; i.e., physical properties, toxicity, etc.

Inspectors could use the database on location to reference the site history including the declaration, the inspection checklist, prior readings from sensor, seals or process monitoring devices,

previous inspection results and similar on-site information needed to complete and confirm inspection findings.

The Technical Secretariat could compile the data necessary for reporting by consolidation of inspection results and develop information needed to respond to specific questions which may arise. Similarly, simulations could be developed and tested to serve as guides for improved, modified or new inspection procedures which may be required.

Thus the composition, operation and users of the database represent a widely varied group. The database program(s) must be designed to respond easily and quickly to meet these diverse requirements.

CONFERENCE ON DISARMAMENT

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Ad Hoc Committee on Chemical Weapons

UNITED STATES OF AMERICA

INFORMATION PROCESSING FOR CW MONITORING

1.0 Introduction

Upon entry into force of the Chemical Weapons Convention (CWC), State Parties will be required to declare all facilities and chemicals covered by the provisions of the Convention. These declarations fall into two general categories: initial and periodic. Initial declarations may take place either when the CWC first enters into force, or at later times when newly initiated activities in a member state are subject to the provisions of the Convention. Periodic declarations would pertain to chemicals on any or all Schedules 1, 2, and 3. The Organization will be required to verify compliance of all State Parties with these provisions.

To carry out its obligations the Organization would have to implement an information processing system that would receive reports from member states, and data generated by its own data-collection mechanism as input to the evaluation of compliance. The totality of the various data sets would be analyzed, and the results would be evaluated, in the process of monitoring compliance by the state.

The information processing system should be designed to process the data in a cost-effective manner within the constraints imposed on the Organization by the CWC. Some of the parameters that would affect the design are: classes of data, locations where particular classes of data are needed, timeliness of access to a particular class, frequency of use, type and complexity of processing for a given class, security of, and access control to the data.

At this time, the information processing requirements have not been identified in any substantive detail. It is generally accepted that reports from the member states and monitoring activities by the Organization will contribute to the establishment of a data base; some efforts are under way to determine some of its contents. In a companion paper the United States has suggested some elements that should be included in an analytical data base.

This paper attempts to identify some of the components of the information processing system using the current rolling text CD/1046 as a basis. The paper is organized according to the difference types of verification functions outlined in CD/1046, namely verification of initial declarations, routine monitoring of facilities, chemicals and activities covered by the Convention, and extraordinary monitoring required by unforseen circumstances such as those involving challenge inspections.

2.0 Verification of Declarations

Initial declarations would contain information about facilities, chemicals and chemical weapons. Each facility and each National Authority would constitute an information processing node; these nodes and their links to the headquarters or other facilities of the Organization would form the global information processing system of the Organization. Quantitative information about facilities such as location, size, production, or storage capacity, can easily be stored and utilized in an automated information processing system. However, plans, drawings and photographs would be a type of data which would not be easily amenable to automation, although an automated cross-reference system could be implemented.

Verification of facility declarations would involve such activities as: a) manual examination of plans, photographs, and drawings of process lines and storage facilities, b) site visits to confirm the location and layout of the facility, the specifications of process lines and equipment, and the capacities of storage buildings and containers, c) analysis of plant specifications to confirm declared capacities, and possibly, d) analysis of samples taken from the facility to confirm past production of chemicals reported to have been produced at the facility.

Product declarations would contain past production of Schedule 1 chemicals, stored Schedule 1 chemicals in bulk or as weapons, and the identification per facility of past and/or current production of chemicals listed in Schedules 2 and 3. Confirmation of these declarations might involve, at least, examination of the facility records, and at most, collection and analysis of samples taken from each declared facility.

The information processing requirements with respect to the initial declarations and their verification would consist of, inter alia, processing and accounting information about facilities and products. A classification system would store data about chemicals on the basis of the three schedules. It is anticipated that, initially, the most detailed information would be about chemical weapons, chemical weapons facilities, and schedules for the destruction of the weapons. Chemicals would be identified by chemical name, structural formula and Chemical Abstracts Service registry, if available, In the case of declaration of chemicals not already listed in the Schedules, toxicity levels and methods of

determining toxicity would be required.

As part of verifying the initial, and other, declarations, the rolling text provides for analysis of samples. Since the objective would be to confirm known and declared chemicals, instruments and procedures capable of such confirmation would already be in existence; this information would also be part of the information processing system of the Organization.

3. Verification of Destruction

Member states possessing chemical weapons would be required to provide the Organization with detailed plans for their destruction. These plans would describe the destruction facility and the procedures to be followed. An archival system could be implemented in an automated mode. Since the number of destruction facilities would be small worldwide, the information processing requirements for the verification of declarations of destruction facilities would likely be smaller worldwide than for Schedule 2 and 3 monitoring.

Monitoring the destruction of chemical weapons would require more elaborate information processing. One type of information would be accounting data about items and agents being destroyed and remaining stock balances. Another would contain information about the status of the destruction facility, including safeguards information to prevent diversion of agents or weapons during the final destruction step. Analytical chemical data would be needed for independent verification of the identity of the substances being destroyed. If the Organization were to adopt procedures for utilizing existing facility instrumentation to confirm the identity of chemicals, verification would consist of ensuring that instruments are calibrated properly, and that the integrity of the measurments is maintained.

The continuous presence of inspectors at the destruction facility, or possibly at other facilities, as well a the volume of data and the complexity of processing would likely require the establishment of a local information processing system; thus, some of the nodes of the global information system would have their own data bases and local processing requirements. The distributed nature of the information processing system would also pose substantial requirements for the security of the equipment and the data. Security requirements and system specifications to meet them need to be established.

4.0 Routine Monitoring

During the first ten years of the Convention, routine monitoring would provide data for evaluating compliance with non-diversion of

chemical weapons, the progress on destruction of the declared stockpiles, and whether facilities and chemicals in Schedules 2 and 3 are not used for purposes prohibited by the Convention. After all weapons have been destroyed, routine monitoring would be confined mainly to the latter function.

Routine monitoring of stockpiles would involve the collection and processing of accounting data from storage sites, and the operation and maintenance of some type of security systems as supplements to the inspection regime. The data, periodically updated, would be part of the central data base, which would be used to monitor compliance. The identification of substances stored at various sites would be done primarily by portable or transportable analytical instruments designed to identify any of the declared chemicals at a given site.

Monitoring of Schedule 2 and 3 declarations would place on the Organization demands substantially different from those for Schedule 1. The number of states declaring the possession and destruction of chemical weapons would be a fraction of those making declarations related to chemicals on Schedules 2 and 3. Furthermore, the total number of facilities and the number of chemicals involved in Schedule 1 declarations would be small compared to facilities and chemicals covered by Schedules 2 and 3. At this time, there are no data on the number of facilities world-wide that would be affected by the declarations required for Schedule 2 and 3. The primary impact of this number would be on the data storage requirements at the headquarters of the Organization. Data processing procedures would not be substantially affected, although their might be an indirect impact on the speed and throughput specifications of the central processing unit. The current language of the rolling text requires annual reports for Schedule 2 and 3 chemicals; even if inspections occur more frequently than once a year, time intervals of weeks or months might elapse between successive data sets. The slowest of the present generation minicomputers should be able to process the data well within the required time intervals.

Routine compliance monitoring of Schedule 2 and 3 chemicals would entail the verification of declarations pertaining to these chemicals. These declaration would identify facilities, chemicals and quantities. The analytical information required to confirm that a declared chemical was present at the associated facility would be minimal, since the operator would have no incentive to declare the presence of a controlled chemical, if the chemical was not present. Most information of interest to the Organization would be quantities produced, processed, traded, and consumed. If these data are to be used for constructing materials balance within a closed boundary, elaborate processing of data would be involved. Thus, the size of the central processing facility might become substantial.

5.0 Challenge Inspections

By their nature, challenge inspections would not be considered routine operations. The actions undertaken in these non-routine investigations would be directed toward the identification of prohibited facilities, activities, or chemicals.

A method would be to analyze samples taken from the suspect site. One of the objectives of a challenge inspection could be the collection of data for detecting substances prohibited by the CWC; such known substances are listed in the Schedules. The prohibitions, however, would also extend to all toxic chemicals that would be inconsistent with the purposes of the CWC. The analytical procedures would have to be sophisticated enough to identify trace amounts not only of those chemicals listed in the Schedules, but also of unknown substances. This particular function could require the operation of one or more laboratory facilities equipped with sensitive instruments and having access to an extensive chemical data base. If the laboratory facilities are operated by the Organization, the analytical data base would be part of the information processing system of the Organization. If, on the other hand, the laboratories are operated by member states or other independent organizations on behalf of the Organization, the analytical data base would either be accessible by, or an extension of the central information processing system. The topic of analytical data bases is discussed in a separate paper submitted by the United States.

6.0 Structure of the Information Processing System

As standard operating procedure, official analysis of the data would have to be performed at the headquarters of the Organization, where the authoritative data base would reside, and where the Organization would evaluate the data, although preliminary evaluation of some data might also take place at the site. Therefore, some of the activities of the Organization involving continuous on-site presence of inspectors would require a distributed information processing system with the capability of being interconnected to other systems. Monitoring the destruction of stockpiles is a good example to discuss some of the possible design requirements. Some of the concepts presented in the following paragraphs could also be applicable to other types of facilities; destruction is used only for illustrative purposes.

The proper operation of the destruction facility would be the responsibility of the State Party. The facility would use appropriate monitoring and control equipment and procedures to ensure safe and effective operation. At the time of the initial declaration, the design of the facility, the equipment and procedures would be approved by the Organization. During destruction of stockpiles, the role of the Organization would be to verify that a) the facility is operated as intended, and b) the

items that were intended, by declaration, to be destroyed would indeed be destroyed.

The proper operation of the facility could easily be monitored by collecting data on the status of the facility equipment. Access to the control room data would provide the necessary information. It would probably be necessary for the inspectors to have some independent local data processing capability. These data and their associated processing system would constitute the local processing system of the Organization; it would provide the inspectors with needed information on a real-time basis. If the official data base is to reside at the headquarters of the Organization, the central information processing system must have access to the local system through some data transfer mechanism. The characteristics of the data link between the central and the local system would depend on the volume of data being transferred and on the frequency of such transfers.

Verification of the destruction of the declared items would require a) verification of the identity of the items being destroyed, and b) verification of the quantity. One approach to confirming the identity of the contents of a projectile or similar item would be to verify the operator's declaration by independent mean such as sampling and analysis or non-destructive evaluation. This approach would require the use of equipment maintained and operated by the Organization. Another approach could be to develop an operating profile of the facility and the destruction process for each type of agent being destroyed. In the latter approach the role of the Organization would be to monitor the operating profile of the plant in order to confirm the declaration of the chemical. The required information would be derived from a set of sensor data that would be collected and processed by the Organization. As in the former scenario, there would be a need for both a local and a central system linked by some type of data network.

Verification of the quantities of items being destroyed would be an accounting function. Since these destruction activities would be depleting the initially declared stock, there should be in place a real-time inventory control system. Daily information from the destruction facility could be transmitted to central headquarters for continuous update and analysis of the official stocks.

Similar kind of access from the field to central headquarters could be useful for other routine activities of the Organization. Routine monitoring of stockpiles might also require reference to past information. One approach might be for the inspectors on routine visits to the site to carry with them all data on computer disks or stored in portable computers. An alternative would be to have a small portable computer with a modem, as a portable terminal to establish direct communications with the central computing facility. Standardized reporting formats would allow the inspectors to interact directly with the central computers.

Interaction, in real-time, with the central processing system would offer the possibility of detecting discrepancies and resolving ambiguities at the time the data are collected. Transporting the data on disks or portable machines would interject a delay in the formal evaluation and it might necessitate additional visits to the site in case of discrepancies.

Another factor that would affect the choice between direct access from the field to central headquarters would be the concern for security of the information. The question has to be examined whether transporting the information from the field to central headquarters would allow the possibility of more or fewer potential security breaches than electronic transmission. Information protection would be an important factor in the routine monitoring of commercial facilities.

7.0 Conclusions

This paper has attempted to present some of the factors that would affect the design of the information processing system. Although a more thorough analysis of the requirements derived from the rolling text is needed, the emerging architecture would be a distributed system consisting of a central node linked to local processing nodes at remote sites. Some of the remote nodes, such as destruction facilities, would consist of local monitoring systems; others would only be portable computers, local instruments, or portable and transportable instruments. Other possible nodes might be the headquarters of the National Authorities.

The links between the central and the local nodes could be public or private. The particular implementation would depend on the volume of transmitted data, the frequency of transmissions, the security requirements and the need for timelines. For some applications there might not even be any need for electronic transmission of information; data would be transported by the people who collect them.

The system should have a hierarchical structure. To preserve the confidentiality of the information each facility should be linked only to central headquarters; if there would be a need to mix information from different facilities, this should only be done at the central processing node and with appropriate safeguards.

CONFERENCE ON DISARMAMENT

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Ad Hoc Committee on Chemical Weapons

UNITED STATES OF AMERICA

QUALITY ASSURANCE OF VERIFICATION ANALYTICAL LABORATORIES

Verification of prohibited activities under the multilateral convention to ban chemical weapons will rest largely on laboratory findings and analytical results obtained from samples acquired during inspection. To insure that these critical activities have the required credibility, accuracy and reliability, a quality assurance program must be developed for each laboratory accredited to perform sample analysis. The system must produce data that is scientifically sound, thoroughly documented and legally defensible.

There are many types of quality assurance techniques used around the world in the analytical community but there is little consistency among them, partly because the analytical needs and the laboratories themselves vary greatly. The "Round Robins" which have been completed and which are in progress are an attempt to measure these inconsistencies. Those completed to date indicate the diversity among the laboratories. The following is an attempt to develop some principles which could lead to more consistent results among analytical laboratories.

The first step is to achieve some agreement on definitions of critical importance. Quality Assurance can be defined as a system of activities which provide the producer of a product or service the assurance that it meets defined standards of quality. It consists of two separate but related activities, quality control and quality assessment. Quality Control is the overall system of activities which control the quality of a product or service so that it meets the needs of the users. The aim is to provide quality that is satisfactory, adequate, dependable and economic. Quality Assessment, the second component of Quality Assurance, is the overall system of activities which provides assurance that the quality control activities are done effectively. It involves a continuing evaluation of performance of the production system and the quality of the products produced. (1)

(1) Taylor, J.K. Quality Assurance of Chemical Measurements (Lewis Publishers, Inc., Chelsea, MI, 1987) P. 2.

Figure 1 illustrates the relationship of these three elements and the basic ingredients of a laboratory quality assurance (QA) and a laboratory quality control (QC) program, required for verification of compliance. Table 1 presents some of the basic components of the quality control segment of a typical laboratory quality program. Table 2 presents some of the basic components of a typical laboratory assessment program. These two activities form the backbone of a laboratory quality assurance program.

The other component that is critical to quality assurance is a validated sample handling program, since the analysis can be no better than the sample acquisition and handling procedures. A sample management information system is required to maintain a valid chain of custody and validate the analysis reported. Such a system can be manual through bound, controlled manuals or automated through local secured computers. Table 3 provides the basic components of a laboratory sample control and reporting system.

There are three basic types of quality assurance that should be considered in order to assure that the laboratory data developed is high quality with accurate results reported. Some activities are specific to the instrumentation, some to the analytical method and others to the general laboratory quality assurance requirements. Each of these types of quality assurance is essential to assure the generation of accurate, precise and reliable data.

The first type of QA relates directly to the performance of the instrumentation being used for the particular analysis. It includes basic operations such as the proper tuning of the instrument to make sure that it is monitoring the correct wavelength or the correct ion mass, as well as the performance of routine maintenance operations that assure optimum sensitivity, stability and reproducibility of the response. Typically this type of QA operation is carried out when the instrument is installed or when major changes are made, such as replacement of parts. It is also carried out on a routine basis at prescribed times (preventative maintenance).

Analytical method QA is related to the performance of the specific method. This includes items such as verification that the solvents, reagents and glassware are free of target analyte contamination and that the method can accurately and precisely determine the levels of the target analyte. Other items include verification of chromatographic resolution, detector sensitivity and stability of the response. This type of QA is usually defined in the method and is carried out prior to sample analysis as well as on a routine basis at prescribed times. Method QA is usually well defined in the written procedure. A properly designed method

should include a clear description of what measures are required and what the acceptance criteria are for each activity.

Laboratory QA is typically related to staff training and certification. It includes operations that ensure that the laboratory staff is initially trained to carry out specific analyses and that they receive additional training whenever changes or updates to methods occur. Laboratory QA can also encompass operations that are above and beyond the manufacturers recommendations or the analytical method. This may include the analysis of performance evaluation standards, the analysis of split samples or the analysis of additional blank samples.

Detailed QA procedures can be developed for each instrument or method employed in the analysis procedure. As an example, the majority of analyses employ some form of chromatography to separate the analyte(s) from other materials in the sample. After separation, a detector provides a response to the target analyte(s) and it is hoped, little or no response to the other compounds in the sample being analyzed. For chromatography, critical issues can be identified and those activities can be further explored to enhance the QC measures. Retention time monitoring, detector response, continuing calibration, evaluation of matrix interferences and computer data acquisition are such critical issues and are indicative of the procedures employed by experienced analysts in the generation of high quality analysis data.

Conclusion: Quality assurance/quality control must be an integral part of every laboratory that generates data relevant to the verification process. Every analytical method requires a minimum level of QA/QC and the laboratory must institute additional QA/AC elements in order to provide the highest levels of credibility. This paper introduces the analytical community to the variety of elements which are available. Each laboratory must meet internationally agreed minimum elements with some laboratories employing more than the minimum level. Furthermore, each individual program may require that even more measures will be adopted to maintain the level of credibility, accuracy and reliability which will be required to insure that verification of the chemical weapons ban is uniformly enforced in a credible manner.

Note: This paper is adapted from an article: "Basic ingredients for assuring quality in the analytical laboratory: Some precautions in the analysis of organic materials; by Deroos, F.L., Bicking, M.K.L. and Nosek, Jr. W.J., American Environmental Laboratory, V2, No. 4, October 1990.

Figure 1: Laboratory Quality Assurance Program Elements

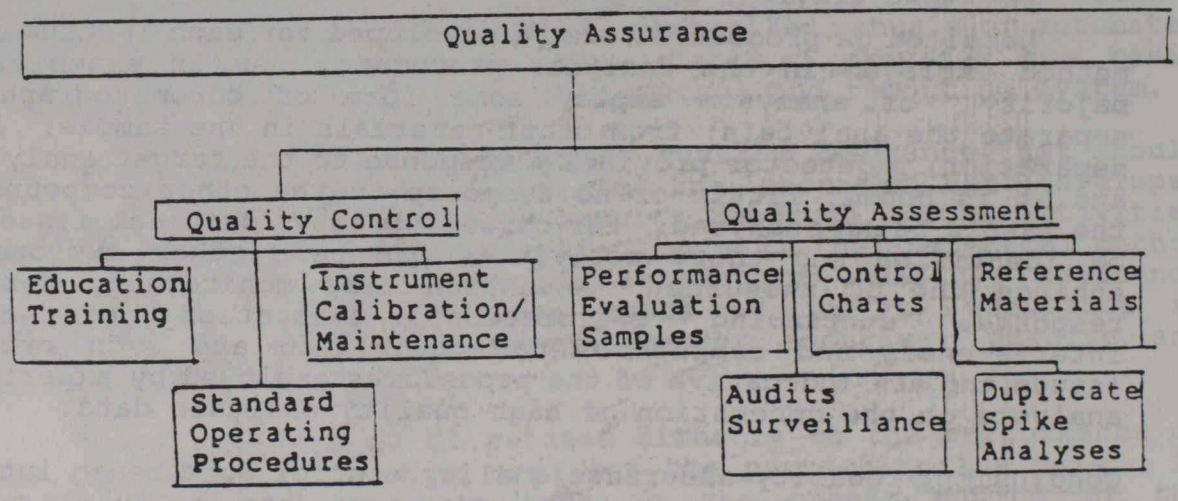

Table 1: Basic Components of a Laboratory Quality Control Program

- Documented QA/QC plan and protocols
- Trained and qualified analysts and support personnel
- Standard operating procedures
- Instrument maintenance and calibration system
- Reliable instruments and equipment
- Documentation of analytical and operational information
- Proper sampling and sample tracking techniques
- Appropriate methodology usage
- Good laboratory practices

Table 2: Basic Components of a Laboratory Quality Assessment Program

- Performance evaluation samples
- Audits and surveillance
- Duplicate, spike, surrogate, blind analyses
- Standard reference materials
- Control Charts
- Round Robin Collaborative testing
- Statistical analysis of data
- Corrective actions

Table 3: Laboratory Sample Control System
Basic Components

- Chain of custody procedures
  - Acquisition to laboratory
  - Internal to laboratory
- Verification of labeling of sample container
- Assignment of unique laboratory code number
- Assignment of proper test methodology
- Proper sample preservation
- Regulated environmentally controlled storage
- Controlled access to samples
- Monitoring of sample holding times
- Internal paperwork/computer files containing sample information
- Timely transmittal of samples and information to analysts
- Proper logging of analytical results into sample log
- Timely, accurate reporting of analytical results

CONFERENCE ON DISARMAMENT

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Ad Hoc Committee on Chemical Weapons

EGYPT, ETHIOPIA, INDONESIA, ISLAMIC REPUBLIC OF IRAN,
KENYA, NIGERIA, PAKISTAN AND YUGOSLAVIA

Verification of the Chemical Industry under Article VI and its Annexes

From the very outset of the negotiations to conclude a Chemical Weapons Convention, the importance of establishing a verification system of chemical industry received special attention. The results of the negotiations on this subject are now reflected in Article VI and its Annexes of the Rolling Text.

Table (1) summarizes the verification régime accordingly:

The national trial inspections conducted by a number of countries according to the present system have shed light on the advantages as well as possible lacunas of this régime. The results of these inspections to a large extent indicate that Article VI and its Annexes can meet the objectives set by the Convention with some modifications and improvements.

The negotiations and discussions in the past two years also contributed to the identification of certain operational problems, among which are "the financial burden of the régime" and "inadequate coverage of all capable facilities". Various suggestions have been tabled to address these problems.

This paper, while basing its approach on the principles embodied in the Rolling Text, has tried to offer additional measures to bridge the shortcomings and lacunas.

1. The basic elements of the proposal

1.1 The objective of the Convention is prohibition of the development, production, stockpiling, acquiring and use of chemical weapons;

1.2 The verification system of the chemical weapons Convention should be feasible, effective and reliable. This system should take into account the present industrial realities and the future rapid development of chemical industry;

1.3 As most of Schedule 1 chemicals are produced through Schedule 2 chemicals, the effective verification of Schedule 2 chemicals is the key to attain the objectives of the Convention;

1.4 The aim of the verification of chemical industry is not to look for violators but rather to verify compliance;

1.5 Such a system will constitute an important instrument for confidence building and universal adherence.

2. Detailed outline of the proposal

The verification of chemical industry is divided into two categories:

(i) Schedule 2 chemical facilities
(ii) Schedule 3 chemical facilities and capable facilities.

2.1 Verification of Schedule 2 chemical facilities

The facilities which produce, process or consume chemicals listed in Schedule 2 over threshold declared under Annex 2, will be subjected to initial inspection and accordingly a facility agreement will be concluded to govern the conduct of the inspection.

According to certain criteria including the duration of the initial inspection, a report will be provided to the Technical Secretariat. Based upon this report and taking into account elements such as actual production in the preceding three years and production capacity, toxicity of the chemicals, multi-purpose potential of the facility, etc., the Technical Secretariat will decide whether the facility should be subjected to:

- Systematic Routine Inspection (SRI);

- Random Selective Inspection (RSI).

Based on the risk assessment to the objectives of the Convention, the facilities subjected to RSI shall be classified in three baskets and their inspections shall be carried out in ratio of (3:6, 2:6 and 1:6). These ratios will be in respect of the most, less and the least dangerous to the Convention.

(The Conference of the States Parties) (Executive Council) shall determine the annual number of inspections in each category for the coming year. The optimization of the number of inspections will be made accordingly.

2.2 Declaration

In parallel to the declarations under Article VI of the Convention, the States Parties should also declare any changes in the structure or layout of the facilities previously declared, and in case the assessment indicated such changes are considerable resulting in an increase in capacity by ... per cent the initial inspection will be repeated.

2.3 Verification of Schedule 3 chemical facilities and capable facilities

To establish a verification régime of the facilities which produce chemicals, listed in Schedule 3 as well as capable facilities, four important elements are taken into consideration:

1. Assessment of activities based on statistical surveillance of such facilities is indicative and not conclusive;

2. Abundance of such facilities worldwide;

3. Deterrence against any possible non-compliance;

4. Avoidance of hampering the production process.

The number of Schedule 3 chemical facilities as well as capable facilities is estimated to stand at the minimum of 10,000 now and will be increased by 15,000 by the turn of the century. They also enjoy a special and significant place in the overall industrial production. In addition, the finite financial and budget resources of the Organization are a determining factor. These elements justify and require the establishment of a special mechanism of general surveillance of data and Random Selective Visits (RSV) to verify the declared data through evaluating the records on-site and visits to the plants, etc.

The random selection of facilities declared under Annex III to Article VI shall be conducted by the Technical Secretariat through appropriate mechanism including the use of especially designed computer software.

The same classification as indicated in RSI can also apply to RSV.

The general view of the new proposal can be summarized as shown in table (2).

Definition of Capable Facility

Capable facility is any facility which:

1. Is capable of producing organic chemicals containing the elements of phosphorous, fluorine, and sulfur or those involving the processes of phosphorylation, fluorination or sulfurilation identical to those chemicals included in Schedule 1 as well as Schedule 2 chemicals;

2. Has the same process equipment and machineries as for the Schedule 2 chemicals;

3. Has the same process equipment layout as for production of Schedule 2 chemicals.

Table (1): The verification régime according to CD/1046

Schedules	SCH.3 (over threshold)	SCH.2 (over threshold)	SCH.1 small scale facility
Type of chemicals	raw chemicals	precursors and relevant chemicals	declared toxic chemical
Nature of facility	civil industry	civil industry	civil industry and university pilot plan
Activities under CWC	permitted	permitted	restricted
Verification régime	data reporting	systematic routine inspection	systematic on-site monitoring
Facility agreement	not required	required	required

Table (2): The verification régime according to the proposed system

Schedules	SCH. 3 and capable facilities		SCH. 2	SCH. 1
	SCH. 3 (over threshold)	capable facilities (over threshold)	(over threshold)	small scale facility
Type of chemicals	relevant raw chemicals	irrelevant chemicals	precursors relevant chemicals	declared toxic chemicals
Nature of industry	civil industry	civil industry	civil industry	civil facility industry or university pilot plant
Activities under CWC	permitted	permitted	permitted	restricted
Verification régime	1. data reporting and 2. random selective visits (RSV)		1. data reporting and 2. initial inspection for all 3. systematic routine (SRI) or 4. random selective inspection	systematic on-site monitoring
Facility agreement	not required		required	required

CONFERENCE ON DISARMAMENT

CD/CW/WP.348/Corr.1
30 July 1990

ARABIC, CHINESE and
ENGLISH ONLY

Ad Hoc Committee on Chemical Weapons

EGYPT, ETHIOPIA, INDONESIA, ISLAMIC REPUBLIC OF IRAN
KENYA, NIGERIA, PAKISTAN AND YUGOSLAVIA

Verification of the Chemical Industry under Article VI and its Annexes

Corrigendum

On page 3, paragraph 2.3, sub-paragraph 2, third line:

"...increased by 15,000" should read "...increased to 15,000".

CONFERENCE ON DISARMAMENT

CD/CW/WP.349
12 July 1991

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Ad Hoc Committee on Chemical Weapons

REPORT OF THE TECHNICAL GROUP ON ANALYTICAL DATA BASE AND LABORATORIES

The Ad Hoc Committee on Chemical Weapons decided on 27 March 1991 to establish the Technical Group on Analytical Data Base and Laboratories, as a successor to the Technical Group on Instrumentation, and appointed Dr. Marjatta Rautio of Finland as the chairman of this expert group. The group held 9 meetings from 17 to 21 June 1991.

The group discussed extensively issues related to
- analytical data base, its sources and the types and quality of data to be included;
- accreditation of designated laboratories, tasks of these laboratories and options for the laboratory network.

Background papers on different aspects of data bases and verification laboratories were submitted by the delegations of Canada and the United Kingdom (jointly), Finland and the Netherlands (jointly) and the United States. Australia introduced a draft paper on on-site chemical analysis of families of scheduled chemicals. In addition material on quality assurance for analytical chemical laboratories was distributed. A list of all background material is included as an annex to this report.

The results of the discussions are presented in this report. Repetition of views presented in the reports of the Technical Group on Instrumentation (CD/CW/WP.272 and CD/CW/WP.306) has been avoided. A detailed analysis of the use of different analytical techniques for the verification of the Chemical Weapons Convention, which formed the background for the discussions in the group, can be found in those reports.

This report has been divided into three parts. The first part deals with the analytical data base, the second part with the accreditation of laboratories, and the third part contains a summary of the findings and recommendations of the group. All parts were approved in the last meeting of the group on 21 June 1991.

PART I. ANALYTICAL DATA BASE

1. The information base of the Technical Secretariat will be broad in scope as all data and information from declarations, inspected sites, inspection protocols and all other relevant data requiring easy processing, will be included in it. The data base of the analytical functions will form part of it.

2. In order to distinguish the difference between the nature of the data and information which is required in the analytical work, the discussion is divided into two sections: Compound Identification Data Base, and Knowledge Base (Procedures Manual).

1. Compound Identification Data Base

3. The Compound Identification Data Base should be structured to allow easy search and combination of data in a variety of ways. It will contain data needed for the identification of treaty-related chemicals with different types of analytical instrumentation.

4. The Group agreed that the data base should, in principle, include and be restricted to data on all scheduled chemicals and those other chemicals which are covered by the Convention. Of Schedule 1 chemicals, also their precursors and by-products as well as the typical stable degradation products, impurities, artefacts formed during sample preparation, analytical derivatives, and decontamination products should be included. Data on stable by-products and degradation products of Schedule 2B and Schedule 3 (dual-purpose) chemicals should also be included. In practice such a complete data base will not be within reach in the near future. Therefore certain priorities have to be established. One possible procedure is described in the following paragraphs.

5. The most immediate inclusion should be the existing data on scheduled chemicals and their related chemicals. This data is available from several sources including research laboratories and commercially available data bases. On some of the chemicals there will probably be an abundance of different data while on some there may also be conflicting data. In order to assist the Technical Secretariat to decide the appropriate entries to the CWC data base, preliminary work should commence before the entry into force of the Convention.

6. In order to chart the present availability of relevant data, it was proposed that the laboratories compile a list of chemicals on which they possess the necessary information (hard copy or digital form), and of those chemicals on which in their view data should be available by 31 December 1991. It was also proposed to include the electron ionization mass spectra together with mass/intensity tables of the Schedule 1 chemicals that are mentioned by name in the present Rolling Text. The Netherlands has already presented a list of chemicals for which it has spectral and chromatographic data in digital form (CD/CW/WP.328).

7. After the inclusion of the existing data and data which is considered urgent by various countries, the next step would be the inclusion of additional data on families in the Schedule 1.

8. Further additions to the data base of other scheduled chemicals should continue by the Technical Secretariat. All chemicals which are declared under the Convention as chemical weapons should be included immediately. Any chemicals which are added to schedules after the entry into force of the Convention should also be included as soon as possible.

1.1. Compound Data

9. For each compound the following data should be included, as available, after technical review:

- chemical names (IUPAC, CAS), common names, CAS-numbers, molecular formula, structural formula;

- physical constants at given purity, indicating the important differences between pure and technical grade chemicals: physical state, colour, boiling point, melting point, diffusion coefficient, liquid density, vapour pressure, specific heat, surface tension, volatility, viscosity, solubility and partition coefficient;

- chemical properties: chemical half life, hydrolysis rate;

- toxicity data: species, application method, dose unit, dose value

- source(s) of data for each parameter separately;

10. The Group considered it important to have data on typical impurities and by-products formed during the different production routes of schedule 1 chemicals. Sensitive data should, however, be protected from unauthorized access.

1.2. Identification data

11. The Group discussed the data base requirements for the techniques identified in the first report of the Technical Group on Instrumentation (CD/CW/WP.272). These techniques are: gas chromatography (GC), high performance liquid chromatography (HPLC), mass spectrometry (MS), infrared spectrometry (IR), nuclear magnetic resonance spectrometry (NMR), and the non-destructive measument technology. In addition to these, thin-layer chromatography (TLC), ion mobility spectrometry (IMS) and super-critical fluid chromatography (SFC) were discussed.

12. It is recommended that existing data be submitted by laboratories. Data submitted should include details of recording conditions, sample purity etc. in order to facilitate critical evaluation before inclusion in the data base. When evaluating the data, expert groups could use quality indices published by various scientific societies.

13. In order to facilitate evaluation of the available data in the

future, it was recommended that recording conditions and reporting be harmonized.

14. The Group discussed the various parameters that should be included for each of the techniques, to enable different analytical tasks to be carried out successfully in all sites requiring on-site analysis. Although the resulting data base will be fairly extensive, its storage should not pose any technical problems. In order to protect proprietary or sensitive information during inspections certain precautions have to be taken. These include the use on-site of "blinded" instruments which can only identify chemicals (scheduled and related chemicals) covered by the CWC. Furthermore, during inspections which do not require off-site analysis, the instruments should be cleared of all data collected during the analyses before leaving the site. Any relevant data could, however, be stored at the facility.

15. The authoritative analytical data base will form part of the Technical Secretariat's general purpose data base. The on-site instruments utilized by the inspectors, and the off-site instruments of the accredited and national laboratories, will be provided with the appropriate sections of the data base. The data transfers will be facilitated by the standards (e.g. JCAMP.DX) compatible with data processing systems and applied by all instrument manufacturers.

16. The Group emphasized that the data bases alone do not guarantee reliable analytical results. The key elements in this respect are always the skilled operators of the instruments and the analysts interpreting the analytical results.

Gas chromatography (GC)

17. Primary use of GC is separation, for further compound identification by other detectors. GC can be used as a confirmation method and for quantification. When GC is used for sample introduction to various spectrometers, it would be beneficial to use the same types of columns (e.g. SE-54) to enable the use of prerecorded retention data.

18. For the use of GC as an independent monitoring method, the retention indices should be recorded using two different columns. However, the Group identified the difficulty in specifying a particular pair of columns, as improved stationary phases may well be developed. At present, SE-54 and OV-1701 were proposed. In addition to the stationary phases, column length, internal diameter, film thickness and temperature programming (e.g. 40°(1 min) - 10°/min - 280°(10 min)) must be recommended later.

High performance liquid chromatography (HPLC)

19. The present reproducibility of HPLC column packings does not allow the retention index monitoring to be used for screening. On the other hand, HPLC or ion chromatography (IC) is suitable for analysis of specific ionic species (e.g. methylphosphonic acids) in aqueous solutions. For preliminary identification, authentic

reference standards have to used. Accordingly no prerecorded parameters can be stored in a database.

Mass spectrometry (MS)

20. MS is the most widely used spectrometric technique in verification analysis. The spectra depend to some extent on the type of instrument and recording conditions. For the evaluation of the quality, it is essential to include details of the recording conditions and the purity of samples with the spectra. Quality indices have been introduced to aid the assessment (Terwillinger et al., Biomed. and Environ. Mass Spectrom. 14, 263-270 (1987)). There are no universally accepted recording conditions. However, international recommendations have been made, and laboratories which do not have extensive experience might be adviced to use conditions described in literature (e.g. Dillard et al. Org. Mass Spectrom., vol 16, pp. 48-49, 1981 or Blue Book, F.1, 1990).

21. In general, the most valuable information for mass spectrometrist is the positive electron ionization spectra recorded using 70 eV. Positive chemical ionization spectra recorded using isobutane, methane or ammonia as reactant gases were also considered useful. For those compounds for which negative ion spectra are more sensitive than positive ion spectra, these spectra should be included. The high resolution MS and MS/MS data, while not considered essential, provide additional information for the analysts, and, when available, should be included in the data base.

Infrared spectrometry (IR)

22. The Coblentz Society has published specifications for the recording of infrared spectra. Specifications are described for gas-phase spectra and for condensed-phase spectra (Applied Spectroscopy, vol. 44, pp. 211-215 (1990) and Analytical Chemistry, vol. 47, pp. 945A-952A (1975), respectively). It was noted that the new deposition technique produces spectra from sub-nanogram quantities of chemicals and allows spectral comparison with those in the condensed-phase libraries. Detailed recording conditions are included in the specifications and should follow the spectra submitted for the data base. Published quality criteria are available to aid selection of spectra.

23. There are numerous spectra of relevant chemicals recorded well before any specifications were established. The Group recommends that these spectra be included in the data base when new data have not been recorded.

24. In the central data base the spectra should be stored as interferograms, when possible, to allow later reprocessing of spectra.

Nuclear Magnetic Resonance Spectrometry (NMR)

25. The NMR database should include ^{1}H, ^{13}C, ^{31}P, and ^{19}F spectra. The spectra should be recorded in different deuterated solvents.

The data base should include chemical shifts and coupling constants and also other important spectral parameters (e.g. line widths). For the central data base, the free induction decay (FID) signal should be stored, when available, to allow reprocessing of spectra. The Group considered it essential to define the accepted reference standards for each nucleus. All recording parameters should be supplied with the spectra.

Other techniques

26. The Group discussed the usefulness and possible data base requirements of thin-layer chromatography, ion mobility spectrometry and super-critical fluid chromatography.

Thin-layer chromatography (TLC). This technique was considered as potentially useful as a rapid screening method. The standardized Rf-values of the chemicals can be stored in the data base.

Ion mobility spectrometry (IMS). IMS systems are today used for CW agent detectors (e.g. CAM) but can be modified for verification purposes in the future to be used e.g. as a rapid screening method. IMS can be readily modified to detect many chemicals under the Convention.

Super-critical fluid chromatography (SFC). This method was considered potentially useful in the future when the reproducibility problems have been solved.

Non-destructive measurement technology (NDE)

27. **Radiographic methods.** These give visual representations of the inner contents of various munitions, which may limit its application as it may reveal sensitive information. Liquid fills which might indicate a chemical munition can be easily confirmed by radiography. The data obtained are not appropriate for a reference data base.

28. **Acoustic methods.** Acoustic methods, so far in early research stages, would appear to offer potential in verification technology. "Fingerprinting" of authentic items can be used to confirm identity of like items. Since acoustic responses are affected by many parameters, the device needs "calibration" at each time of use with each munition/agent combination. A library of standard acoustic spectra may not be appropriate. However, a library of measured spectra could well be useful in the field.

29. **Neutron methods.** Feasibility studies have demonstrated that elemental composition of munitions fills can be identified by neutron interrogation. As the device, as envisaged, carries out 'ab initio' measurements, there is no need for a standards data base. A data base of measurements could prove useful.

2. Knowledge Base (Procedures Manual)

30. Standard operating procedures should be developed for e.g.

a) sampling, preservation of samples, packaging, labeling, guaranteeing of sample integrity, transportation, and storing in laboratories before sample preparation for analysis;

b) preparation of samples for analysis: adsorbents in sampling kits, environmental matrices (e.g. soil, water, air, vegetation), materials (e.g. concrete, rubber, paint, activated carbon in respirator canisters, protective clothing), munitions fragments, industry samples, biological samples, or other types of samples;

c) analytical methods for each technique including use and calibration of instruments, collection of data, quality control;

31. Other technical support information, e.g. records of agreed instruments and their specifications, should also be included in the knowledge base.

32. The Technical Secretariat shall establish and maintain, for the use of any requesting State Party, a data bank containing freely available information concerning various means of protection against chemical weapons, as well as such information as may be provided by States Parties.

33. The analytical data base would be part of the information processing system established and operated by the Technical Secretariat; it would be a subset of a much larger data base which needs to be specified. The contents of the larger data base should be selected to help the Technical Secretariat perform its verification functions which are dynamic. In this context, data bases would be evolving with time; consequently, it would be difficult, if not counterproductive, to try specifying their contents, a priori.

34. A more productive approach would be to identify the information processing requirements for an optimal implementation of the verification functions of the CWC. From this point of view, the data submitted to the Technical Secretariat by each State Party would be one of the inputs to the information processing system; these data, which would form the reported data set, would consist of the initial declarations and the recurring annual declarations. The reported data set may consist of a number of subsets such as site and product data. Another input would be the data collected by the Technical Secretariat using inspectors and instruments. Yet another set would be the analytical reference data.

35. These data would form the collected data set, which may contain similar or different types of data from those of the reported data set. The collected data could be viewed as measurements performed at times different from those of the reported data. The function of the information processing system would be to process these two data sets, in order to generate output data which would contain the necessary and sufficient information to allow evaluation of compliance with the CWC.

36. Posing the problem in the context of information processing would allow technical experts to identify a system architecture

both in the context of physical nodes such as facilities, National Authority and the Technical Secretariat, as well as information processing modules such as identification of chemical compounds, accounting etc. Other functions might involve analysis procedures tailored to satisfy only the requirements of the convention, with particular emphasis on the maximization of the information need for verification, while minimizing any other information which might compromise national interests or business confidentiality.

37. Since the verification functions require knowledge from many disciplines, the issue of information processing needs to be addressed in a series of meetings and by groups of experts from many disciplines, with the purpose of developing recommendations to the CD on the technical feasibility of implementing specific components such as types of instruments, computing requirements, operation of the information processing system, data evaluation procedures etc. It would be useful to provide cost estimates for the implementation of the recommendations. In addition to having meetings involving specialists from only one area such as chemistry, other meetings should involve specialists from many areas such as chemistry, chemical engineering, computing and communications, systems theory and process control to ensure a comprehensive treatment of the issues. These expanded meetings would allow for interaction among the various experts whose knowledge would be essential for the development of the information processing system. Therefore, there is a need to establish an agenda for examining various components of the information processing system and to identify the areas of the required expertise.

PART II. ACCREDITATION OF DESIGNATED LABORATORIES

3. Tasks of the laboratories

38. In the context with the accreditation of designated laboratories, the Group discussed the different tasks the laboratories are required to perform under the Chemical Weapons Convention, and how these tasks could be distributed between the Technical Secretariat, its laboratory and accredited laboratories. The following tasks were identified:

- Verification analysis
- Analytical methods development
- Other methods development
- Recording of reference data
- Organizing, certifying and updating of data bases
- Synthesis and certification of reference compounds
- Preparation and distribution of analytical standards
- Toxicological data collection and determination
- Certification of analytical methods
- Quality control/quality assurance
- Handling of authentic samples
- Training of inspectors
- Accreditation of laboratories

- Technical support for the Organization
- Maintenance and storage of verification equipment and protective means

Verification analysis

39. Inspectors may encounter situations when the available instrumentation is not sufficient to give unambiguous results, and in some situations the inspectors may decide that further chemical analysis in off-site laboratories may be required. This is especially obvious in situations when the on-site analyses would indicate a possibility of non-compliance with the Convention.

40. The draft Convention (CD/1046) requires in cases of off-site analyses independent results from at least two laboratories. If their results are contradictory a third laboratory should be used for confirmatory analysis.

41. The analytical tasks that the off-site laboratories would be required to do are:

- Unambiguous identification of scheduled compounds
- Structure elucidation of possible novel agents
- Quantification

42. Identification of a scheduled chemical is considered unambiguous when two independent spectrometric (MS, IR, NMR) techniques produce corroborative results. Thus at least two of these techniques must be available at laboratories accredited for verification analysis.

43. While recognizing the fact that the MS technique may be the only technique sensitive enough for identification, the Group considered important that an accredited laboratory is able to use another technique, and if possible, all three spectrometric techniques. This may be especially important for structure elucidation.

44. The Group discussed the role of other laboratories in verification analysis, and concluded that while they may analyse the duplicates of samples collected during inspections which the National Authority has the right to receive, these laboratories do not form part of the international laboratory network of the CWC Organization.

Analytical methods development

45. In order to ensure that the Organization has at all times at its disposal the best available means for verification, it is an absolute necessity to continue to develop these analytical methods. It is evident that the verification laboratories should improve the existing methods and develop new ones.

46. Although the methods development is in essence a voluntary activity for the laboratories, there may be cases when the

Technical Secretariat may request work to be done in order to focus the work on a particular problem encountered during verification activities.

47. The Technical Secretariat may contract any laboratory to develop methods.

Other methods development

48. Other methods development, in support of the analytical requirements does not require analytical facilities (sampling, packaging, coding etc.), can be assigned to non-analytical institutions.

Recording of reference data

49. Recording of reference data for the analytical data base is a voluntary function of accredited laboratories. Laboratories can be accredited for this task alone, and even for a single technique.

50. Directions from the Technical Secretariat might be needed on both the chemicals and analytical recording techniques to cover all data base needs.

Organizing, certifying and updating of reference data base

51. All reference data that will be included in the data base have to be validated before their insertion. This requires that the quality of the data submitted to the Technical Secretariat be confirmed by 1-3 accredited laboratories. After the necessary confirmations, the Technical Secretariat will certify the data and update the data base.

Synthesis and certification of reference compounds

52. The verification laboratories will need several types of reference compounds for comparison of analytical data for identification purposes. For this purpose the Technical Secretariat may accredit synthesis laboratories. These laboratories have to document the identity and purity of the compound, which then will be validated by 1-3 accredited analytical laboratories. Complete physical and chemical data should be obtained of new compounds. After the validation the Technical Secretariat will certify the compounds.

53. The Group noted that the number of different reference compounds that will eventually be needed may be large due to the large number of members in the schedule 1 and 2 families.

Preparation and distribution of analytical standards

54. The laboratory of the Technical Secretariat will prepare analytical standards for the use of the accredited laboratories and distribute them.

Toxicological data collection

55. In case relevant toxicological data are not available from other sources, the Technical Secretariat may have to contract a toxicological laboratory to evaluate toxic properties of chemicals, especially the ones that may be under consideration for addition to the schedules.

Certification of analytical methods

56. New methods developed by any laboratory may be validated by a number of accredited laboratories by international collaborative tests arranged by the Technical Secretariat. If a method proves to be acceptable, the Technical Secretariat will certify it.

Quality control and quality assurance

57. Verification may rest on laboratory findings and analytical results obtained from samples acquired during inspections. To ensure that these critical activities have the required credibility, accuracy and reliability, a quality assurance programme must be developed for each accredited laboratory. The system must produce data that is scientifically sound and thoroughly documented.

58. There are certain basic types of quality assurance techniques that should be considered in order to assure that the laboratory data developed is high quality with accurate results reported. Some activities are specific to the instrumentation, some to the analytical method, and others to the general laboratory quality assurance requirements. Each of these types of quality assurance is essential to assure the generation of accurate, precise and reliable data.

59. It will be the responsibility of the Technical Secretariat to assure the reliability of the accredited laboratory network and on-site analytical activities. Quality assessment must be an integral part of the accreditation procedure for laboratories and be carried out continuously thereafter. The main assessment methods are laboratory audits and proficiency tests.

60. Quality assurance standards are required for both on-site and off-site analysis.

61. The quality assurance activities for the network of accredited laboratories should be carried out by a special division in the Technical Secretariat. This division should also supervise the quality assurance programmes in the Technical Secretariat's laboratory.

62. The Group recommends that work should be started as soon as possible to create the quality assurance programmes and the plans for their implementation.

Handling of authentic samples

63. The samples, which the inspection team dispatches to off-site laboratories, must be handled according to a specified procedure in which their identity is concealed from the laboratories which analyse them. At the same time the integrity of the samples has to be secured. This involves several stages including transport to the Technical Secretariat utilizing approved means, splitting of samples, preparation of necessary control samples, recoding and record keeping of all samples, transport to 2-3 verification laboratories, decoding of the results, and informing the relevant section of the Technical Secretariat of the results.

Training of inspectors in analytical chemical tasks

64. The Group emphasized the importance of training, both in sampling and analytical tasks, required during on-site inspections. In sampling, both the strategy and methods have to be covered. In analytical methods, both the technique and operating procedures of instruments are important.

Accreditation of laboratories

65. If the laboratory network has to be operational when the Convention enters into force, the accreditation process for laboratories should be started by the Preparatory Commission.

66. The accreditation procedure may comprise the following steps:

- Sending a detailed questionnaire to the laboratory seeking accreditation.
- Inspection of the laboratory by a group of assessors.
- Analysis by the laboratory of proficiency samples.
- Evaluation of the quality assurance programme (personnel, facilities, instruments, procedures, safety rules) of the laboratory.

67. Laboratories can be accredited for all tasks or for selected purposes. A specific time period should be set for the accreditation (e.g. 3-5 years).

Technical support for the Organization

68. To support the Organization, accredited laboratories may be involved in developing new equipment. Equipment may also be developed commercially or under contracts financed by the Organization or by States Parties.

Maintenance and storage of verification equipment and protective means

69. The maintenance and storage of verification equipment and protective means would be the responsibility of the Technical Secretariat, but can be assigned under contract to accredited laboratories.

4. Options for the structure of the laboratory network

70. There are three basic approaches for the structure of the laboratory network of the Technical Secretariat:

- A centralized structure, with a central, fully equipped analytical laboratory and possibly a synthetic laboratory, which will perform as many tasks as possible.

- A decentralized structure, consisting of a group of accredited laboratories not belonging to the Technical Secretariat. There would not be a central analytical laboratory operated by the Technical Secretariat, however, the Technical Secretariat would have a modest laboratory unit.

- An integrated approach, under which the Technical Secretariat will have fully equipped laboratories (central and regional) to perform all analytical tasks needed to carry out its analytical verification mandate, issued by the Executive Council, on a self-sufficient basis. The accredited laboratories would also carry out functions approved by the Technical Secretariat.

There are several intermediate options between these three basic approaches.

71. If the Organization were to have regional offices, a laboratory unit could be connected to each of them.

72. The minimum number of accredited analytical verification laboratories would have to be three. The Group felt that there cannot be a limit on the number of laboratories in the network. All laboratories applying for accreditation and fulfilling the requirements should be accredited. States Parties may wish to cooperate in establishing joint laboratories.

TABLE 1. OFF-SITE LABORATORY FUNCTIONS AND THEIR DISTRIBUTION

FUNCTIONS	TS	TS LAB N-A incl.REG	TS LAB A	ACC LAB	OTH LAB
Verification analysis			x	x	
Analytical methods development			x	x	x
Other methods development	x	x	x	x	x
Recording of reference data			x	x	x
Organization/update of data base	x				
Synthesis of reference compounds				x	x
Preparation of analytical standards		x	x		
Validation of analytical methods			x	x	
Certification of analytical methods	x				
Validation of data			x	x	
Certification of data base	x				
Validation of reference compounds			x	x	
Certification of reference compounds	x				
Distribution of analytical standards	x				
Toxicological data collection				x	x
Toxicological data determination				x	
Responsibility for quality assurance	x				
Handling of authentic samples		x	x		
Preparation of control samples		x	x		
Technical training of inspectors	x	x	x	x	
Accreditation of laboratories	x				
Technical support for the Organization		x	x	x	x
Maintenance and storage of verification equipment		x	x	x	
Maintenance and storage of protective means		x	x	x	

- - - -

TS = Technical Secretariat
TS LAB N-A = Non-analytical laboratories of the TS
TS LAB A = Analytical laboratories of the TS
ACC LAB = Accredited laboratories
OTH LAB = Other laboratories

- TS LAB N-A and TS LAB A are separate options for network approach, the first representing a decentralized approach and the latter a centralized approach. In the integrated approach all TS-laboratories fall under TS LAB A.

- Other laboratories do not form part of the CWC Organization's laboratory network if they have not been accredited, but there are certain functions in which they can give an input by submitting proposals for the certification of the TS. These functions are described in the OTH LAB column. The exact role other laboratories may have under the CWC has to be elaborated further when the whole structure of the network will be determined.

PART III. FINDINGS AND RECOMMENDATIONS

74. Findings:

- Informal lists of chemicals pertinent to the Convention are being compiled now, to be followed by evaluation of the EI mass spectra of selected schedule 1 chemicals.

- The instruments and related data parameters to be included have been identified by this group.

- Critical items to be included in procedures manuals have been identified.

- The functions of the laboratories of the CWC organization have been identified and defined.

75. Recommendations:

- Additional details of the data base such as uses and users of data and data handling require further study.

- The design and organization of the information processing system for the CWC require further elaboration.

- The Preparatory Commission should begin the accreditation process to ensure that the laboratory network is operational when needed.

- The Preparatory Commission should begin work on the analytical data base before entry into force of the Convention because it will be needed to confirm initial declarations and other immediate matters.

- The Preparatory Commission should begin the creation of quality assurance programmes and the plans for their implementation.

ANNEX 1

BACKGROUND PAPERS

Canada and the United Kingdom:
- Concepts of Use for a Data-Base for Inspectors during CWC Inspections
- Systems Approach to Analytical Procedures, Equipment and Data Base

Finland and the Netherlands:
- The Network of Laboratories under the Chemical Weapons Convention: Possible structure and functions (CD/CW/WP.342)
- Accreditation of Verification Laboratories

The United States:
- Analytical Database Discussion Paper (CD/CW/WP.345)
- Information Processing for CW Monitoring (CD/CW/WP.346)
- Quality Assurance of Verification Analytical Laboratories (CD/CW/WP. 347)

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Australia:
- On-site Chemical Analysis for Verification of Non-production of Families of Scheduled Chemicals

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Material on quality assurance for analytical chemical laboratories:
- excerpt from " Handbook of Quality Assurance for the Analytical Chemistry Laboratory" by James P. Dux (pp. 104 - 109 "Typical Contents of a Quality Manual for Testing Laboratories")
- OECD Principles of Good Laboratory Practice
- European Standard EN 45001: General criteria for the operation of testing laboratories
- Finnish Standard SFS 5121: General requirements for the acceptance of inspection bodies (identical with document ISO-IEC Guide 39)

ANNEX 2

TECHNICAL GROUP ON ANALYTICAL DATABASE AND LABORATORIES
17.-21.6.1991

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Total 44

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Ad Hoc Committee on Chemical Weapons

AUSTRALIA; CANADA; CHINA; THE CZECH AND SLOVAK FEDERAL REPUBLIC; FINLAND; FRANCE; GERMANY; INDIA; THE NETHERLANDS; NORWAY; SWEDEN; SWITZERLAND; THE UNION OF SOVIET SOCIALIST REPUBLICS; THE UNITED KINGDOM OF GREAT BRITAIN AND NORTHERN IRELAND; AND THE UNITED STATES OF AMERICA

Second International Interlaboratory Comparison (Round Robin) Test

1. Introduction

The first International Interlaboratory Comparison (Round Robin) Test was undertaken between ten laboratories in 1989. A short description of the exercise was presented in Geneva as a joint working paper CD/CW/WP.288 in April 1990 and a full report in a Blue Book in early July 1990 (CD/1009, 5 July 1990).

It was agreed to arrange a second round-robin test and to enlarge the number of participants to fifteen. Australia undertook to prepare the samples and Finland was proposed as exercise coordinator.

2. Objective and types of samples

In order that the exercise should follow a likely CWC scenario it was agreed that the samples to be prepared should simulate an inspection of the chemical industry by including appropriate background chemicals. The identification of CW agents and their degradation products in complex sample matrices (water and soil), was identified as a way of testing and further developing existing methods and procedures recommended in the report of the first test.

In the event the laboratory that volunteered to prepare the samples for the test produced water but not soil samples. The preparing laboratory chose to use charcoal, Tenax, XAD-2 and cotton buds as matrices. One consequence of this was an increased number of samples over that originally envisaged.

Some of the samples were spiked with octyl methylphosphonofluoridate, which although a Schedule 1 chemical, is not (within public knowledge) a CW agent. This meant that many laboratories did not have the necessary reference material (authentic reference substance or data base information) to make a positive identification of the principal spiking substance. In the absence of a readily identifiable CW agent upon which to focus the participating laboratories applied varying degrees of effort in identifying as many compounds as possible to allow judgement whether they were scheduled chemicals.

Accordingly, to maximise the value of this second round-robin test for which an unanticipated number and type of samples were received, the objective of the test was modified to the analysis of samples (prepared to simulate inspection of a chemical industrial facility) for their content of any scheduled chemicals.

As previously, a separate round-robin file was created in the Finnish VERIFY database. Usernames and passwords were given to those laboratories for which direct contact information was available to enable them to add their results and experimental details to the database directly.

3. Sample preparation

Australia prepared the samples and analyzed one set of samples immediately after preparation and other sets three weeks and three months later. The following scenario was assumed: the samples were obtained during an inspection to a "Schedule-3" facility declared to consume more than 30 tonnes of trimethyl phosphite annually in the production of an organophosphorus insecticide.

Samples were taken from three feedchemical storage areas, FS1, FS2, and FS3, reaction vessel RV, auxiliary feedline AF leading to RV, two product storage areas PS1 and PS2, organic waste storage tank WS1, and hydrolyzed waste storage tank WS2. Samples were also taken of the charcoal from a respirator canister RC that had been used by an employee at the facility.

Approximately 100 mg of each liquid sample (FS1, FS2, FS3, PS1, PS2, and RV) was absorbed onto a clean cotton bud. Wipe samples of a whitish residue in AF was collected using cotton buds "wetted" with isopropanol. Three types of samples were collected from WS1: samples of liquid on a cotton bud WS1C, samples of liquid on XAD-2 resin WS1X, and headspace samples of vapor on Tenax-TA WS1T. Samples of the waste liquid from WS2 were collected.

Thus each laboratory was supplied with twelve different samples from each inspected facility, and duplicates of other samples than WS1T for which three samples were available. In addition, blanks of each sample matrix was provided.

Composition of the samples

The samples were prepared from either "commercial" or "laboratory grade" chemicals. Dichlorvos and octyl methylphosphonofluoridate and their respective by-products and degradation products were prepared in the laboratory. No attempt was made to accurately weigh a set amount of each sample into the sample tubes. In general, the cotton bud tubes contained slightly more than 100 mg of sample.

FS1: trimethyl phosphite (97%) containing traces of dimethyl phosphite (1%), dimethyl methylphosphonate (1%), and trimethyl phosphate (1%).

FS2: chloral (97%).

FS3: toluene (containing traces of xylenes).

AF: sodium fluoride, iso-octyl alcohol—a commercial product of closely related isomeric branched chain primary alcohols, including 3,4-dimethyl-1-hexanol (20%), 3,5-dimethyl-1-hexanol (30%), and 4,5-dimethyl-1-hexanol (30%).

RV: toluene (80%), dichlorvos (20%), and trace levels of the same impurities as in FS1 and FS3.

PS1: dichlorvos (>90%), and trace levels of the same impurities as in FS1 and FS3.

PS2: the same as PS1, spiked with octyl methylphosphonofluoridate (1%).

WS1: toluene (92%), dichlorvos (3%), dimethyl methylphosphonate (2%), dimethyl phosphite (1%), trimethyl phosphite (1%) and octyl methylphosphonofluoridate (1%).

WS2: sodium hydroxide, sodium salts of degradation/by-products of dichlorvos and octyl methylphosphonofluoridate (2 g of dichlorvos and 0.5 g of octyl methylphosphonofluoridate were added to 250 ml of 5% sodium hydroxide solution and refluxed).

RC: each charcoal sample (100 mg) was loaded with 50 µl of a standard solution containing the following components (mg/ml): toluene 740, dichlorvos 110, octyl methylphosphonofluoridate 50, dimethyl methylphosphonate 9, dimethyl phosphite 5, and trimethyl phosphite 5.

Samples were packed in small sealed glass tubes wrapped individually in plastic film. The sample tubes were then securely housed in a diecast alloy box, which was fully sealed with silicone sealant. No attempts were made to refrigerate the samples during transportation.

The packages were coded in the Embassy of Finland in Canberra before delivery to respective Embassies.

4. Analytical methods

The main analytical methods used in the exercise turned out to be gas. chromatography - mass spectrometry (GC-MS) together with nuclear magnetic resonance spectrometry (NMR) and infrared spectrometry (IR), mostly GC-FTIR. Many laboratories used gas chromatography with phosphorus-selective detectors to confirm the results obtained with mass spectrometry. Three laboratories used liquid chromatography (LC), one laboratory used liquid chromatography - mass spectrometry (TSP-LC-MS) and one laboratory used ion chromatography. One laboratory tested the potential of a mobile mass spectrometer and a further participant used retention spectrometry (RS). One laboratory used

laser Raman spectrometry (LRS) to identify solvents used in sample preparation.

5. Time frame

The samples arrived at the laboratories between late October and mid December; except for one laboratory which never received the samples. The time agreed for the analytical phase was one month from the date when the samples arrived at laboratories. After reporting of the identified chemicals, two further weeks were available to describe the used methods in detail. The coordinating laboratory prepared forms and tables and sent them to each laboratory to facilitate reporting and to obtain all necessary data.

The laboratories were asked to record their results on the VERIFY database directly or through the coordinating laboratory. Only two laboratories did this, the others mailing or faxing their results to the coordinating laboratory. One laboratory sent its results outside the agreed timeframe.

After all participants had reported their findings to the coordinator, Australia described how the samples were prepared and which chemicals were used to spike them and at what concentrations. The coordinating laboratory collected the results and methods used for sample pretreatment and analysis and forwarded this material to the laboratories together with chromatograms and spectra. This procedure enabled the participants to acquaint themselves with the results and methods used by other laboratories before the test was discussed in detail among the experts participating in the test. It was not possible to distribute all material from each laboratory to other participants owing to the amount of paper received by the coordinating laboratory.

6. Results

Table 1 shows 17 scheduled chemicals found in the samples. In addition to these chemicals 31 phosphorus-containing chemicals (see Table 2) and

88 chemicals not containing phosphorus and not included in the schedules were reported. The spiking chemicals are marked with an asterisk. The reference results obtained by Australia immediately after sample preparation are shown in column 15 (laboratory 15) and those obtained three months later are shown in column 16.

All laboratories identified the chemicals used as starting materials in the synthesis (trimethyl phosphite, trichloracetaldehyde (chloral), and toluene) and the planned end product, the pesticide dichlorvos. In addition to these main compounds a number of their impurities resulting from the technical grade feed chemicals were identified.

Only two laboratories reported chemicals belonging to Schedule 1, the mixture of isomeric octyl methylphosphonofluoridates. All laboratories, except one, identified dimethyl methylphosphonate, 6 laboratories identified the mixture of isomeric dioctyl methylphosphonates and 3 laboratories identified the mixture of isomeric methyl octyl methylphosphonates. Two laboratories reported detection of alkyl alkyl methylphosphonates but were not able to identify the actual alkyl groups. In addition to the octyl derivatives, four additional methylphosphonates and methylphosphonic acid were reported.

7. Discussion

International cooperation between laboratories in the form of interlaboratory comparison tests is important for method development and for gaining experience in the verification tasks required by the Convention. The objectives for these tests should be clearly defined beforehand to concentrate the efforts of the participating laboratories on a limited number of problems at a time and to avoid their being unnecessarily burdened with time-consuming tasks outside their normal duties. The testing activities may be planned to proceed from relatively simple tasks to gradually more complicated ones. Furthermore the analytical tasks required of the laboratories should be realistic but may not be accurate simulations of real situations.

The increase in the number of samples demanded greater effort than was originally anticipated except for those laboratories that used simple

extraction procedures and had an extensive database at their disposal. For them the exercise was mainly reduced to monitoring known compounds. Several laboratories used procedures consisting of several extraction and derivatization steps there being no recommended procedures, for example, for cotton buds or charcoal samples. In spite of the unexpected types and numbers of samples many of the laboratories considered the exercise as very interesting and challenging.

After the first round-robin test the experts discussed the need for background information on the samples. Information on the type of location where the samples were collected, whether alleged use or industry, was considered desirable. Information on the particular sampling points in the facility helped in understanding of the production process. This may, however, be considered as highly confidential information by the facility, and hence should not be revealed to a laboratory capable of doing in-depth analysis of all chemicals in the samples. On the other hand, in the presence of appropriate confidentiality agreements, some of this information might be released to assist the analysts. This problem does not arise, however, when the analyses are made on-site with "blinded" instruments having only scheduled chemicals in their databases. In this case all non-treaty chemicals would stay unidentified as structure elucidation would then be impossible. In the absence of extensive databases structure elucidation of unknown compounds would require combined use of several spectrometric methods and, therefore, would be outside the time frame of an on-site inspection.

In the meeting after the first round-robin test the experts discussed, but did not agree, specific criteria for identification. The minimum requirement was considered by many to be electron ionization and chemical ionization mass spectra together with information on the retention behavior. These requirements were discussed in the context of scheduled chemicals for which either identification data or authentic chemicals were available.

The present test strongly emphasized the need for identification criteria in verification analyses. Some laboratories did not list any chemicals for which no reference material was available, and the identification of which would have had to be based on structure

elucidation only. In this respect the isomeric chemicals posed a particular problem since complete analysis would have required solving the detailed structures of the isomeric compounds. Some laboratories listed chemicals solely on the basis of electron ionization spectra after comparison of the recorded spectra with those in commercial libraries.

Two laboratories used infrared spectra of the sample extracts without separating the components by gas chromatography to confirm identifications of the main constituents in the samples. One laboratory made these type of determinations having first recorded the spectra of main components by GC-FTIR.

These differences in the criteria affected the number of reported chemicals and led to a very large number of chemicals. In addition, like in the previous test, naming of the chemicals was not always quite clear and, in the absence of structural formula or CAS numbers, led to a considerable job for the coordinating laboratory.

Some laboratories reported having first monitored the absence of the known CW agents in Schedule 1 and, only afterwards, having continued with the analyses by identification of other scheduled compounds. Some laboratories reported monitoring also of chemicals in schedules 2 and 3 while some laboratories concentrated only in phosphorus-containing chemicals as they seemed to be the essential ones on the basis of gas chromatograms recorded with phosphorus-selective detectors. While the main emphasis in identification was on scheduled chemicals, some laboratories used much effort in identifying as many organic chemicals as possible. This difference in the approaches may explain the detection of phosgene by two laboratories only.

This test showed that most laboratories have sophisticated equipment and skills suitable for identification of known compounds and structure elucidation of unknown compounds in trace quantities. However, in the draft rolling text there is a clear preference for on-site analyses over off-site analyses especially during inspections to chemical industry. Off-site analyses are considered important mainly for confirmatory purposes i.e. after suspect samples have been identified on-site. Therefore, the results of this test have to be evaluated

against the success in finding those samples which would have required transportation and a more detailed analysis in verification laboratories. The most important samples were those containing Schedule 1 compounds and those containing methylphosphonates, especially the octyl derivatives.

The only Schedule 1 chemicals, the mixture of isomeric octyl methylphosphonofluoridates, were reported only by two laboratories although sophisticated in-house equipment were available in the majority of the laboratories. This is due to the degradation of the fluoridates during transport and the lack of analytical reference data. This underlines the need for rapid transport and that all verification laboratories have a database of scheduled chemicals as wide as possible. The database will not, however, remove completely the need for fully authenticated and validated reference chemicals.

Both the scope and quality of the database for the instruments used by inspectors on-site is important for the success of on-site analysis. It is the only way allowing on-site detection with chromatographic methods and, as shown by this test, very important also when mass spectrometry is used as the GC detector. Mass chromatography allows monitoring of scheduled compounds using different ion combinations and, in this respect, is more versatile than selected ion monitoring, although less sensitive. To reduce the amount of work, information on retention times is still essential to know where to look for compounds present in very low concentrations especially, when the samples also contain high concentrations of compounds having the same ions of interest.

NMR spectrometry, especially phosphorus NMR, turned out to be an important method for detecting the phosphorus compounds. The selectivity and sensitivity of fluorine NMR make it important for the detection of phosphonofluoridates. However, at present NMR spectrometers do not belong to portable, or even to transportable, equipment.

Infrared spectrometry seemed to afford a convenient method for additional confirmation of the identifications especially when reference spectra were available. Only one laboratory reported

infrared spectra of any of the isomeric octyl methylphosphonates. This may be due to the difficulty in obtaining infrared spectra from closely located GC-peaks and to the low concentrations of the fluoridates.

In this test the VERIFY database was not very helpful. Only a few of the chemicals were included in the database. There were no recommended operating procedures for the preparation of samples from cotton buds, charcoal and liquid samples. The procedures in the database for analytical methods were mainly for monitoring of known compounds, not structure elucidation. And the addition of the analysis results would have been unreasonably slow due to the large number of both samples and chemicals in the samples.

Finally, this test stressed the need to solve the problems associated with the transportation of samples. This will require designation of the United Nations' code numbers to the chemicals or classes of chemicals and placing them into the proper transportation class. The problem of declaring samples of unknown composition might be overcome by placing all samples into the strictest category of the transportation classes. At present, the laboratory responsible for the preparation of the samples is, however, the only one that can give information on the toxicity of the samples with a view to finding the appropriate transportation means. Rules acceptable for every government should, therefore, be found for the transportation of test samples in order to facilitate the present work of the future Convention geared to improving the reliability of the verification procedures.

8. Conclusions

Using these round-robin samples as specific examples, the following (inspection scenario) conclusions regarding the inspection of a chemical facility can be drawn.

An inspector, an expert in CW chemical analysis, and equipped with a transportable gas chromatograph - mass spectrometer, would have been able to obtain a preliminary identification on-site, that a Schedule 1 compound was present provided that its characteristics were in the data base. Because the reference data for this methylphosphonofluoridate

was not available in the database, preliminary identification on-site could not have been possible. However, an indication alone of the presence of a Schedule 1 chemical would have required the samples to be sent to two designated laboratories for confirmatory analyses.

Unambiguous identification of the Schedule 1 compound would require the use of two or more independent spectrometric techniques. The unambiguous identification may also require the synthesis of a authentic standard for comparison which may increase the time required for a complete report to the Technical Secretariat.

With sophisticated instruments skilled analysts can identify almost any chemicals present in the samples in minute quantities if reference data is available. Therefore, to protect confidential business information use of "blinded" instruments on-site and sending of coded samples to off-site laboratories are recommended.

The nature of the Schedule 1 chemicals in this round-robin exercise also emphasized the requirement for proper sampling methods, means for sample stabilization during transport, and rapid transport of the samples to designated laboratories.

TABLE 1. Compounds identified in the samples of round-robin 2.

	SCHEDULE/COMPOUND NAME	LABORATORY															
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
	SCHEDULE 1:																
1	*Octyl methylphosphonofluoridate	x				x										x	
	SCHEDULE 2:																
2	Methylphosphonic acid	x	x	x	x	x	x	x				x			x	x	x
3	Methyl methylphosphonate	x						x				x			x		
4	*Dimethyl methylphosphonate	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
5	Ethyl methyl methylphosphonate	x			x	x	x										
6	Isopropyl methylphosphonate	x			x										x^T		
7	Octyl methylphosphonate	x				x	x								x^T	x	x
8	Methyl octyl methylphosphonate	x				x	x									x	x
9	Dioctyl methylphosphonate	x		x		x	x				x				x^T	x	x
10	Methylphosphonic acid, octyl ester pyrophosphate						x^T										
	SCHEDULE 3:																
11	*Trimethyl phosphite	x	x	x	x	x	x	x	x^T	x	x	x	x	x	x	x	x
12	Dimethyl ethyl phosphite	x								x^T							
13	Diethyl methyl phosphite									x^T							
14	Triethyl phosphite									x^T							
15	*Dimethyl phosphite	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
16	Ethyl methyl phosphite									x^T							
17	Phosgene	x				x											

* spiking chemical
x^T tentative identification

TABLE 2: Other organophosphorus compounds reported in the samples of round-robin 2.

	OTHER ORGANOPHOSPHORUS COMPOUNDS:	LABORATORY															
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
18	•Dichlorvos	x	x	x	x	x	x	x	x^T	x	x	x	x	x	x	x	x
19	Monochlorvos	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
20	2,2-Dichloroethyl dimethyl phosphate	x															
21	2,2-Dichloroethenyl ethyl methyl phosphate	x													x		
22	4,4-Dichloro-1-butenyl methyl phosphate														x		
23	4,4-Dichlorobutyl dimethyl phosphate																
24	[Trichloro-(dimethylphosphoryl)ethyl] dimethyl phosphate	x															
25	[Dichloro-(dimethylphosphoryl)ethyl] dimethyl phosphate	x															
26	Trichlorfon		x			x			x^T				x		x		
27	1,2,2,2-Chloroethyl dimethylphosphate														x		
28	Phosphonic acid (orthophosphoric acid)		x	x													
29	Methylphosphate	x	x	x		x	x				x	x			x		
30	•Dimethyl phosphate	x	x	x	x	x	x	x				x			x	x	x
31	•Trimethyl phosphate	x	x	x	x	x	x	x		x	x	x	x	x	x	x	x
32	Ethyl dimethyl phosphate				x		x										
33	Diethyl methyl phosphate						x								x		
34	Dimethyl propyl phosphate														x		
35	Triethyl phosphate		x														
36	Dimethyl 2-methylbutyl phosphate						x										
37	Dimethyl pentyl phosphate	x															
38	Dimethyl n-pentyl phosphate		x														
39	Dimethyl n-hexyl phosphate		x														
40	Dimethyl benzyl phosphate	x													x		
41	Methylphenyl dimethylphosphate											x					
42	Phosphonic acid (Phosphorous acid)		x	x	x							x					
43	Methyl phosphite (Methyl phosphonate)																
44	Methyl dimethylphosphinate	x															
45	Dimethyl phosphorofluoridate	x				x						x					
46	Dimethyl phosphorochloridate																
47	Trimethyl phosphorothionate				x												
48	Dibutyl ester of phosphoramidic acid		x														

• spiking chemical

x^T tentative identification

TABLE 3: Nonphosphorus compounds reported in the samples of round-robin 2.

	NONPHOSPHORUS COMPOUNDS:	LABORATORY															
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
49	*Trichloroacetaldehyde (chloral)	x	x	x	x	x	x	x	x[T]	x		x		x	x	x	x
50	Chloral hydrate (1,1-ethanodiol, 2,2,2-trichloro)		x				x	x			x		x		x	x	x
51	Methyl chloral														x		
52	Methyl dichloroacetate														x		
53	Methyl trichloroacetate						x										
54	Dichloroacetaldehyde	x			x		x										
55	Benzene						x										
56	*Toluene	x	x	x	x	x	x	x	x[T]	x	x	x	x	x	x	x	x
57	*Dimethyl benzene (xylene, xyle)	x	x				x	x		x		x			x	x	x
58	Trimethyl benzene		x				x										
59	Ethyl benzene		x					x				x			x		
60	2,4-Di(ter-butylphenol)														x		
61	Ethyl methyl benzene		x														
62	Methanol	x	x								x	x					
63	Ethanol	x					x				x	x				x	x
64	Isopropyl alcohol (2-propanol)	x	x				x	x				x		x		x	x
65	3-Methyl-1-pentanol						x										
66	2-Ethyl-4-methyl-1-pentanol						x										
67	2,2,4-Trimethylpentanol													x			
68	6-Methyl-1-heptanol		x											x			
69	*Octanol	x	x	x		x	x				x	x			x		
70	*Isooctanol (isooctyl alcohol)						x							x		x	x
71	*3,4-Dimethyl-1-hexanol																
72	*2,5-Dimethyl-1-hexanol																
73	*4,5-Dimethyl-1-hexanol																
74	Nonyl alcohol						x										
75	Decanol		x														
76	1-Dodecanol (dodecyl alcohol, lauric alcohol)		x												x		
77	2,2-Dimethylpentanol														x		
78	3-Methyl-1-hexanol														x		
79	2,2-Dimethyl-1-hexanol														x		
80	6-Methyl-1-heptanol														x		
81	2,3 Dimethyl-2 butanol														x		
82	2-Methyl-2-pentanol														x		
83	3-Methyl-3-pentanol														x		
84	1,2-Propandiol		x														
85	Diethyl phthalate						x										

* spiking chemical

TABLE 3: continued

	NONPHOSPHORUS COMPOUNDS:	LABORATORY															
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
86	Dioctyl phthalate						x								x		
87	Dibutyl phthalate						x										
88	Dibenzyl maleate [2-butenedioic acid, bis(phenyl-methyl) ester]																
89	Methyl hexadecanoate (methyl palmitate)		x														
90	Methyl stearate (methyl octadecanoate)		x														
91	Ethyl palmitate (ethyl hexadecanoate)						x										
92	Ethyl stearate (ethyl octadecanoate)						x										
93	Hexachloroethane		x			x		x									
94	Tetrachloroethylene							x									
95	Hexachlorobutadiene							x									
96	Fluoromethane		x														
97	Hexachlorobenzene		x														
98	5-Nonene-4-one		x														
99	Benzaldehyde		x														
100	2,5-Dimethylthiophene		x														
101	3-Ethylhexane		x														
102	2,4-Dimethylheptane		x														
103	Propylbenzene		x														
104	1,3,5-Trimethylbenzene		x														
105	Butyl benzoate (butyl ester of benzoic acid)		x														
106	Tetrachloroethane		x														
107	Thiophene-2-carboxamide		x														
108	Butyl 2-butenoate (butyl ester of 2-butenoic acid)		x														
109	2,6-Bis(1,1-dimethylethyl)-4-ethyl-phenol		x														
110	3-Hydroxy-2-pyridinethione		x														
111	3-Methoxy-5,2-cyclopentene-1-one		x														
112	1-Methyl-1-hydroxy-2-cyclohexene		x														
113	1,4-Diphenyl-1-butanone ($C_{16}H_{16}O$)										x	x					
114	Water (H_2O)					x						x					
115	Sodium ion (Na^+)	x				x											
116	Fluoride ion (F^-)					x										x	x
117	*Sodium fluoride (NaF)	x															
118	Dichloromethane (methylene chloride)	x	x		x	x	x								x		
119	Trichloromethane (chloroform)																
120	Methyl naphthalene		x														
121	Boric acid																

* spiking chemical

TABLE 3: continued

	NONPHOSPHORUS COMPOUNDS:	LABORATORY															
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
122	2-Hexanone														x		
123	3-Methylheptane														x		
124	n-Undecane														x		
125	Methyldecane														x		
126	o-Cresol														x		
127	2-Methylheptane														x		
128	3-Methylheptane														x		
129	3,4-Dimethylhexane														x		
130	Phenylacetaldehyde														x		
131	Hexaethylbenzene														x		
132	Tetramethylphenanthrene														x		
133	Cyclooctatetraene														x		
134	n-Nonane														x		
135	2,6-Tertbutyl-4-methylphenol														x		
136	Furan														x		
137	3,4-Epoxy-2-butanone														x		

* spiking chemical

CONFERENCE ON DISARMAMENT

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Ad Hoc Committee on Chemical Weapons

FRANCE

REPORT ON A NATIONAL TRIAL INSPECTION TO VERIFY AN INDUSTRIAL FACILITY

I. INTRODUCTION

As the provisions of the rolling text of the Convention now stand, verification of activities that are not prohibited is limited, as regards the chemical industry, to routine checking of facilities producing, processing or consuming Schedule 2 substances and to the provision of data concerning Schedule 3 substances.

A number of countries, including France, have expressed the view that this inspection procedure does not cover all the potential risks of circumvention of the Convention, especially those associated with facilities "capable" of, but not actually producing substances on Schedules 1, 2 or 3.

In order to fill this gap, two proposals aimed at supplementing routine inspection and covering the risks described above have been made by Germany and the United Kingdom (ad hoc checks: CD/791 and CD/869; ad hoc inspections: CD/909). The United States has also made a proposal to this end (ad hoc visit: CD/CW/WP.300). In addition, Germany (CD/950) and the Netherlands (CD/925) have conducted ad hoc trial inspections in order to define and evaluate the relevant procedures. Recently, Sweden has proposed an integrated system of verification in the chemical industry that takes into account "capable" facilities (CD/1053).

With a view to assessing the effect of a system of ad hoc verification on the industry, and particularly to measuring its impact on confidentiality, a trial inspection of this type has been carried out at an industrial site in the south of France. This exercise was conducted before the announcement of the above-mentioned Swedish proposal.

The objective of this practical experiment was twofold:

To assess the relevance or suitability of ad hoc verification as part of a routine system;

To elaborate and evaluate procedures taking account of the interests of all the parties involved.

This document describes the inspection and sets out the main conclusions drawn from it.

II. SITE INSPECTED

The distinguishing feature of the industrial site subjected to inspection was that it contained facilities producing for civilian and military use. There were three types of production activity at the site:

(a) Explosives for civilian and military use, with some of the plant sections being considered sensitive in terms of national security;

(b) Nitro products, the production facilities for which are multi-purpose shops with stainless steel equipment;

(c) Fine chemistry producing, inter alia, organophosphorus derivatives and using a Schedule 3 product; the facilities in this case include multi-purpose equipment with enamelled reactors.

In addition, the site contained:

Workshops for the development of new products (pilot or medium-sized synthesis facilities considered as sensitive areas in terms of industrial secrecy);

A laboratory for analysis and synthesis that is also a sensitive area;

An effluent storage and processing facility;

An infirmary.

The plant did not comprise any facilities producing Schedule 2 or 3 products above the permitted thresholds set in the rolling text.

III. BACKGROUND INFORMATION

3.1. Declarations

Two types of register were tested during the inspection:

(1) A national register as proposed by Germany (CD/984) and the United States (CD/CW/WP.300) listing the plants possessing one or more facilities potentially capable of producing substances subject to declaration;

(2) A two-part register comprising:

A part - termed the International Register - intended for the Technical Secretariat, identical to the register described in (1), which also mentioned the number of "capable" facilities within, and the total area of the plant;

A part - termed the National Register - reserved for the National Authority, more detailed, and certain information from which was communicated to the inspectors upon their arrival at the point of entry.

The part held by the National Authority included:

For use by the inspectors:

Information on the site available in the public literature (directories, publicity brochures);

Information on the activities, the main products (with quantity ranges), certain information on product quality;

Information on previous production of substances listed in the Schedules (pursuant to the requirements concerning declarations laid down in the annexes to articles IV and VI);

A technical description of the site, including in particular the sampling points that could be used if required and other technical details of a nature to facilitate the inspection;

For the exclusive use of the National Authority:

Precise descriptions of the "capable" facilities;

The production capacities and the actual production;

Information on production and use of scheduled products below the declaration thresholds;

The sensitive and/or non-relevant facilities subject to access restrictions for reasons of national security or industrial secrecy.

3.2 The mandate

The mandate, which was presented at the point of entry by the inspection team leader, contained in particular:

The name and location of the site to be inspected as they appeared in the register at the disposal of the Technical Secretariat, and the number of "capable" facilities on the site;

The composition of the inspection team and of the technical team;

The authorization for the team of inspectors to have access to additional information on the site contained in the register in the possession of the National Authority;

The points to be verified, namely: non-production of Schedule 1 substances; non-production above the permitted thresholds of Schedule 2 and 3 substances; verification that, if there was production, it was below the permitted thresholds;

Descriptions of the analytical instruments brought by the inspectors and of the method of analysis employed;

A restatement of the relevant provisions of the Convention, particularly those pertaining to confidentiality.

IV. THE OPENING CONFERENCE

Its purpose was to supplement the information given in the registers. Consequently, the following were provided:

A general description of the site: location and layout, personnel, main types of activity;

A quite detailed description of the facilities considered "capable" by the industry, with reference to a shop plan, but with preservation of the confidentiality of the production activities unrelated to the Convention;

A declaration on the use of substances on the Schedules in amounts below the declaration thresholds.

Concerning the main types of activity, the following were stated: the names and the total volumes of output of the main products, the uses of the products and the general processes employed for their manufacture, the numbers and unit volumes of the reactors. Descriptions were provided of the equipment, i.e.: standard of the reactor linings, numbers and useful volumes of fixed storage facilities, safety facilities.

Regarding the fine chemistry, a classification was made of the production units, i.e., from the point of view of the representative of the site, the production units capable of making scheduled products and the shops not in principle capable of making such products while adhering to the usual safety rules. Mention was also made of the unit using small quantities of a Schedule 3 product.

In addition, a description was given of the safety rules to be observed on the site.

Lastly, with regard to confidentiality, the representative of the site defined the sensitive areas (areas for the manufacture of explosives for military use) with respect to which access was limited to the shop doorway.

Replies to questions following this opening conference were confined to those deemed pertinent by the representatives of the site and the National Authority.

V. INSPECTION PLAN

5.1 The notion of "capable" facility

In the absence of a definition of the term "capable" (capable of producing, processing, consuming substances subject to declaration?), which lies at the basis of the purpose of ad hoc verification and inclusion in the registers, the representatives of the site proposed the following criteria during the opening conference:

The nature of the equipment, particularly the inner lining;

The volume of the reactors ($1m^3$);

The capacity for production, processing or consumption of a Schedule 2 or 3 substance, the limit being higher than or equal to one-tenth of the thresholds mentioned in the ruling text.

However, since it was a trial inspection, the inspectors felt that all the facilities could be inspected, or at least visited, in order to verify that they were or were not "capable".

5.2 Elaboration of the plan

Following a general tour of the site, the inspection team decided to proceed by random sampling both for the facilities considered "capable" facilities by the representatives of the site and for the other facilities.

In the case of the research and development facilities (whether laboratories or pilot production shops), the representatives of the site authorized only sampling at the exit points from the facilities, with, in order to prevent the transfer outside the plant of precise information concerning the actual products being studied or developed, the proviso that the analysis must be carried out on the site by the inspection team.

VI. VERIFICATION ARRANGEMENTS

6.1 - Composition of the inspection team

The inspection team comprised the following:

One chemical engineer specializing in chemical weapons;

One chemical engineer who was formerly the site manager;

One chemical engineer specializing in the inspection of classified facilities (environment);

One specialist and one assistant for sampling.

6.2 - Duration of the inspection

The inspection itself took two half-days; drafting the report and general discussion after the exercise, half a day. The analyses were not conducted on-site; if they had been, the inspection would have taken about half a day longer.

6.3 - Verification process

The following criteria were used by the inspection team to determine whether facilities were "capable":

Characteristics of the major equipment used (size, standard of inner linings, treatment of effluents, etc.);

The raw materials and processes used, the nature of the chemicals produced or used;

The environment of the facility (security arrangements, substance, containment devices).

6.4 - Access to materials records

One of the site facilities consumed a Schedule 3 substance. In addition, in the course of the inspection, the team discovered the presence of another Schedule 3 substance, repackaged by the enterprise.

The inspectors accordingly expressed the wish to be allowed to check the throughput of these Schedule 3 substances by consulting accounting records so as to verify that no diversion was taking place. This seemed particularly necessary in view of the fact that the facility was not declared for the purposes of production of these substances and produced or consumed a scheduled substance in a quantity just below the permitted threshold.

The problem was thus to gain access to the materials records in order to ascertain whether a threshold had or had not been exceeded.

In the event, the representative of the site refused to hand over the accounting records on the grounds that they did not fall within the scope of the inspection and that there was no such obligation for this type of inspection.

6.5 - "Capable" facilities located in a sensitive area

The inspectors expressed the desire to inspect a facility considered "capable" but situated in a sensitive area and secret by virtue of the fact that its production was defence-related, but not within the scope of the Convention. The representative of the site refused from the outset access to such a facility.

In this particular case, he authorized only the sampling of an effluent outside the facility, and permitted the leader of the inspection team to go as far as the threshold of the facility so that he could make a visual assessment of the capacity and nature of the facility.

6.6 - Sampling and analysis

Several samples were taken by the sampling team, either in the facilities visited or (effluents) outside the facilities to which it did not have access. It was not possible to estimate how long the analyses would really take, given the lack of adequate instrumentation.

VII. MAIN FINDINGS

(1) For this inspection, the concept of a "capable" facility was determined essentially on the basis of the nature of the equipment. This concept should be established either by a simple and precise definition, or on the basis of criteria allowing for a clearer definition, for the term "facility capable of" is the key element in registration and thus in ad hoc inspections.

(2) The facilities to be inspected on-site, in an inspection of this type, are the following:

Facilities producing, using or consuming Schedule 2 or 3 substances;

Facilities "capable" of producing such substances.

Where, because the site is large, there are many such facilities, the inspection team should make a random selection so as to reduce inspection time.

One way of doing this might be to inspect at random either one of the "capable" facilities, or a limited number of "capable" facilities, based on the total number of declared "capable" facilities.

(3) Inspection conducted like this trial, i.e. by the sampling of all facilities, "capable" or otherwise, was considered by the industry representatives as highly intrusive.

Nevertheless, the question does in fact arise in the case of a "capable" facility producing, by a confidential process, a substance intended, for example, for defence and outside the scope of the Convention. It should be made clear, perhaps in the Protocol on Inspection Procedures, how to deal with "capable" facilities access to which is covered by confidentiality linked with the security of the State party. Logically, such facilities should be registered since they have production capacity. In such cases, there can be no question of a thorough visual examination or of sampling inside or at the outlet of a reactor. This suggests that provision must be made for the possibility of managed access.

(4) As far as the actual conduct of the inspection is concerned, it became clear that a detailed inspection protocol for ad hoc verification is absolutely essential.

In the absence of specific facility agreement, there is definitely a need for clearly defined inspection procedures. The development of standard provisions applicable to all types of facility liable to be inspected is of great importance for the industry, which must have a clear idea of what to

expect in an inspection of this type. Otherwise, as the site representatives pointed out, such an inspection could be regarded as a challenge inspection.

Furnishing inspectors with information on the site contained in the National Register, as described in paragraph 3.1(2), is another way of offsetting the lack of a facility agreement.

(5) The effectiveness of this type of inspection depends primarily on the technical resources at the inspectors' disposal, particularly those for sampling and analysis. Technical back-up for an inspection of this type raises a variety of problems, and inspectors should have the services of a team specifically responsible for sampling and analysis. In addition, sampling rules and procedures applicable in all cases must be established.

It would be preferable for on-site analytical inspections to focus only on specific chemical compounds. However, particularly in the case of sensitive facilities, but also in the case of "capable" facilities not producing scheduled substances, a study should be made of the feasibility and reliability of procedures for analysis by exclusion enabling the confidentiality of production processes to be preserved. Such checks, being made in an industrial environment, would require truly operational portable equipment.

There is no question that the analysis of samples must be conducted on-site if subsequent disputes and counter-evaluations, not to mention loss of confidentiality, are to be avoided. Moreover, the results of such analyses might, in some cases, determine the subsequent course of an inspection operation.

Off-site analysis of samples should be authorized only if two successive on-site analyses of a single sample, confirm the presence of Schedule 1 substances. When that occurs, a duplicate sample, sealed by the inspectors, should be handed over to the National Authority.

(6) In some cases, the inspection team may consider an examination of records necessary in order to ascertain the throughput of a substance on the site and to verify that no diversion of a scheduled substance is taking place. If the facility is not declared as producing a scheduled substance, but produces such a substance in a quantity below the permitted threshold, access to materials records would seem essential.

Such records are not always available on-site, or are not presented in a form which is immediately usable by the inspection team. In the view of the industry, examination of materials records seems much too intrusive. In the view of the inspectors, it can prove ineffective because of its complexity and the length of time required in the absence of prior knowledge or preparation.

(7) The inspection report should contain only factual information, i.e. conclusions as to the existence or non-existence of evidence or indications of

unlawful activities in the facility inspected. Indeed, only the existence of such evidence can provide the basis for a conclusion. In the absence of such evidence, no definite conclusion is possible.

VIII. CONCLUSION

Conducting such a trial exercise, in which everything had to be thought out from scratch, proved difficult, as the placement of each element depended on basic data that have as yet been defined only poorly or not at all in the rolling text.

However, from the standpoint of an integrated approach to verification in the chemical industry, it was felt worthwhile sharing the results of this experiment and the lessons learned from it.

CONFERENCE ON DISARMAMENT

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Ad Hoc Committee on Chemical Weapons

Working Paper

**AUSTRALIA, JAPAN,
THE UNITED KINGDOM OF GREAT BRITAIN AND NORTHERN IRELAND
AND THE UNITED STATES OF AMERICA**

Recommended Text for Article IX - Challenge Inspection

General

1. Each State Party shall have the right to request an on-site challenge inspection of any facility or location in any other State Party for the purpose of clarifying and resolving any questions concerning compliance with the provisions of the Convention, and to have this inspection conducted anywhere without delay by an inspection team designated by the Director-General and in accordance with the Protocol on Inspection Procedures. Each State Party shall only make requests that are within the scope of the Convention.

2. For the purpose of verifying compliance with the provisions of this Convention, each State Party shall permit the Technical Secretariat to conduct on-site challenge inspections pursuant to paragraph 1.

3. When challenged, each State Party shall have the right and the obligation to make every reasonable effort to demonstrate its compliance with the Convention and to enable the inspection team to fulfill its mandate, in accordance with procedures provided for in the Protocol on Inspection Procedures. The challenged State Party is under the obligation to provide access within the requested site for the sole purpose of establishing facts relevant to the request, and has the right to take measures to protect sensitive installations and to prevent disclosure of confidential information not related to the Convention, in accordance with the Protocol on Inspection Procedures.

4. The challenging State Party shall have the right to send a representative to observe the conduct of the inspection. The challenged State Party shall grant access to the observer in accordance with the Protocol on Inspection Procedures.

Notification

5. The challenging State Party shall present a request for an on-site challenge inspection to the Director-General of the Technical Secretariat. The Director-General shall notify the challenged State Party not less than 12 hours prior to the planned arrival of the inspection team at the point of entry. Contemporaneously the members of the Executive Council shall be informed about the request.

Inspections

6. The Director-General of the Technical Secretariat shall issue a mandate for the conduct of the inspection. The mandate shall be the challenging State Party's request put into operational terms, and shall conform with the request.

7. The inspection shall be conducted in accordance with Part III, or, in the case of alleged use, in accordance with Part IV of the Protocol on Inspection Procedures. The inspection team shall be guided by the principle of conducting the inspection in the least intrusive manner possible, consistent with the effective and timely accomplishment of its mission.

8. The challenged State Party shall assist the inspection team throughout the inspection and facilitate its task. Should the challenged State Party propose, pursuant to Part III, Section III.B of the Protocol on Inspection Procedures, arrangements to demonstrate compliance, alternative to full and comprehensive access, it shall make every reasonable effort, through consultations with the inspection team, to reach agreement on the modalities for establishing the facts with the aim of demonstrating its compliance.

9. The Director-General of the Technical Secretariat shall promptly transmit the final report of the inspection team to the challenging State Party, to the challenged State Party, to the Executive Council and to all other States Parties. The final report shall contain the factual findings as well as an assessment by the inspection team of the degree and nature of access and cooperation granted to the inspectors and the extent to which this enabled them to fulfill their mandate. The Director-General shall further transmit promptly to the Executive Council the assessment of the challenging State Party, the view(s) of the challenged State Party, and the view(s) of other States Parties which may be conveyed to the Director-General for that purpose, and then provide them to all States Parties.

10. When requested by any State Party, the Executive Council shall meet within 48 hours to review the situation and consider any appropriate further action necessary to redress the situation and to ensure compliance with the Convention, including specific proposals to the Conference of the States Parties. At such a meeting, the challenging State Party and the challenged State Party shall have the right to participate. The Executive Council shall inform the States Parties of the outcome of its meeting.

PROTOCOL ON INSPECTION PROCEDURES

PART I: GENERAL

I. Definitions

"Inspection Site" means an area or facility at which the inspection is carried out and which is specifically defined in the respective facility agreement or inspection request or the inspection request as expanded by the alternative, provisional or final perimeter.

"Perimeter" in case of a challenge inspection means the external boundary of the inspection site, either defined by geographic co-ordinates or by description on a map.

- "Requested Perimeter" means the inspection site perimeter as specified in the inspection request; it shall conform to the requirements of paragraph 4 of Part III, Section II.B.

  "Alternative Perimeter" means the inspection site perimeter as specified, alternatively to the requested perimeter, by the challenged State Party; it shall conform to the requirements of paragraph 2 of the Part III, Section II.C.

- "Provisional Perimeter" means the inspection site perimeter as agreed if necessary in negotiations between the inspection team and the challenged State Party; if such negotiations should not lead to an agreement, the alternative perimeter would also constitute the provisional perimeter.

- "Final Perimeter" means the final inspection site perimeter as agreed if necessary in negotiations between the inspection team and the challenged State Party; if such negotiations should not lead to an agreement, the provisional perimeter would also constitute the final perimeter.

PART III: ON-SITE CHALLENGE INSPECTIONS FOR UNDECLARED FACILITIES CONDUCTED PURSUANT TO ARTICLE IX

I. Designation and Selection of Inspectors and Inspection Assistants (See CD/1046, Appendix I, Addendum, page 155, paragraphs 1 and 2)

II. Pre-Inspection Activities

A. Notification

1. The request for an on-site challenge inspection shall be submitted to the Director-General of the Technical Secretariat and shall contain the following information:

a) the State Party to be inspected and, if applicable, the Host State;

b) the concern regarding compliance with the Convention including a specification of the relevant provision of the Convention about which concerns have arisen, and the nature and circumstances of the suspected non-compliance;

c) the point of entry to be used;

d) the size of the challenged site;

e) the name(s) of the observer(s) of the challenging State Party; and

f) any additional information the challenging State Party deems necessary.

2. The Director-General of the Technical Secretariat shall within [one] hour(s) acknowledge to the challenging State Party the receipt of its request.

3. The Director-General of the Technical Secretariat shall notify the challenged State Party and the members of the Executive Council of the on-site challenge inspection not less than 12 hours prior to the arrival of the inspection team at the point of entry. The notification shall contain the following information:

a) the name of the challenging State Party and the name of the observer of the challenging State Party;

b) the point of entry to be used;

c) the size of the inspection team; and

d) relevant information regarding aircraft arrangements.

B. Entry into the Territory of the Challenged State Party or Host State

1. The Director-General of the Technical Secretariat shall dispatch an inspection team as soon as possible after a request is received by the Technical Secretariat. The inspection team shall arrive at the point of entry specified in the request in the minimum period of time possible, consistent with the provisions of paragraph A.3 above of this Protocol.

2. Unless already included in the request for a challenge inspection, the challenging State Party shall, within 24 hours after arrival of the inspection team at the point of entry, simultaneously inform the inspection team and the challenged State Party of the location of the challenged site. At the same time the challenged State Party shall also be informed by the inspection team of the inspection mandate. Contemporaneously, the Executive Council shall be informed of the above information.

3. When presented to the challenged State Party by the challenging State Party, the challenged site shall be designated as specifically as possible by providing a site diagram related to a reference point with geographic coordinates specified to the nearest second if possible. Where specification to the nearest second is not possible owing to the absence of sufficiently detailed maps, or where it would be helpful, site diagrams shall be supplemented by written descriptions. If possible, the challenging State Party shall also provide a map with a general indication of the inspection site and a diagram specifying precisely the boundaries of the site to be inspected.

4. The requested perimeter shall:

- Run a reasonable distance outside any structures;
- Not cut through existing security enclosures;
- Run a reasonable distance outside any existing security enclosures that the challenging State Party intends to include within the requested perimeter.

5. If the requested perimeter is acceptable to the challenged State Party, it shall be designated as the final perimeter as early as possible but in no case later than 60 hours after specification of the location of the challenged site. The challenged State Party shall transport the inspection team to the final perimeter of the inspection site. Such transportation shall be accomplished as soon as practicable, but in any case shall take no more than 12 hours after agreement on the perimeter.

C. Alternative Determination of Final Perimeter

1. At the point of entry, if a challenged State Party cannot accept the requested perimeter, it shall propose an alternative perimeter as soon as possible, but in any case no later than 60 hours after having been informed of the location of the challenged site. Differences shall be negotiated between the challenged State Party and the inspection team with the aim of reaching agreement on a final perimeter.

2. The alternative perimeter should be designated as specifically as possible in accordance with paragraph B.3 above. It shall include the challenged site and should as a rule bear a close relationship to the requested perimeter taking into account natural terrain features and man-made boundaries. It should normally bear a close relationship to the surrounding security barrier if such a barrier exists. The challenged State Party could seek to establish such a relationship between the perimeters by one or more of the following means:

- An alternative perimeter that does not extend to an area significantly greater than that of the requested perimeter;

- An alternative perimeter that is a short, uniform distance from the requested perimeter;

- At least part of the requested perimeter is visible from the alternative perimeter.

3. If the alternative perimeter is acceptable to the inspection team, it shall become the final perimeter and the inspection team shall be transported from the point of entry to that perimeter as soon as possible, but in any case no longer than 12 hours after acceptance.

4. If a final perimeter is not readily agreed upon at the point of entry, the inspection team and the challenged State Party should agree on a provisional perimeter which shall include the challenged site and which shall meet the same criteria as in paragraph II.C.2 above.

5. The perimeter negotiations at the point of entry shall be concluded as early as possible, but in no case shall they continue more than 60 hours after specification of the site by the challenging State Party. If no agreement is reached at the point of entry, the challenged State Party shall designate the alternative perimeter as the provisional perimeter. The challenged State Party shall transport the inspection team to a location at the provisional perimeter as soon as practicable, but in any case shall ensure their arrival at the location no later than 12 hours after agreement on, or designation of, the provisional perimeter.

6. Once at the location, the challenged State Party shall provide the inspection team with prompt access to the provisional perimeter to facilitate negotiations and agreement on the final perimeter and access within the final perimeter.

7. If no agreement is reached within 96 hours after the arrival of the inspection team at the location, the provisional perimeter shall be designated the final perimeter.

D. Verification of Location

1. The inspection team shall have the right to use location finding equipment and have such equipment and other approved equipment installed according to its directions. The inspection team may verify their location by reference to local landmarks identified from maps. The challenged State Party is to assist them in this task.

E. Securing the Site

1. No later than 24 hours after specification of the location of the challenged site, the challenged State Party must identify all exit points for all land, air, and water vehicles from the requested perimeter and provide the inspection team with evidence of all vehicular exit activity from the requested perimeter. Such evidence must consist of at least one of the following, to be selected by the challenged State Party:

- Traffic logs;
- Photographs;
- Video recordings;
- Chemical evidence equipment provided by the inspection team to observe but not interfere with such exit activity;
- Allowing one or more members of the inspection team to observe but not interfere with such exit activity.

2. Upon the inspection team's arrival at the provisional perimeter or final perimeter, whichever occurs first, exit monitoring by the inspection team shall begin using procedures agreed upon by the inspection team and the challenged State Party. Such procedures will include identification of vehicular exits and could include:

- Provisions for shrouding of equipment;
- Use of sensors;
- Random selective access;
- Sample analysis.

3. Personnel and vehicles entering, and personnel and passenger vehicles exiting, the site are not subject to inspection.

4. The application of such procedures, to include exit monitoring, may not unreasonably hamper or delay the normal operation of the facility.

F. Perimeter Activities

1. At the final perimeter, the inspection team shall have the right to commence immediately perimeter activities in accordance with the procedures set forth in this section, and to continue these activities until the completion of the inspection, or longer at the discretion of the challenged State Party. Subject to agreement of the challenged State Party, the inspection team may conduct perimeter activities at the provisional perimeter.

2. The inspection team shall have the right at the final perimeter around the inspection site to:

a) conduct perimeter inspection using monitoring instruments (consistent with Part I, Section IV.D of the Protocol on Inspection Procedures);

b) take wipes, air, soil or effluent samples, and

c) conduct any additional activities which may be agreed between the inspection team and the challenged State Party.

3. The perimeter activities of the inspection team may be conducted within a band around the outside of the final perimeter up to 50 meters in width measured outward from the perimeter. If the challenged State Party permits, the inspection team may also have access to any building or structure within the perimeter band. All directional monitoring shall be oriented inward.

G. Inspection Plan

1. To facilitate development of an inspection plan, the challenged State Party shall provide a safety and logistical briefing to the inspection team prior to access. The team shall be briefed by facility representatives, with the aid of maps and other documentation as appropriate, on the activities carried out at the facility, safety measures, and administrative and logistical arrangements necessary for the inspection. The time spent for the briefing shall be limited to the minimum necessary.

2. In the course of the briefing, the challenged State Party may indicate to the inspection team the equipment, documentation or areas it considers sensitive and not related to the purpose of the inspection. Additionally, personnel responsible for the site will brief the team on the physical layout and other relevant characteristics of the site. The team shall be provided with a map or sketch drawn to scale showing all the structures and significant geographic features at the site. The team shall also be briefed on the availability of facility personnel and records.

3. The inspection team shall then prepare an initial inspection plan that specifies the activities to be carried out by the inspection team, including the specific areas of the site to which access is desired. The inspection plan shall be provided to the challenged State Party. Its implementation shall be consistent with the provisions of Section III below, including those related to access and activities.

III. Conduct of Inspections

A. General Rules

1. The challenged State Party shall provide access within the requested perimeter as soon as possible, but in any case no later than 168 hours after specification of the location of the challenged site in order to clarify the compliance concern raised in the inspection request.

2. The challenged State Party shall make every reasonable effort to demonstrate to the inspection team that any object, building, structure, container or vehicle to which the inspection team has not had full access, is not being used for purposes related to the compliance concern raised in the inspection request.

3. In carrying out the inspection in accordance with the request, the inspection team shall use only those methods necessary to provide sufficient relevant facts to clarify doubts about compliance with the provisions of the Convention, and shall refrain from activities not relevant thereto. It shall collect and

document such evidence as is related to the compliance with the Convention by the challenged State Party but shall neither seek nor document information which is clearly not related thereto, unless the challenged State Party expressly requests it to do so. Any material collected and subsequently found not to be relevant shall not be retained.

4. The inspection team shall be guided by the principle of conducting the inspection in the least intrusive manner possible, consistent with the effective and timely accomplishment of its mission. Wherever possible, it shall begin with the least intrusive procedures it deems acceptable and proceed to more intrusive procedures only as it deems necessary.

B. Managed Access

1. In meeting the requirement to provide access within the requested perimeter, the challenged State Party will be under a treaty obligation to allow the greatest degree of access taking into account proprietary rights, its legal obligations and national security.

2. The challenged State Party shall designate the perimeter entry/exit points and the inspection team and the challenged State Party shall negotiate: the extent of access to any particular place or places within the requested and final perimeters as provided in paragraphs III.B.3-5 below; the particular inspection activities to be conducted by the inspection team; the performance of particular activities by the challenged State Party; and the provision of particular information by the challenged State Party.

3. In conformity with the relevant provisions in the Annex on the Protection of Confidential Information the challenged State Party shall have the right to take measures to protect sensitive installations and prevent disclosure of confidential data not related to chemical weapons. Such measures may include inter alia:

- Removal of sensitive papers from office spaces and securing them in safes;

- shrouding of sensitive displays, stores and equipment that cannot be secured in safes;

- shrouding of sensitive pieces of equipment, such as computer or electronic systems;

- restriction of sample analysis to appropriate element-specific on-site test except where suitable facilities are not provided;

- logging off computer services and turning off data-indicating devices;

- random selective access whereby the inspectors are requested to select a given percentage or number of buildings of their choice to inspect; the same principle can apply to the interior and content of sensitive buildings;

- giving exceptionally only individual inspectors access to certain parts of the inspection site.

4. In the event the challenged State Party restricts or denies requested access to places, activities, or information, it will be under a treaty obligation to make every reasonable effort to provide alternate means to satisfy the compliance concerns which generated the challenge inspection.

5. The challenged State Party would be under the obligation to provide access within the requested perimeter by selecting at least one of the following:

- Access on the ground for one or more members of the inspection team to portions within the requested perimeter;

- Aerial access for members of the inspection team. The challenged State Party, at its option, would provide the aircraft and pilot or rely on the inspection team's aircraft and pilot. Procedures patterned after the proposed Open Skies regime could be followed;

- Observation into the area enclosed by the requested perimeter from an elevated platform (e.g., tower, ladder, or hoist) placed or erected by the challenged State Party outside the requested perimeter;

- Use of tamper-evident sensor suites specifically designed to detect relevant chemicals as developed and approved by States Parties in accordance with the Convention. At the option of the challenged State Party, such sensor suites could be used--either by members of the inspection team or remotely--as the aerial or surface access permitted by the challenged State Party.

C. Observers (see CD/1046, paragraphs 1, 2, 3, 4, page 161, with paragraph 3 revised as follows:)

3. The observer(s) shall have the right to make recommendations to the inspection team, which the team shall take into account to the extent it deems appropriate. The observer(s) shall generally have the access to the inspection site as granted by the challenged State Party to the inspection team. However, if there is a place

into which the challenged State Party is willing to allow the inspection team or a team member to go, but into which it does not wish the observer(s) to go, the observer(s) shall remain outside. Throughout the inspection, the inspection team shall keep the observer(s) informed about the conduct of the inspection and the findings.

The following are to be developed (based on CD/1046, pages 162-63)

D. Extension of Final Perimeter

E. Duration of Inspection

IV. Departure

V. Reports

A. Contents

The inspection report shall summarize in a general way the activities conducted by the inspection team and the factual findings of the inspection team, particularly with regard to the concerns raised in the request for the challenge inspection. It shall also include an assessment by the inspection team of the degree and nature of access and cooperation granted to the inspectors and the extent to which this enabled them to fulfill their mandate. Detailed information relating to the concern cited in the inspection request shall be submitted as Appendix to the Final Report and be retained within the Technical Secretariat under the appropriate safeguards to protect sensitive information.

B. Procedures (cf. CD/1046, page 163).

CONFERENCE ON DISARMAMENT

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Ad Hoc Committee on Chemical Weapons

AUSTRALIA

On-site Chemical Analysis for Verification of Non-production of Families of Scheduled Chemicals

INTRODUCTION

One of the problems in defining the chemicals to be monitored under the future Chemical Weapons Convention (CWC) is that certain classes of chemical warfare (CW) agents cannot be represented as a short list of chemicals.

In order to overcome this problem, the schedules of chemicals to be covered under the CWC contain some individual chemicals and some families of chemicals. For example, there are several families of CW agents in Schedule 1 which contain several thousand members. For the majority of these chemicals, analytical data are not available. This raises the question of how to verify the "non-production" of such chemicals.

It has recently been suggested that without a defined list of banned chemicals and pre-recorded identification data, the analyst would be faced with the horrendous task of identifying all compounds containing phosphorus, including intermediates, by-products, and impurities, in order to decide whether or not they belonged to the families covered by Schedule 1.

In this working paper, we re-examine the problem of detection of family members. Australia has developed an alternative approach that would be suitable for rapid on-site screening of family members of scheduled chemicals, and would not result in the identification of non-scheduled chemicals. The example considered here is of one family, the alkyl methylphosphonofluoridates which are included in item 1 of Schedule 1. Nevertheless, the approach we have developed should be applicable to other families.

ON-SITE SCREENING PROCEDURE

It is recognized that on-site inspections can be conducted more efficiently if analysis is done on-site, especially if rapid screening methods can be used. On-site analysis also greatly reduces the risk of loss of

sensitive technical information, especially if the on-site analytical procedures are designed to indicate the presence of relevant scheduled chemicals, and provide no information on chemicals of no relevance to the CWC.

However, to be useful, a screening method should be very reliable - an unreliable screening method could miss the detection of violations (by "false negatives"), and cause unnecessary ambiguities and suspicions (by "false positives").

Two of the methods which have been suggested as suitable for on-site chemical analysis are GC-MS (with either EI or CI) and GC with selective detectors, including the flame photometric detector for phosphorus or sulphur containing chemicals. We have chosen these two methods as the basis of a rapid screening procedure (see Figure 1).

STEP 1

The initial step in our procedure is GC-MS(EI) analysis. The mass spectrum of each component is compared with the library of mass spectra of individual scheduled chemicals in the analytical database. We have chosen to use computer matching of the eight most intense peaks (using a logarithmic standardization procedure) because this method gives very reliable identification of known CW agents.

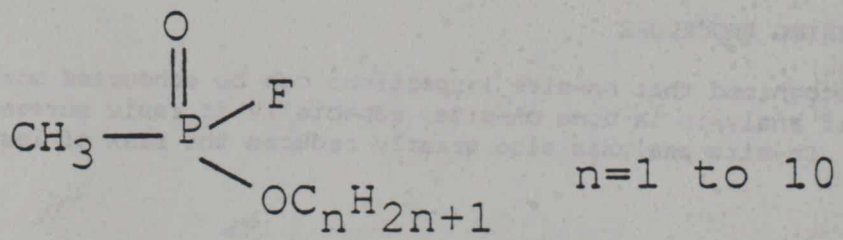
STEP 2

If the unknown is not identified as one of the scheduled chemicals in the database, the EI mass spectrum of the unknown is tested with appropriate pattern classifiers. We have developed very reliable pattern classifiers for the presence of a number of structural features (Reference 1), including the structural features present in the families of chemicals in Schedule 1. One method that we have used is the "learning machine" (References 2 and 3).

STEP 3

If the pattern classifier gives an affirmative response to a particular family of chemicals, the next step is to determine from the CI mass spectrum of the unknown, whether the molecular weight corresponds with the molecular weight of the members of that particular family.

It is fortunate that even though there are 879 different chemicals represented in the family



there are only 10 different molecular weights (see Table 1).

STEP 4

If the MW determined in step 3 is consistent with the unknown belonging to the particular family, then determine whether the unknown contains the appropriate heteratoms using gas chromatography with selective detectors.

RESULTS TO DATE

The system as outlined in Figure 1 has been developed with mass spectral data from 50 alkyl methylphosphonofluoridates that have been synthesized from the most readily obtainable alcohols, and a database containing approximately 10,000 mass spectra of other chemicals. The system correctly classified all of these chemicals as either "alkyl methylphosphonofluoridate" or "not alkyl methylphosphonofluoridate".

We are presently in the process of extending the database with mass spectra from an additional 20 different alkyl methylphosphonofluoridates (obtained from less readily available alcohols), and by using a larger database of other chemicals.

We are also developing a similar classification system for the dialkyl methylphosphonates family (alkyl CH3 to C10H21) which are covered under Item 1 of Schedule 2A.

CONCLUDING COMMENTS

Based on our results to date, and a survey of mass spectral data available for other families in Schedule 1, we are confident that it will be possible to develop similar classification schemes for the other families of chemicals in Schedule 1 and related chemicals in Schedule 2A.

However, we are not suggesting that we have necessarily achieved the most efficient scheme. For example, with some families it may be possible to replace the learning machine classifier in Step 2 with a simple question "Is peak height of M/Z [ ] greater than [ ]%?".

We hope that this preliminary communication of our work will encourage other laboratories to consider development of classifiers for other families of scheduled chemicals.

REFERENCES AND NOTES

1. Mathews, R.J., "Preliminary Classification of Mass Spectral Patterns using a Simplified Learning Machine", Aust. J. Chem. 1973, 26, 1955-61.

2. In the learning machine method, each mass spectrum is represented as a vector X, which when multiplied by a weight vector W gives a scalar S, such that S is positive for all members of one category (e.g. presence of methylphosphonofluoridate moiety) and negative for all others. The weight vector W is generated in iterative fashion by repeatedly classifying a "training set" of mass spectral patterns, with negative feedback error correction being applied to W when any pattern is assigned to the wrong category.

3. Nilsson, N.J., "Learning Machines", (McGraw-Hill: New York, 1965).

4. Henze, H.R., and Blair, C.M., "The Number of Structurally Isomeric Alcohols of the Methanol Series", J. Amer. Chem. Soc., 1931, 53, 3042-3046.

FIGURE 1. SCREENING SYSTEM FOR CHEMICALS IN FAMILIES

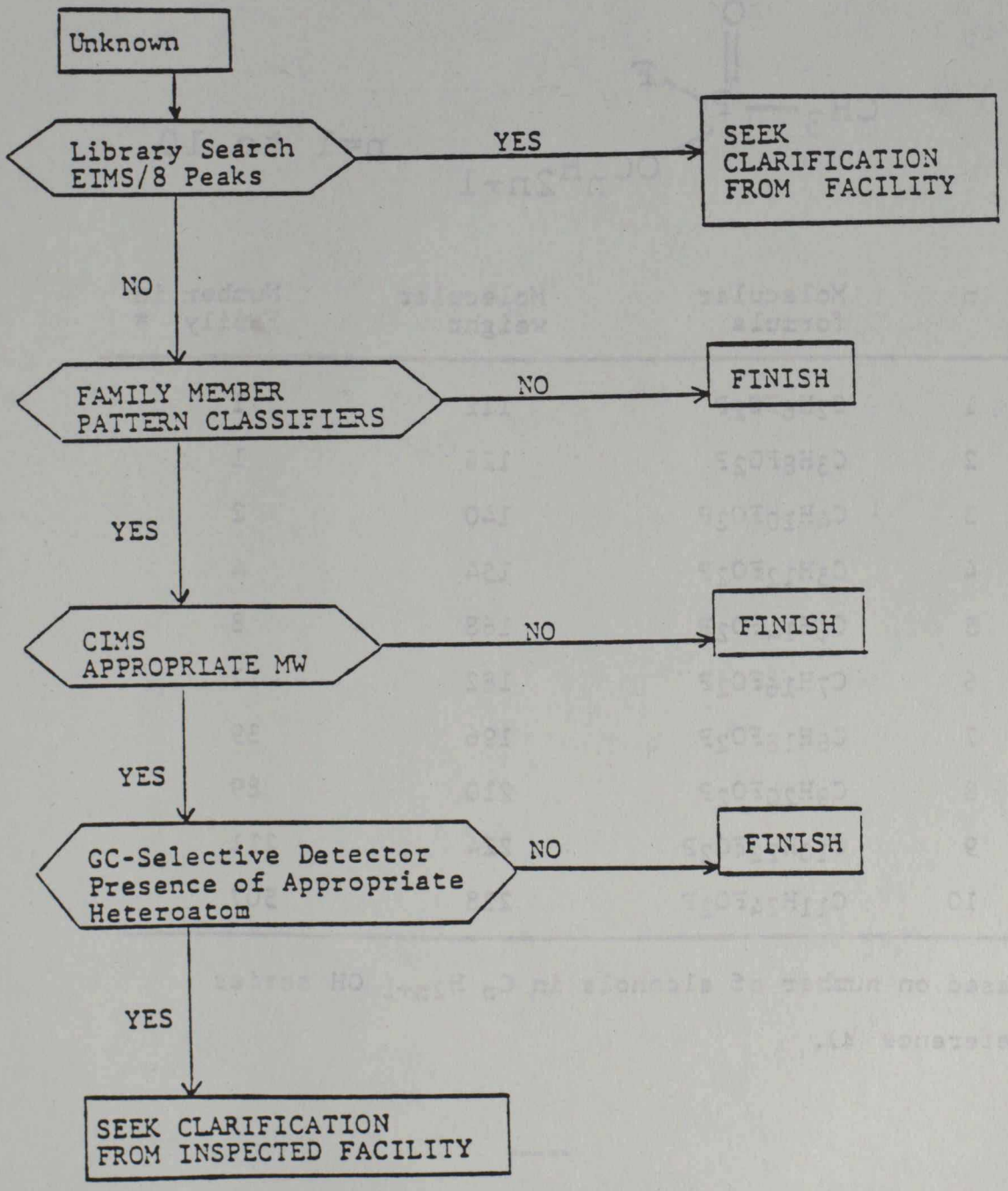
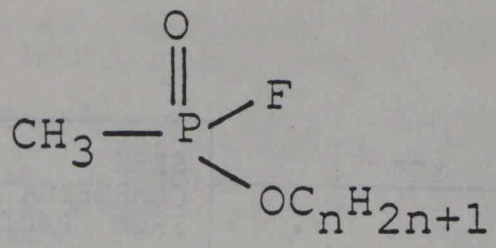


TABLE 1. MEMBERS IN ALKYL METHYLPHOSPHONOFLUORIDATE FAMILY



n=1 to 10

n	Molecular formula	Molecular weight	Number in Family *
1	$C_2H_6FO_2P$	112	1
2	$C_3H_8FO_2P$	126	1
3	$C_4H_{10}FO_2P$	140	2
4	$C_5H_{12}FO_2P$	154	4
5	$C_6H_{14}FO_2P$	168	8
6	$C_7H_{16}FO_2P$	182	17
7	$C_8H_{18}FO_2P$	196	39
8	$C_9H_{20}FO_2P$	210	89
9	$C_{10}H_{22}FO_2P$	224	211
10	$C_{11}H_{24}FO_2P$	238	507

* Based on number of alcohols in $C_n H_{2n+1} OH$ series (Reference 4).

Poland, USSR	CD/CW/WP.354	Joint report on a trial inspection on request NOT REPRODUCED (see WP volume)	Also issued as CD/1093 6.8.91

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CONFERENCE ON DISARMAMENT

CD/CW/WP.355
6 August 1991

ENGLISH ONLY

Ad Hoc Committee on Chemical Weapons

THE NETHERLANDS

THE USE OF THERMOSPRAY-LIQUID CHROMATOGRAPHY MASS SPECTROMETRY FOR THE VERIFICATION OF CHEMICAL WARFARE AGENTS

Working Paper

SUMMARY

Thermospray-liquid chromatography mass spectrometry (TSP-LC-MS) is a relatively new analytical technique which proved to be useful for the verification of chemical warfare agents and their polar degradation products in aqueous solutions. The principles of the technique are described and comparisons are made with other forms of mass spectrometric analysis. A survey is presented of the results obtained so far at the Prins Maurits Laboratory TNO. The analysis of organophosphorous nerve agents and their hydrolysis products (organophosphorous acids) in various types of water is described. Special attention is paid to the nerve agent VX. Direct analysis of vesicants in water by TSP-LC-MS is limited. However, analysis of their hydrolysis products is possible. Finally, the use of TSP-LC-MS for the analysis of other compounds of chemical warfare interest (toxins) is indicated.

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CONTENTS

INTRODUCTION

For a successful implementation of a Chemical Weapons Convention a continuous development of analytical procedures to verify the presence or absence of chemical warfare (CW) agents will be necessary. Analytical procedures based on spectrometry are essential for providing the unequivocal proof of the presence of CW agents in samples taken during an inspection or in the event of an alleged use investigation. Owing to its sensitivity, and selectivity mass spectrometry (MS) is at present the most suitable technique for this purpose, especially if environmental and biological samples need to be analysed. Combined with capillary column gas chromatography (GC) as a separation technique, very complicated samples can be analysed for the possible presence of CW agents in a relatively short time. Despite the great analytical potential of a GC-MS combination, it has two major drawbacks. In the first place direct analysis of water samples is difficult. Water samples need to be extracted in order to transfer the analytes from the water phase into an organic solvent, which is a much more suitable medium to inject into a GC-MS combination. Secondly, polar decomposition products of CW agents, e.g. organophosphorus acids, do not elute properly from a GC-column. Therefore these compounds need to be converted into volatile, non-polar derivatives before the analysis takes place. These two drawbacks may be overcome by using liquid chromatography (LC) as a separation technique in combination with MS. However, in contrast to capillary column GC where practically no interfacing problem with a modern mass spectrometer exists, the coupling of LC with MS is troublesome. The main reason is the mass flow through an LC column which is at least a factor of 1000 higher compared with a capillary GC column. Various types of LC-MS interfaces have been developed, each with their own characteristics. Names such as moving belt, particle beam, direct liquid introduction (DLI), thermospray (TSP), ion/electrospray and continuous flow fast atom bombardment (FAB) are associated with the different ways of introducing the LC eluent into a mass spectrometer [1, 2]. Each interface has its own area of application. The moving belt and particle beam interfaces are more suitable for normal phase LC using organic solvents as eluents. Ion/electrospray and continuous flow FAB were developed for the analysis of ionic biological molecules such as peptides and nucleotides. DLI has become obsolete and has been more or less replaced by TSP. Generally, TSP-LC-MS is best suited for polar compounds of medium molecular weight (100-1000 amu) which are analysed by reversed phase LC using an eluent composition with a large water content. The combination which was introduced around 1985 by Vestal et al. [3] has become quite successful according to the number of published applications in various areas of chemistry. In this paper, results obtained at the Prins

Maurits Laboratory TNO (PML-TNO) of the use of this relatively new technique for the analysis of CW agents and their polar decomposition products will be described.

2 EXPERIMENTAL

All the analyses presented in this paper were performed on a Nermag R10-10C quadrupole mass spectrometer which was coupled with a liquid chromatographic system via a Vestec thermospray interface. LC separations were carried out on reversed phase columns (250 x 5 mm) which were packed in the laboratory with LiChrosorb C18 (7 µm particles). For a more detailed description of the instrumentation used, refer to the literature [4, 5].

3 THERMOSPRAY-LIQUID CHROMATOGRAPHY MASS SPECTROMETRY

The TSP-interface between a liquid chromatograph and a mass spectrometer consists of a metal capillary tube (internal diameter 0.1 - 0.15 mm). The LC-eluent is forced through this tube and heated to a precise temperature, resulting in a spray of fine liquid droplets at the end of the tube. Temperatures at the tip of the capillary depend on the eluent composition and have values between 150 - 200 °C. An electrolyte, normally 0.1 M ammonium acetate, is added to the LC-eluent. Introducing the spray into the vacuum of a mass spectrometric ion source results in the evaporation of the droplets, followed by the formation of ions of the analytes. Although the exact nature of this process is still under investigation, gas phase reactions between the added electrolyte and the analytes play an important role.

The TSP-interface is designed to operate with conventional reversed phase LC-columns (length 10 - 25 cm, ID 0.4 - 0.5 cm) using a flow rate of 1 - 1.5 ml/min.. Highest sensitivity is obtained when the eluent contains a high water content. Unsuccessful attempts were made to combine the TSP-interface with LC-columns using smaller flow rates. In contrast to conventional LC-detectors, only volatile buffers can be used. Buffers containing inorganic salts such as the well-known potassium hydrogen sulphate buffers cannot be used due to the fact that they will block the interface. Blocking of the interface may also occur by silica particles originating from dissolved column packing material. This effect may take place especially at pH values of the eluent above 7.

Mass spectra obtained under TSP conditions are different from those obtained with the well-known electron impact (EI) and chemical ionization (CI) modes recorded with a GC-MS instrument. These differences can be illustrated with the nerve agent VX. Intensive fragmentation of the molecule is observed in its EI mass spectrum (Figure 1A) leading to m/z 114 as the base peak in the spectrum. No molecular weight information is obtained and the highest observed fragments

(m/z 252, 224) are very weak. In addition to an abundant protonated molecular ion at m/z 268, intensive fragmentation is still observed in the ammonia CI mass spectrum (Figure 1B).

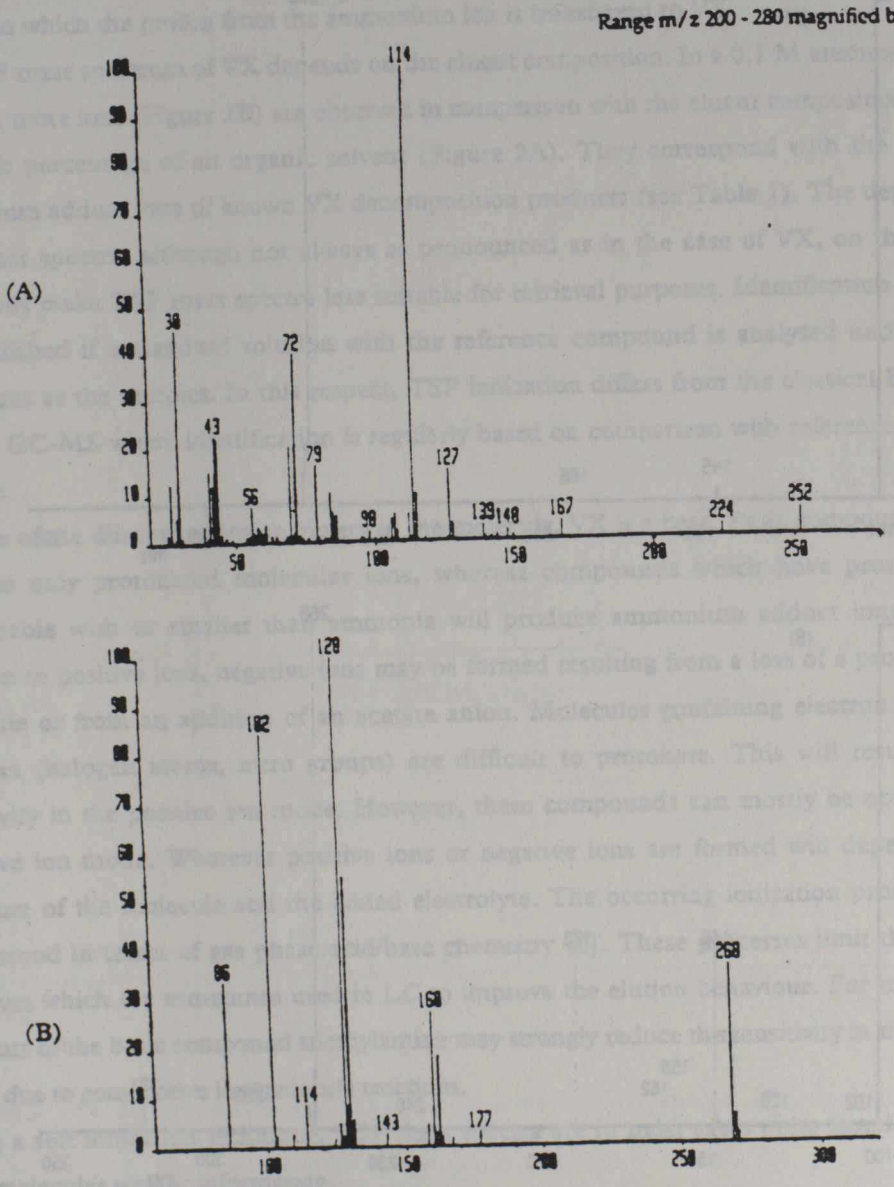
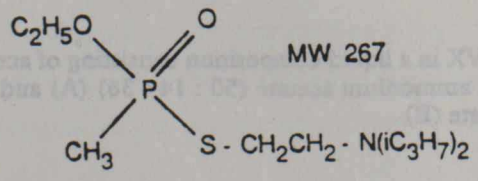


Figure 1 Electron impact (A) and ammonia chemical ionization mass spectrum (B) of VX



MW 267

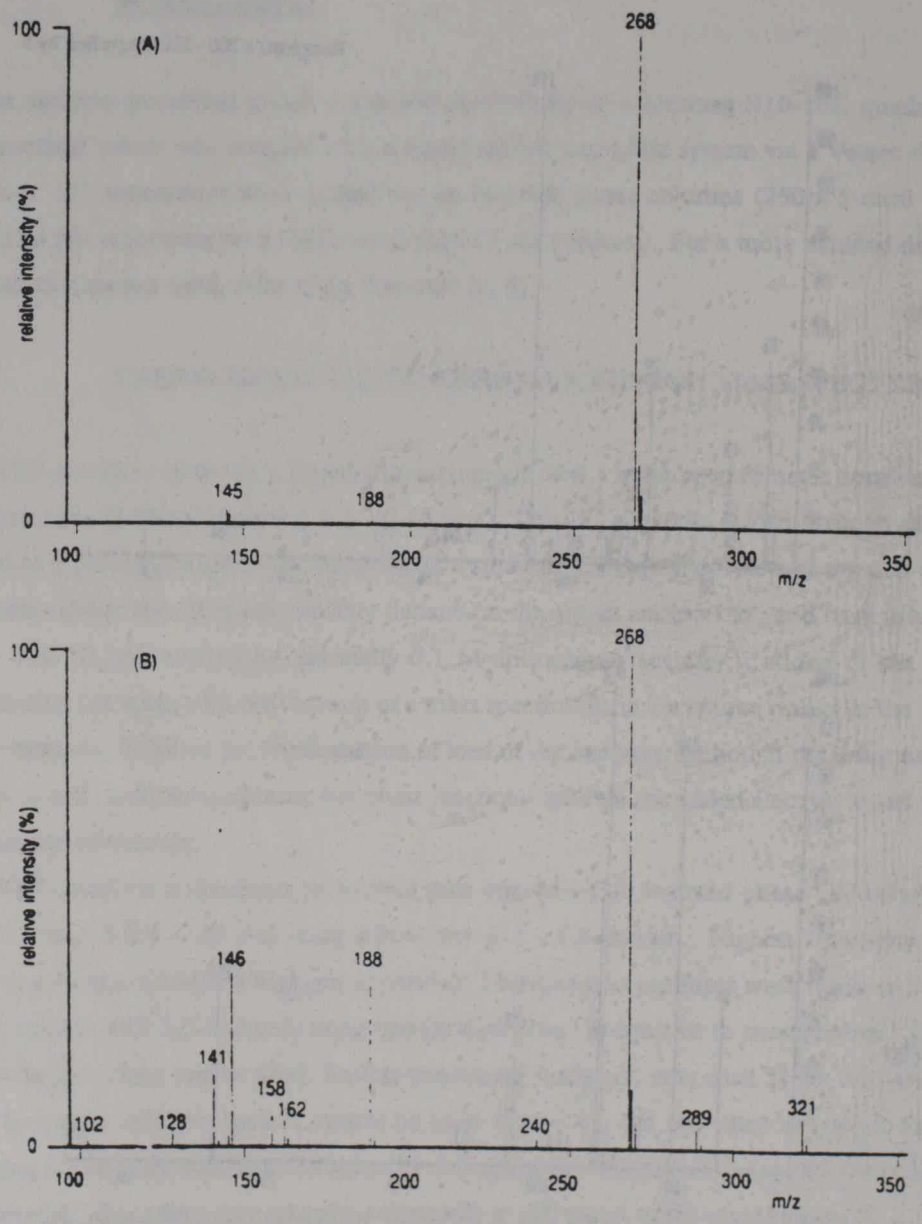


Figure 2 TSP mass spectrum of VX in a liquid composition consisting of acetonitrile, methanol and 0.089 M ammonium acetate (50 : 14 : 36) (A) and in 0.1 M ammonium acetate (B)

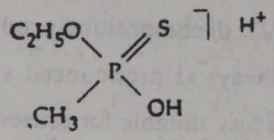
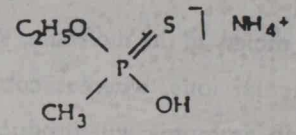
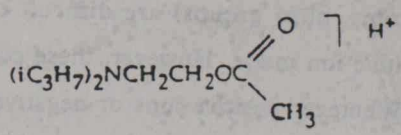
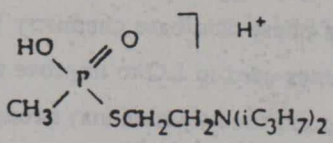
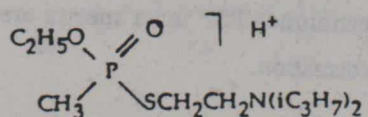
Although there are similarities between ammonia CI and TSP mass spectra obtained by ionization with an ammonium acetate buffer, TSP is a much softer ionization technique. In the case of VX, this leads to a mass spectrum almost exclusively existing of the protonated molecular ion at m/z 268 (Figure 2A). In the case of VX, TSP may be considered as the ideal chemical ionization process in which the proton from the ammonium ion is transferred to VX.

The TSP mass spectrum of VX depends on the eluent composition. In a 0.1 M ammonium acetate solution, more ions (Figure 2B) are observed in comparison with the eluent composition consisting of a high percentage of an organic solvent (Figure 2A). They correspond with the proton and ammonium adduct ions of known VX decomposition products (see Table 1). The dependence of TSP mass spectra, although not always as pronounced as in the case of VX, on the recording conditions make TSP mass spectra less suitable for retrieval purposes. Identification can only be accomplished if a standard solution with the reference compound is analysed under the same conditions as the samples. In this respect, TSP ionization differs from the classical EI ionization used in GC-MS where identification is regularly based on comparison with reference spectra in a data file.

Because of the diisopropylamine moiety in the molecule, VX is a base. Basic compounds normally produce only protonated molecular ions, whereas compounds which have proton affinities comparable with or smaller than ammonia will produce ammonium adduct ions as well. In addition to positive ions, negative ions may be formed resulting from a loss of a proton from the molecule or from an addition of an acetate anion. Molecules containing electron withdrawing moieties (halogen atoms, nitro groups) are difficult to protonate. This will result in a poor sensitivity in the positive ion mode. However, these compounds can mostly be observed in the negative ion mode. Whatever positive ions or negative ions are formed will depend upon the structure of the molecule and the added electrolyte. The occurring ionization processes can be understood in terms of gas phase acid/base chemistry [6]. These processes limit the number of additives which are sometimes used in LC to improve the elution behaviour. For instance, small amounts of the basic compound triethylamine may strongly reduce the sensitivity in the positive ion mode due to competitive ion/molecule reactions.

Being a soft ionization technique, TSP mass spectra are in most cases quite simple and provide only molecular weight information.

Table 1 Structures of fragments found in the TSP mass spectra of VX

m/z 102	$(iC_3H_7)_2NH\rceil H^+$
m/z 128	$(iC_3H_7)_2NCH{=}CH_2\rceil H^+$
m/z 141	
m/z 145	$(iC_3H_7)_2NCH_2CH_2NH_2\rceil H^+$
m/z 146	$(iC_3H_7)_2NCH_2CH_2OH\rceil H^+$
m/z 158	
m/z 162	$(iC_3H_7)_2NCH_2CH_2SH\rceil H^+$
m/z 188	
m/z 240	
m/z 268	
m/z 289	$(iC_3H_7)_2NCH_2CH_2SCH_2CH_2N(iC_3H_7)_2\rceil H^+$
m/z 321	$(iC_3H_7)_2NCH_2CH_2SSCH_2CH_2N(iC_3H_7)_2\rceil H^+$

For identification purposes this may be considered as a disadvantage because a TSP mass spectrum containing only one or two peaks can hardly be called a spectrum. Therefore special techniques have been developed in order to put more energy into the ions using a bombardment of electrons (filament-on technique) or a discharge ionization. Moreover, the resident time in the ion source can be increased using a repeller as an ion retarding device resulting in collision-induced dissociation [7]. The effect of a discharge ionization and various repeller voltages on the TSP mass spectrum of VX is shown in Figure 3.

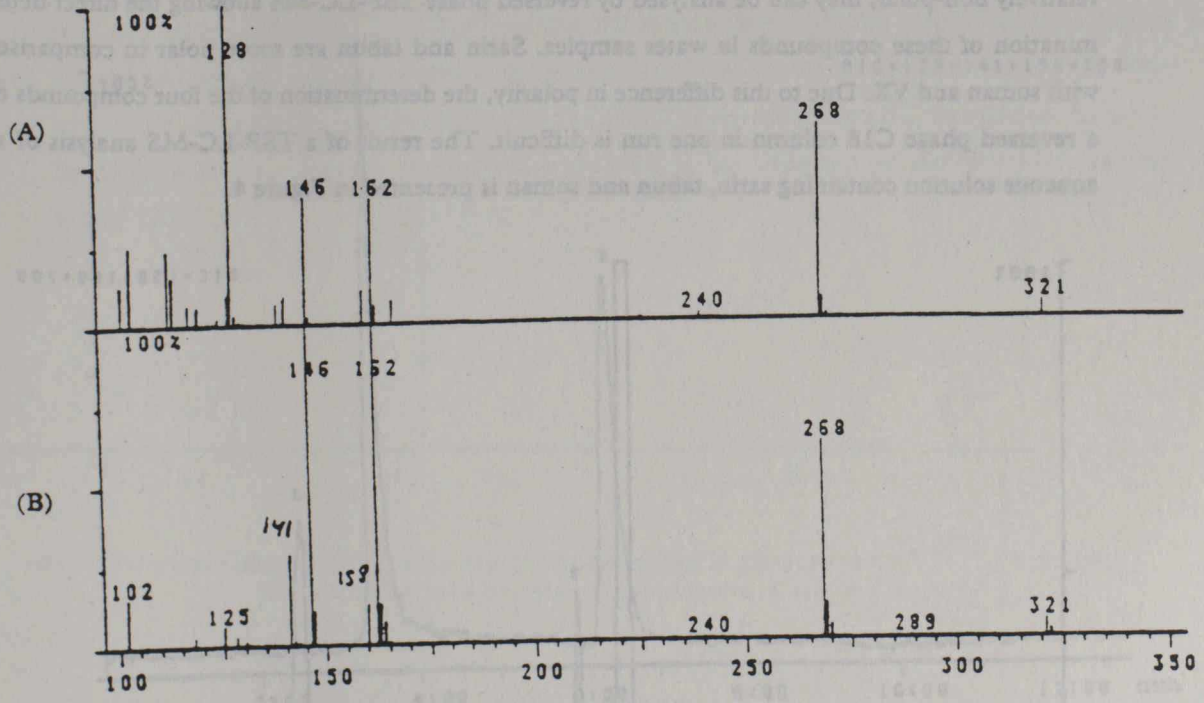


Figure 3 Effect of discharge ionization and repeller voltage on the TSP mass spectrum of VX in a liquid composition consisting of methanol and water (50 : 50).
A : Repeller = 200 V B : Repeller = 0 V ◆

Due to the induced fragmentation, a kind of mass spectrum is obtained, which is as characteristic as an EI or CI spectrum. However, the dependence on the recording conditions remains. Fragmentation may of course also be achieved by using collision-induced dissociation of a protonated molecular ion in a collision cell of an MS/MS combination.

The small information content of the TSP mass spectra obtained by using ammonium acetate buffer ionization may also be considered an advantage. No more is needed for the verification of the absence of certain compounds (negative identification). Samples may be screened for a number of compounds of CW interest by switching from one protonated molecular ion to another. In this way, TSP-LC-MS is essentially the same as GC or LC combined with a specific phosphorus or sulphur detector. However, a mass spectrometer is a more universal detector which may be used for a variety of compounds of CW interest.

4 ANALYSIS OF ORGANOPHOSPHORUS NERVE AGENTS AND THEIR DECOMPOSITION PRODUCTS

Although the four well-known organophosphorus nerve agents sarin, tabun, soman and VX, are relatively non-polar, they can be analysed by reversed phase TSP-LC-MS allowing the direct determination of these compounds in water samples. Sarin and tabun are more polar in comparison with soman and VX. Due to this difference in polarity, the determination of the four compounds on a reversed phase C18 column in one run is difficult. The result of a TSP-LC-MS analysis of an aqueous solution containing sarin, tabun and soman is presented in Figure 4.

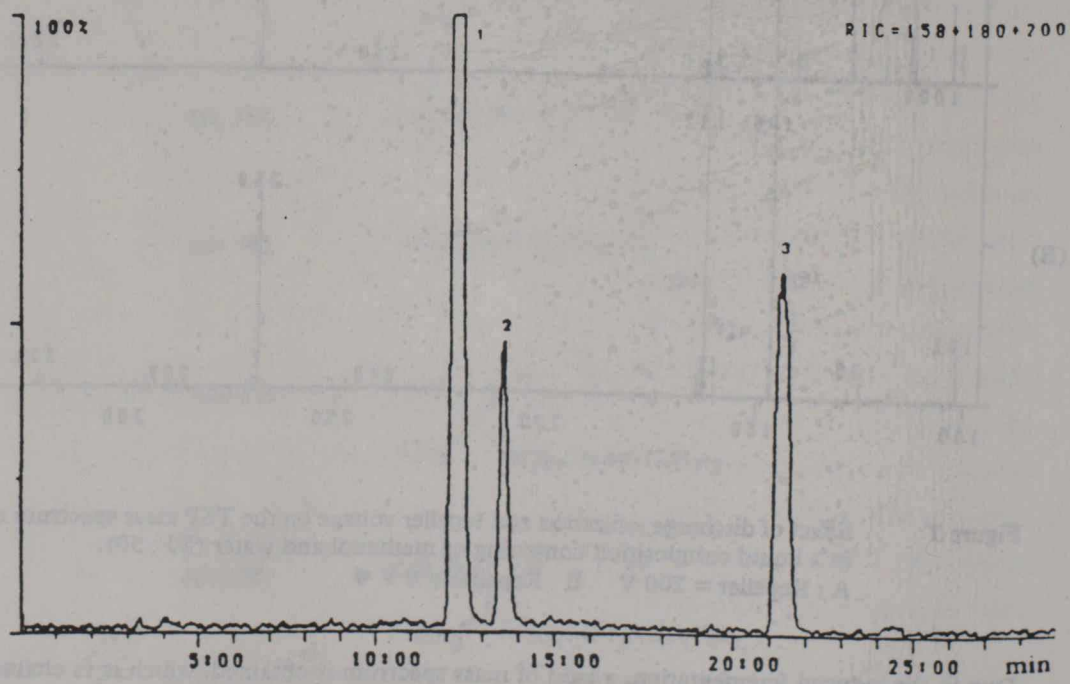


Figure 4 Analysis of a mixture of sarin (1), tabun (2) and soman (3) by gradient elution. Eluent : 0.1 M ammonium acetate - methanol (70 : 30) to 0.1 M ammonium acetate - methanol (30 : 70) in 20 min.

Gradient elution had to be performed to analyse sarin and tabun together with soman in one LC-run. For routine applications, both sarin and tabun may better be chromatographed using a mixture of methanol and 0.1 M ammonium acetate (50 : 50), whereas for soman, a higher percentage of organic modifier (70%) is preferable. The hydrolysis rate of the G-agents is such that providing the pH of the water is below 7, the compounds may still be detected within one day after their disposal. In Figure 5 the resultant chromatogram of an analysis of Rhine water (pH 7) spiked with sarin at a relatively high concentration level of 2.5 µg/ml is presented.

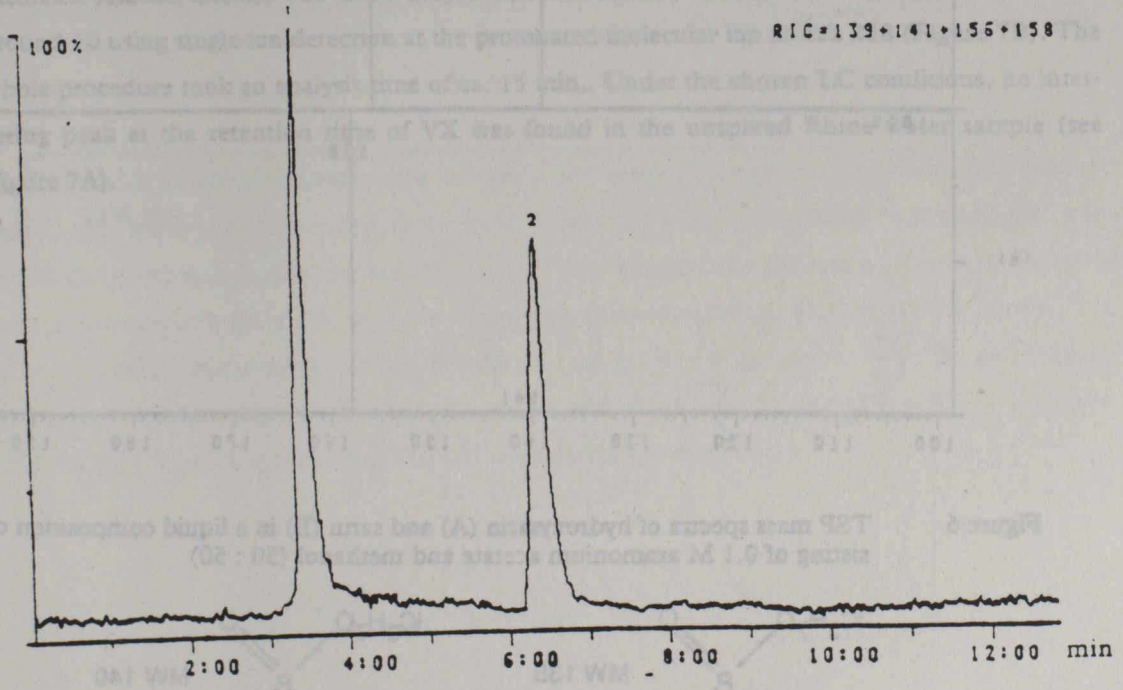


Figure 5 Analysis of a Bond Elut C18 cartridge effluent containing hydroxysarin (1) and sarin (2).
Eluent : 0.1 M ammonium acetate - methanol (50 : 50)

The Rhine water aliquot was sampled ca. 15 min. after the spiking by forcing 10 ml of water through a C18 cartridge with a syringe. After a storage time of 20 h at room temperature, the cartridge was eluted with 1 ml of methanol of which 40 µl was injected into the TSP-LC-MS system. In contrast to GC-methods, no special precautions need to be taken to remove the residual water present on the cartridge before the instrumental analysis. In addition to sarin, its main hy-

drolysis product, hydroxysarin, was found in approximately the same amount. The TSP mass spectra of both compounds obtained by ammonium acetate ionization are presented in Figure 6. The difference in ratio between the protonated molecular ion and the ammonium adduct ion of the two compounds reflects their difference in proton affinities.

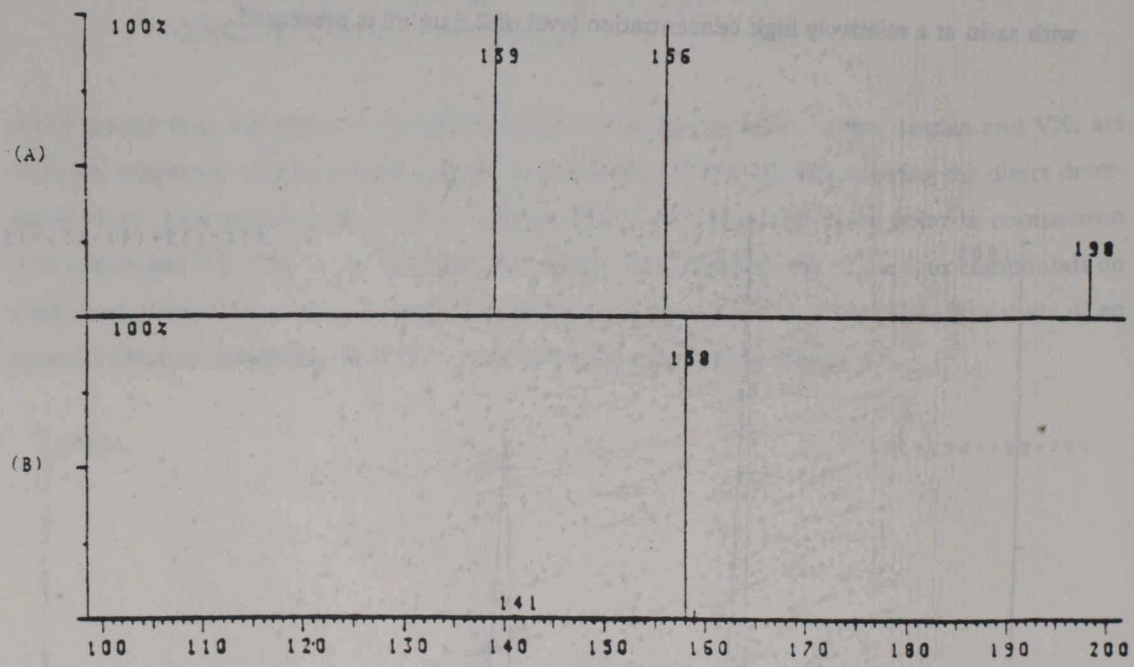
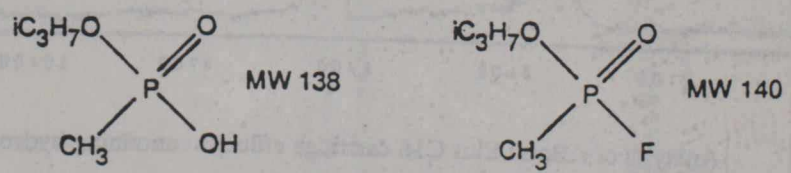


Figure 6 TSP mass spectra of hydroxysarin (A) and sarin (B) in a liquid composition consisting of 0.1 M ammonium acetate and methanol (50 : 50)



Although storage on a C18 cartridge may provide a certain preservation effect, the use of such cartridges in water sampling, extensively described in a number of Norwegian reports [8], is especially advantageous due to the relatively easy way of sample transportation. Of course water samples may also be transported to a laboratory in bottles or even in polymeric bags and directly

analysed by TSP-LC-MS providing that the concentration of the analytes exceeds the 10 ng/ml level. Below this level, preconcentration will be necessary which can be carried out most effectively using a C18 cartridge.

Preconcentration of the analytes may be very effective if the compound is less polar. This is the case for soman and especially for VX. Owing to the basic diisopropylamino group of VX, its retention on C18 material depends on the pH. At a pH of around 7, over 200 ml of VX contaminated water can be passed through a cartridge before breakthrough occurs. This allows the determination of VX in water at a relatively low level, providing that a relatively large water sample is available. Experiments were carried out with Rhine and Meuse water spiked with VX at a concentration level of 0.1 ng/ml. An amount of 50 ml was forced through a cartridge 15 min. after spiking, followed by elution of the cartridge in reverse direction with 1 ml of methanol. After injecting 40 µl of the methanol solution into the TSP-LC-MS system, VX could be detected with a signal-to-noise ratio of around 10 using single ion detection at the protonated molecular ion at m/z 268 (Figure 7B). The whole procedure took an analysis time of ca. 15 min.. Under the chosen LC conditions, no interfering peak at the retention time of VX was found in the unspiked Rhine water sample (see Figure 7A).

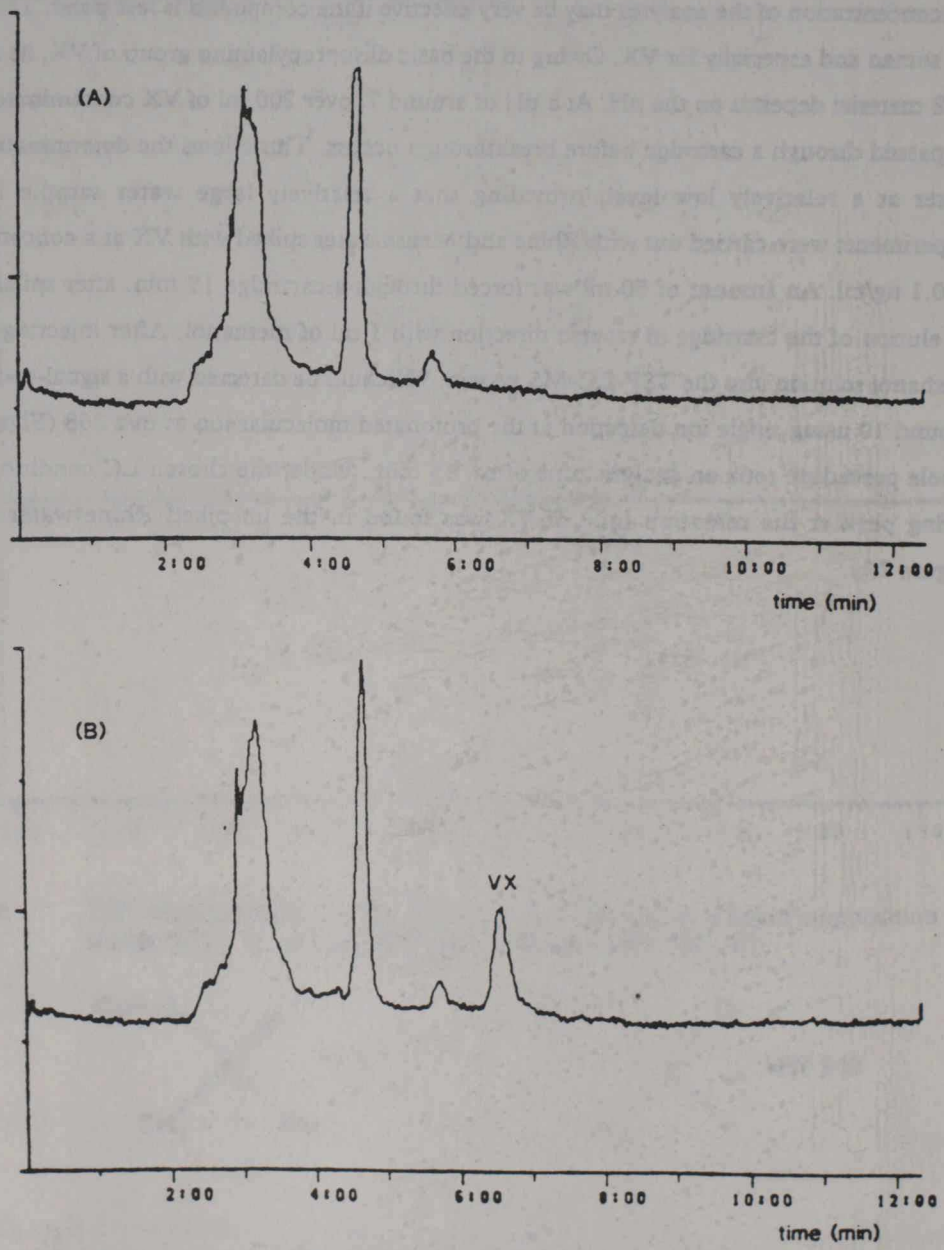


Figure 7 Determination of VX in a 50 ml Rhine water sample
A : unspiked Rhine water
B : Rhine water spiked with 0.1 ng/ml of VX
Eluent : acetonitrile - methanol - 0.25 M ammonium acetate (70 : 20 : 10)

Similar results were obtained with water from the river Meuse. The used LC conditions reflect the relative apolarity of VX in comparison with sarin. In order to elute VX from the C18 column, high quantities of methanol or acetonitrile are necessary. In the case of methanol, a mixture of methanol and 0.1 M ammonium acetate (80 : 20) adjusted to pH 8 must be used. However under these conditions co-elution of VX occurs with the well-known plasticizer tri-n-butyl phosphate. This compound was found in sometimes relatively high concentrations (up to 10 ng/ml) in polluted European rivers [9, 10]. To prevent interference, elution may better be carried out using a mixture of acetonitrile, methanol and 0.25 M ammonium acetate (70 : 20 : 10).
It is obvious that analysis procedures developed for water samples may also be used for other matrices. Especially clayey types of soil containing relatively large amounts of water may be extracted with water followed by TSP-LC-MS analysis. This was successfully carried out during the first Round Robin verification exercise [11] where soil spiked with VX had to be analysed. The TSP-LC-MS analysis provided a good alternative to the analyses based on GC-techniques.

Except for VX which is relatively stable in water (the half life time of VX is approximately 1 week at pH 8) [12] nerve agents hydrolyse to the more polar acids within a few days. As already indicated above for hydroxysarin, organophosphorus acids may be chromatographed on the same reversed phase C18 column using the principle of ion-pair chromatography. The electrolyte ammonium acetate, used for the ionization of the molecules in the ion source also serves as an ion-pair reagent during chromatography. The chromatograms obtained after the TSP-LC-MS analysis of a mixture of 13 organophosphorus acids (see Table 2) are presented in Figure 8.

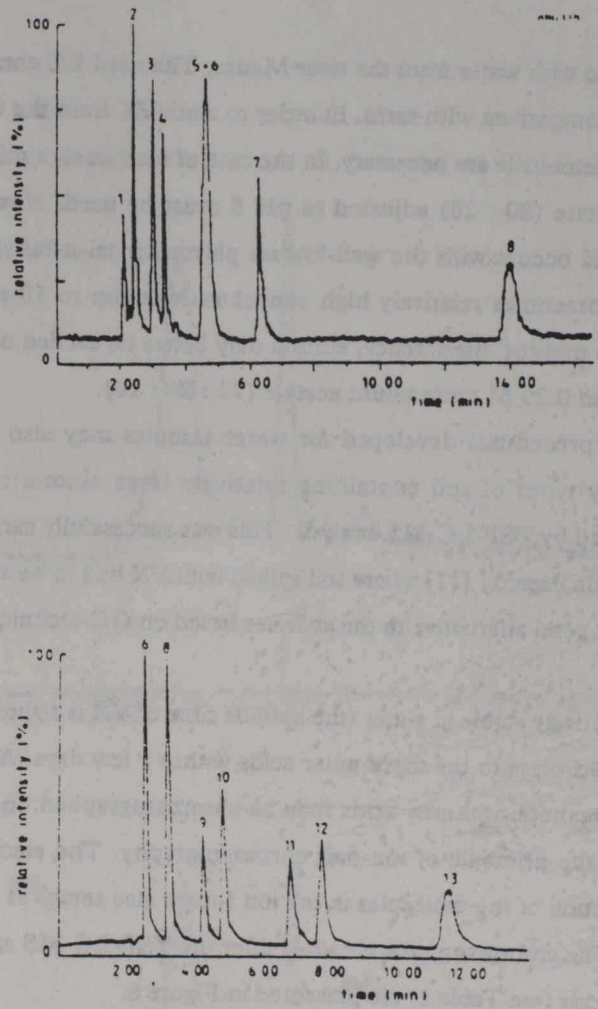
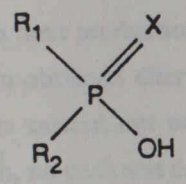


Figure 8 Analysis of a mixture of organophosphorus acids. See Table 2 for compound numbers.
A : Eluent : 0.1 M ammonium acetate
B : Eluent : 0.1 M ammonium acetate - methanol (70 : 30)

Table 2 Organophosphorus acids used as test compounds



No.	Acid	R1	R2	X	MW
1	Methylphosphonic	OH	CH_3	O	96
2	Dimethyl phosphoric	CH_3O	CH_3O	O	126
3	Ethyl methylphosphonic	C_2H_5O	CH_3	O	124
4	Dimethyl thiophosphoric	CH_3O	CH_3O	S	142
5	Ethyl methylthiophosphonic	C_2H_5O	CH_3	S	140
6	Isopropyl methylphosphonic	$(CH_3)_2CHO$	CH_3	O	138
7	Diethyl phosphoric	C_2H_5O	C_2H_5O	O	154
8	Diethyl thiophosphoric	C_2H_5O	C_2H_5O	S	170
9	Cyclopentyl methylphosphonic	C_5H_9O	CH_3	O	164
10	Diisopropyl phosphoric	$(CH_3)_2CHO$	$(CH_3)_2CHO$	O	182
11	Cyclohexyl methylphosphonic	$C_6H_{11}O$	CH_3	O	178
12	Diisopropyl thiophosphoric	$(CH_3)_2CHO$	$(CH_3)_2CHO$	S	198
13	Pinacolyl methylphosphonic	$(CH_3)_3CCH(CH_3)O$	CH_3	O	180

These compounds are the decomposition products of nerve agents and related pesticides. The acids derived from nerve agents contain a methyl group directly linked to a phosphorus atom. Two eluent systems were chosen : a) 0.1 M ammonium acetate and b) a mixture of 0.1 M ammonium acetate and methanol (70 : 30). If all the acids need to be analysed in one LC run, gradient elution will be necessary. Although the more hydrophilic acids eluted very fast (Figure 7A, peaks 1-4), separation was still complete. The hydrolysis product of soman (Figure 8B, peak 13) was the most hydrophobic of the acids studied. Both the proton and the ammonium adduct ions were observed in the positive ion TSP mass spectrum of hydroxysoman (Figure 9A). The ratio of both pseudomolecular ions depends on the temperature of the vaporizer and forms another example of the fact that TSP mass spectra are variable. Fragments derived from methylphosphonic acid (m/z 97 and 114) were also noticed. The intensities of these last fragments increased with increasing ion source

temperature, indicating that decomposition of hydroxysoman into this compound may take place. Organophosphorus acids may also be detected with comparable sensitivity in the negative ion mode at the ion resulting from a loss of a proton. As an example, the negative ion TSP mass spectrum of hydroxysoman is presented in Figure 9B.

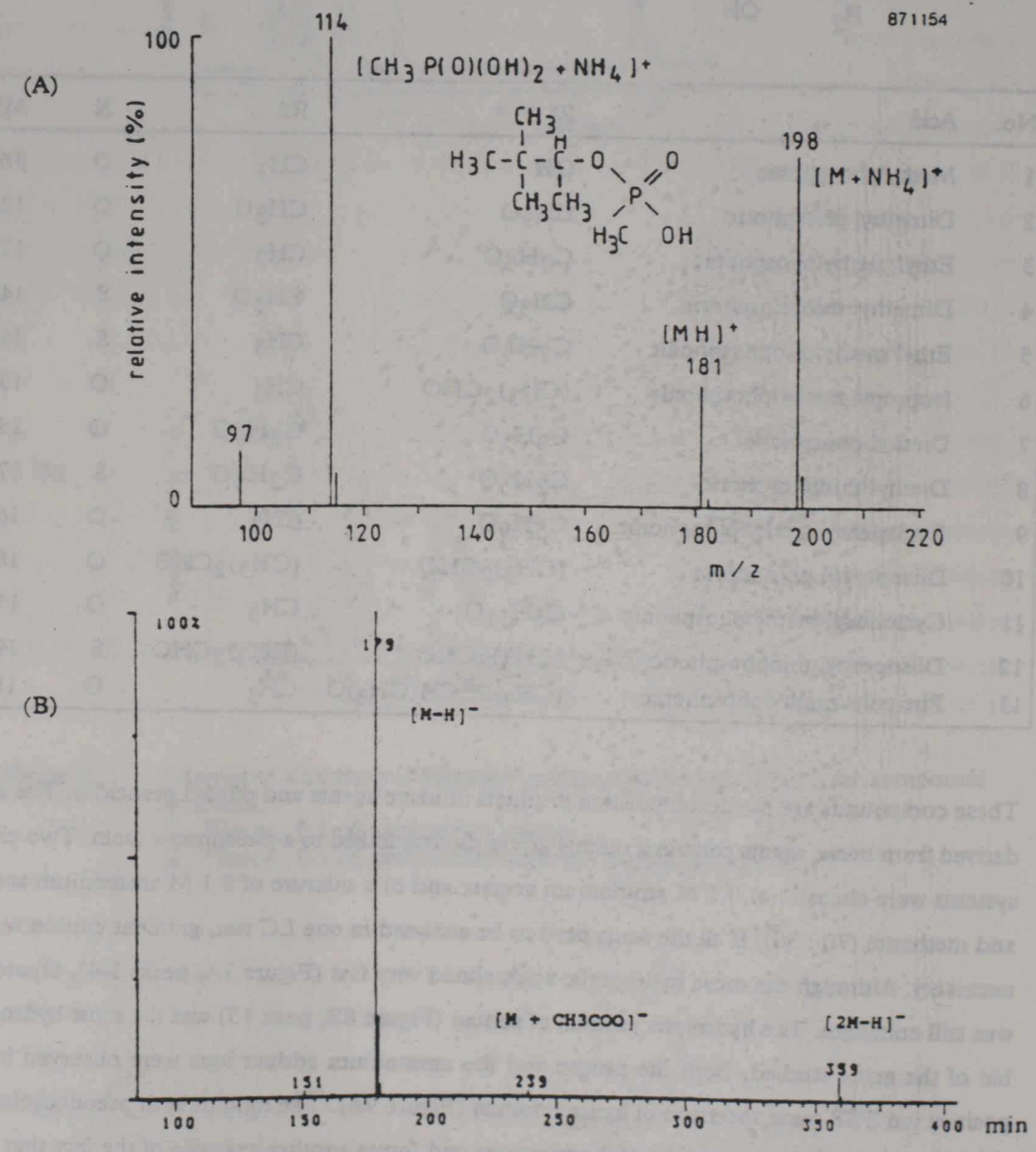


Figure 9 Positive (A) and negative TSP (B) mass spectrum of hydroxysoman in a liquid composition consisting of 0.1 M ammonium acetate and methanol (70 : 30)

The determination of organophosphorus acids in water samples by TSP-LC-MS proceeds much faster in comparison with procedures based on GC-techniques. Generally, for the latter procedures, the acids had to be isolated from the aqueous phase followed by derivatization. To test the TSP-LC-MS procedure on real samples, the waste water of a chemical factory using organophosphorus compounds in their production process was spiked with hydroxysoman at a level of 50 ng/ml. The chromatogram obtained after injecting 40 µl of the spiked waste water is shown in Figure 10B. Detection was carried out on the ammonium adduct ion (m/z 198). At the retention time of hydroxysoman, no peak was detected in the unspiked waste water (Figure 10A). Unfortunately, the minimal detected amount of hydroxysoman (around 1 ng) is quite high in comparison with the smaller acids, which could be measured at a level of 100 pg. Therefore, in order to detect hydroxysoman in an aqueous solution below a concentration level of 50 ng/ml, preconcentration will be necessary.

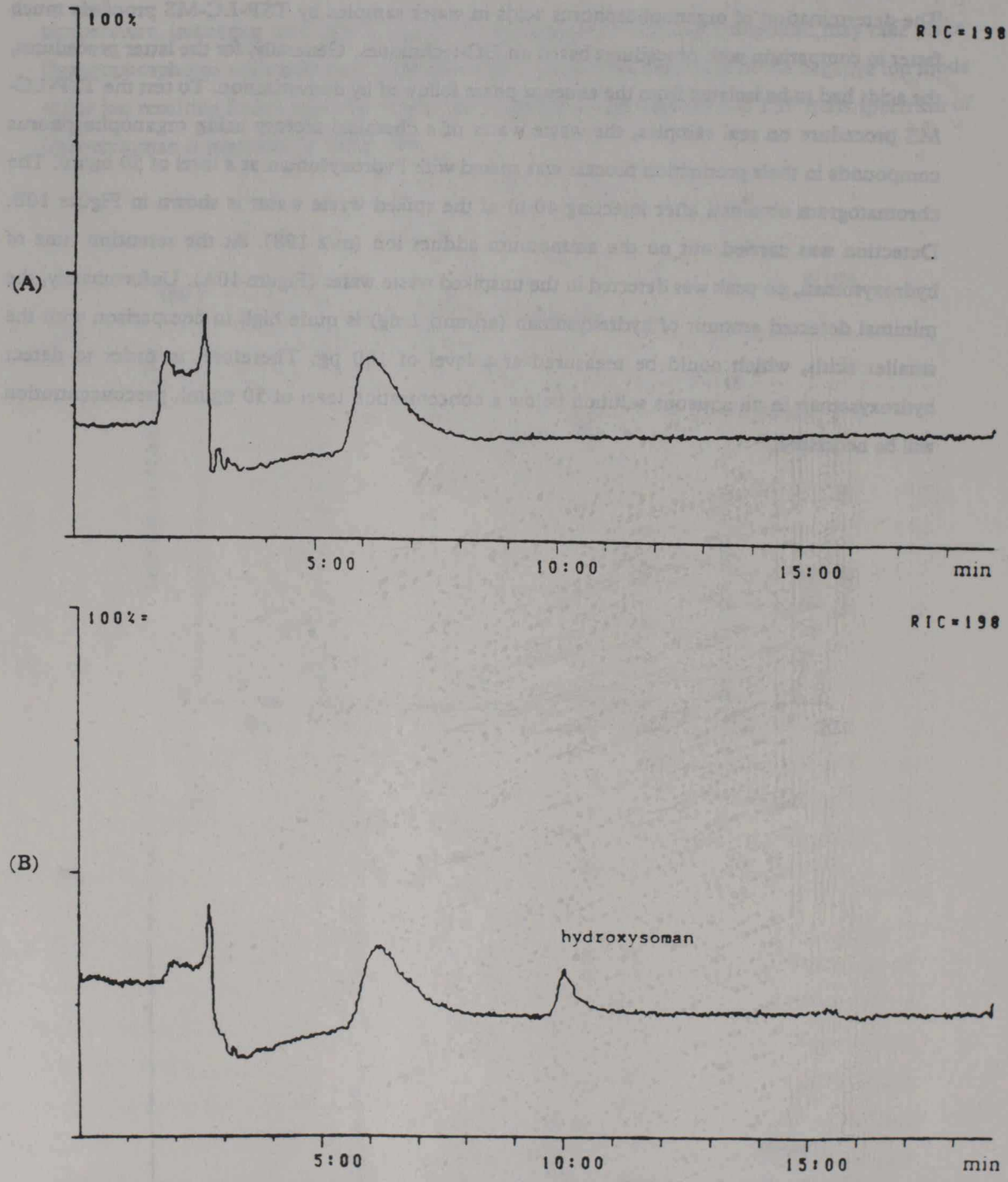


Figure 10 Determination of hydroxysoman in a waste water sample of a chemical factory.
A : unspiked water
B : water spiked with 50 ng/ml of hydroxysoman
Eluent : 0.1 M ammonium acetate - methanol (60 : 40)

5 ANALYSIS OF VESICANTS AND RELATED COMPOUNDS

In contrast to nerve agents, the vesicant mustard hydrolyses relatively fast in water. This fact hampers its determination by TSP-LC-MS on a reversed phase column using an aqueous eluent. However, thiodiglycol, the hydrolysis product of mustard as well as its main precursor, may well be determined by TSP-LC-MS. The result of a TSP-LC-MS analysis of a crude commercial thiodiglycol product is shown in Figure 11. In addition to the compound itself, several impurities were found which, based on their measured molecular weights, were tentatively identified as thiodiglycol related compounds, including T-diglycol, the hydrolysis product of the vesicant di-mustard ether (T).

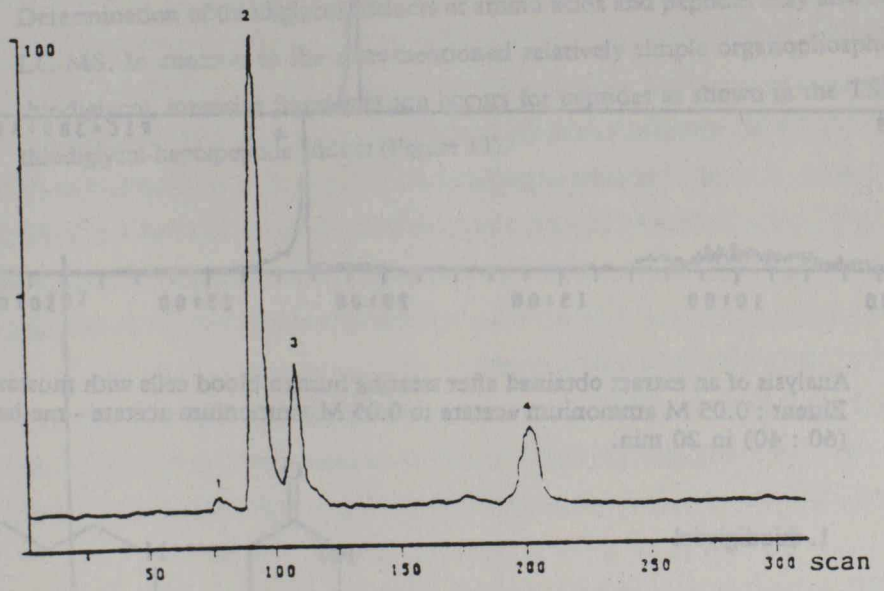
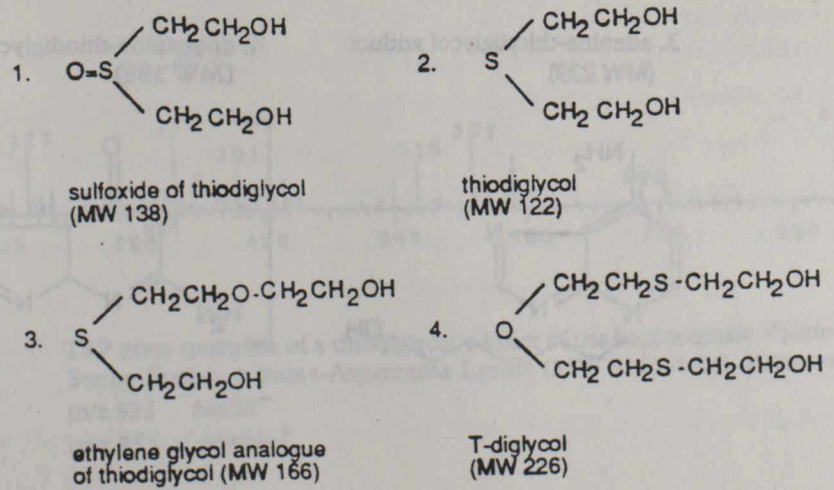


Figure 11 Analysis of a crude commercial thiodiglycol product.
Eluent : 0.1 M ammonium acetate - methanol (65 : 35)



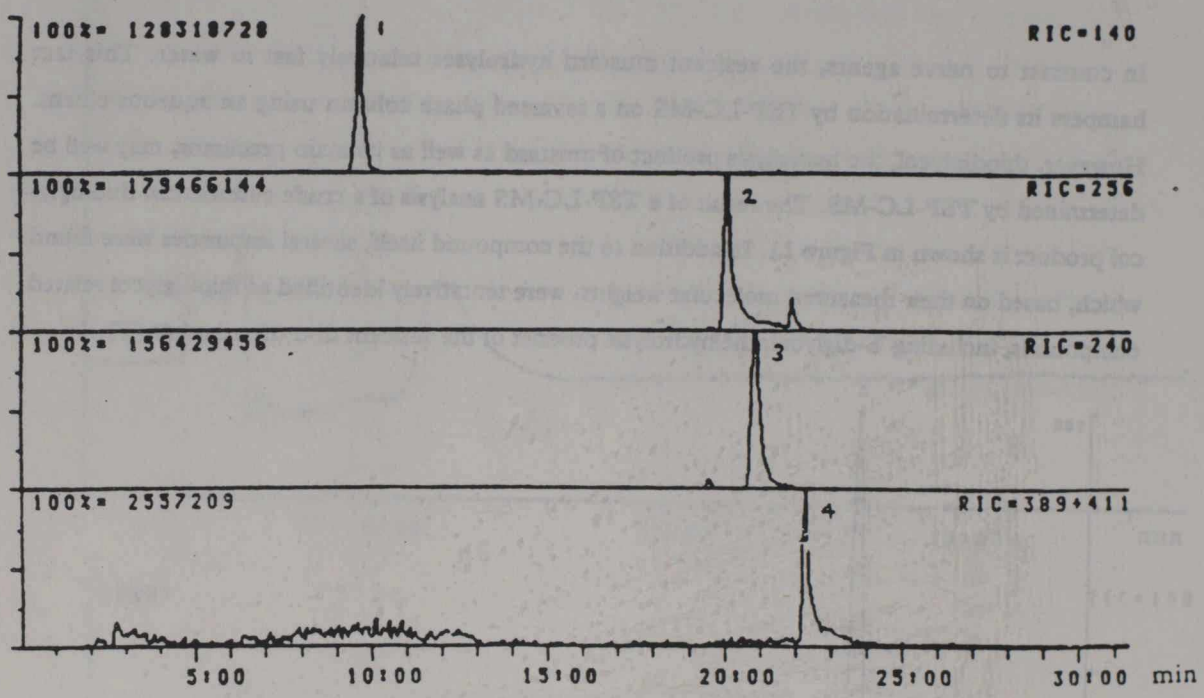
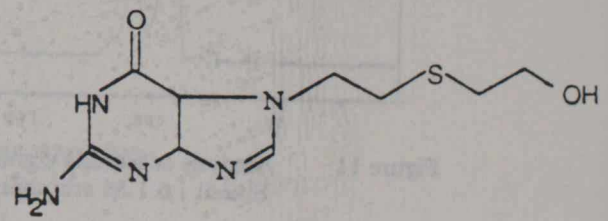


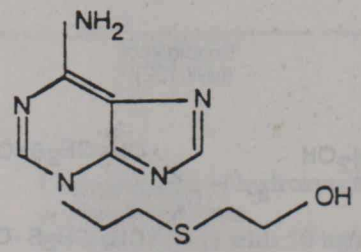
Figure 12 Analysis of an extract obtained after treating human blood cells with mustard. Eluent : 0.05 M ammonium acetate to 0.05 M ammonium acetate - methanol (60 : 40) in 20 min.

1. thiodiglycol

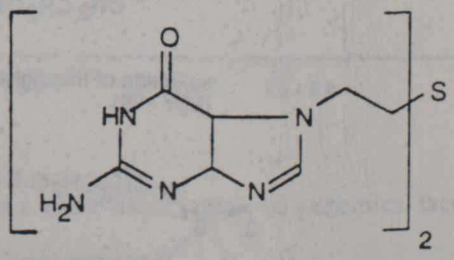


2. guanine-thiodiglycol adduct (MW 255)

3. adenine-thiodiglycol adduct (MW 239)



4. di-guanine-thiodiglycol adduct (MW 388)



Mustard is an alkylating agent which forms adducts with purine bases (DNA) and amino acids (proteins) after exposure. TSP-LC-MS offers the possibility to detect these adducts in biological samples. The result of the TSP-LC-MS analysis of a biological sample is presented in Figure 12, in which a number of chromatograms are depicted corresponding with the thiodiglycol adducts of guanine and adenine. The sample was obtained after treating human blood cells with 1 mM mustard for 30 min. at 37 °C, followed by a work-up procedure consisting of enzymatic degradation of the reacted DNA to nucleosides and subsequent heating to release the alkylated purine bases [13]. The thiodiglycol adducts of the purine bases were chromatographed on a C18 column using linear gradient elution ranging from 0.05 M ammonium acetate to a 0.05 M ammonium acetate/methanol mixture (60 : 40) in 20 min..

Determination of thiodiglycol adducts of amino acids and peptides may also be carried out by TSP-LC-MS. In contrast to the aforementioned relatively simple organophosphorus compounds and thiodiglycol, intensive fragmentation occurs for peptides as shown in the TSP mass spectrum of a thiodiglycol-heptapeptide adduct (Figure 13).

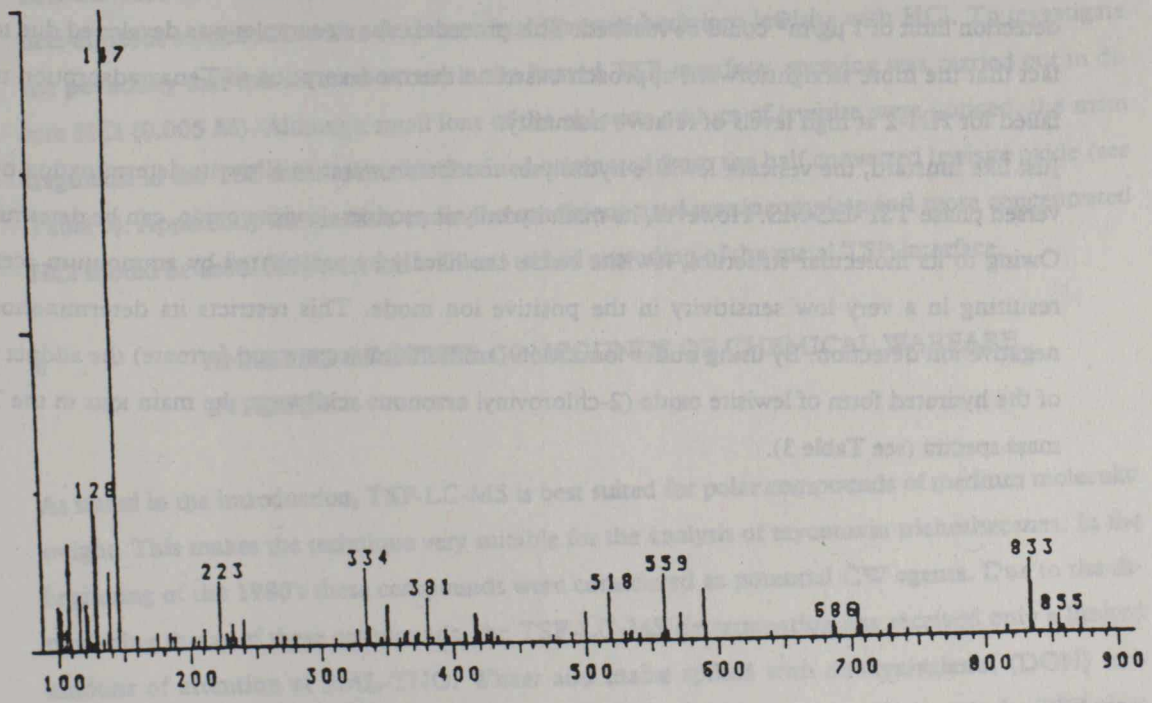


Figure 13 TSP mass spectrum of a thiodiglycol adduct of the heptapeptide Valine-Leucine-Serine-Proline-Alanine-Asparagine-Lysine (MW 832) in 0.1 M ammonium acetate

m/z 833 $M+H^+$

m/z 855 $M+Na^+$

The heptapeptide (Valine-Leucine-Serine-Proline-Alanine-Asparagine-Lysine) corresponds with the N-terminal part of the α-chain of human haemoglobin. The relative intensities of the ions in the TSP mass spectrum recorded strongly depended on the experimental conditions, so the spectrum presented has to be regarded as an example. Although these results look promising, the application of TSP-LC-MS in the field of biological molecules is limited to rather small biopolymers. Other LC-MS combinations based on continuous flow FAB and ion/electrospray offer better possibilities.

The hydrolysis rates of the nitrogen mustards are lower in comparison with mustard. This fact allows the determination of these compounds by reversed-phase LC. Using a mixture of acetonitrile and 0.1 M ammonium acetate (60 : 40), methyl nitrogen mustard (HN-2) could be chromatographed. However, the compound was not measured as such, but quantitatively converted into an acetoxy derivative in the heated TSP-interface due to the reaction of the chlorine atoms with ammonium acetate. This reaction was also observed for the related nitrogen mustard cytostatic drugs phenylalanine mustard and cyclophosphamide [14]. Based on the TSP-LC-MS analysis, a procedure was developed for the determination of HN-2 not only in water but also in the atmosphere. The procedure consisted of the adsorption of the compound on a silica gel tube, followed by extraction with a 0.05 M HCl/acetonitrile (50 : 50) mixture. With an air sample volume of 10 l, a detection limit of 1 $\mu g/m^3$ could be reached. This procedure for air samples was developed due to the fact that the more straightforward approach based on thermodesorption of Tenax adsorption tubes failed for HN-2 at high levels of relative humidity.

Just like mustard, the vesicant lewisite hydrolyses too fast in water to allow its determination by reversed phase TSP-LC-MS. However, its main hydrolysis product, lewisite oxide, can be determined. Owing to its molecular structure, lewisite oxide can hardly be protonated by ammonium acetate, resulting in a very low sensitivity in the positive ion mode. This restricts its determination to negative ion detection. By using buffer ionization (ammonium acetate and formate) the adduct ions of the hydrated form of lewisite oxide (2-chlorovinyl arsonous acid) were the main ions in the TSP mass spectra (see Table 3).

Table 3 Main ions in the TSP mass spectra of lewisite oxide

Solvent	discharge	main ions	probable structure
NH_4-Acetate	off	229/231•	$(HO)_2As\text{-}C{=}C\text{-}Cl \mid OOCCH_3^-$
NH_4-Formate	off	215/217	$(HO)_2As\text{-}C{=}C\text{-}Cl \mid OOCH^-$
Methanol	on	152/154	$O{=}As\text{-}C{=}C\text{-}Cl^-$
HCl	off	223/225/227	$(HO)(Cl)As\text{-}C{=}C\text{-}Cl \mid Cl^-$

Discharge ionization led to the appearance of even mass fragments. However, ions originating from the hydrated form of lewisite oxide (m/z 170/172) could not be detected during spraying in an aqueous medium. The ions from lewisite oxide (m/z 152/154) itself only became predominant in non-aqueous solution. Lewisite oxide can be converted back into lewisite with HCl. To investigate the possibility that this reaction occurs in the heated TSP-interface, spraying was carried out in dilute HCl (0.005 M). Although small ions of the chlorine adduct of lewisite were noticed, the main fragments in the TSP mass spectrum obtained originated from the half converted lewisite oxide (see Table 3). Apparently the reaction under the conditions used was incomplete and more concentrated HCl should be used. However, this will lead to bad corroding of the metal TSP-interface.

6 ANALYSIS OF OTHER COMPOUNDS OF CHEMICAL WARFARE INTEREST

As stated in the introduction, TSP-LC-MS is best suited for polar compounds of medium molecular weight. This makes the technique very suitable for the analysis of mycotoxin trichothecenes. In the beginning of the 1980's these compounds were considered as potential CW agents. Due to the diminishing threat of these compounds, the TSP-LC-MS determination has received only a limited amount of attention at PML-TNO. Wheat and maize spiked with deoxynivalenol (DON) at a 0.5-1 mg/kg (ppm) level were analysed. A characteristic chromatogram together with the TSP mass spectrum of DON is presented in Figure 14. The selectivity of the determination by TSP-LC-MS was quite good. No peak of any significance could be detected in the blank maize and wheat extracts with the used eluent composition (water : methanol = 70 : 30 with post-column addition of 0.2 M

ammonium acetate). The extraction and clean-up procedures used were carried out according to Tanaka et al. [15] and existed of an extraction with methanol/water, defatting with hexane and further purification using a Florisil column. More extensive work in this field has been carried out by Krishnamurthy et al. who recently reported several applications of the determination of trichothecenes by TSP-LC-MS [16, 17].

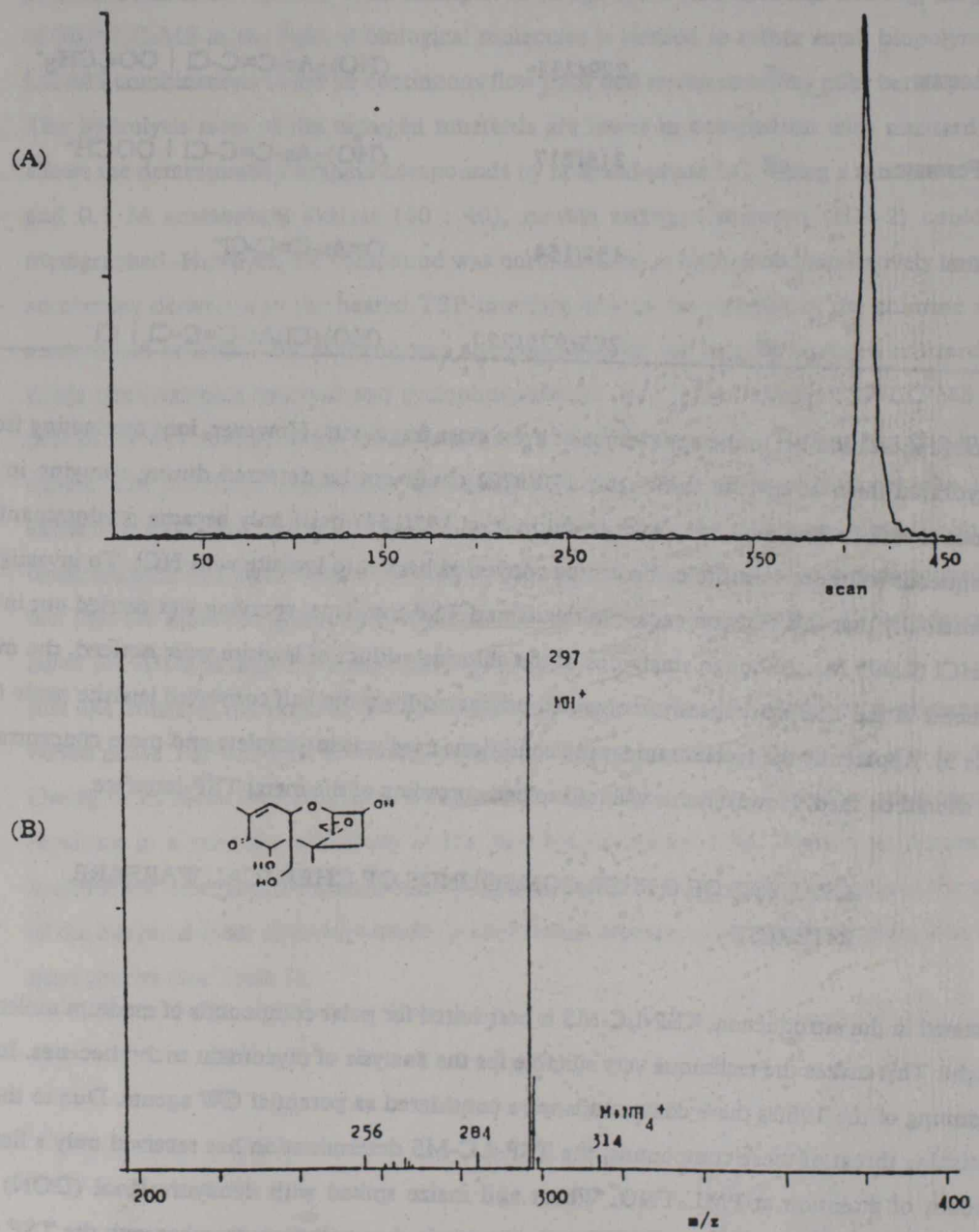


Figure 14 Analysis of a maize extract (after clean-up) spiked with deoxynivalenol at a level of 0.5 - 1 mg/kg.
Eluent : water - methanol (70 : 30)
A : Obtained chromatogram at m/z 297. B : TSP mass spectrum of DON

Psychochemically active glycolates such as BZ are compounds with molecular weights over 300 amu and polar moieties. Although BZ can be analysed by GC, its determination is somewhat at the edge of the possibilities of this technique. Determination by TSP-LC-MS is quite straightforward. Possessing a basic quinuclidinyl ring the TSP mass spectrum of BZ consisted, as in the case of VX, almost exclusively of the protonated molecular ion at m/z 338 when ionization by the ammonium acetate buffer is applied.

7 CONCLUSIONS

TSP-LC-MS offers great potential for the verification of CW agents and their degradation products in aqueous samples. Analyses of environmental or industrial water samples need hardly any work-up and can therefore be carried out relatively quickly. Combined with preconcentration, reasonable low detection levels (below ng/ml) may sometimes be achieved. Work-up procedures will be required for the analysis of biological samples (urine, blood etc.). The technique may be used for the analysis of other matrices (soil, natural products and even air) as well.

Generally, the information content of TSP mass spectra is small. In most cases the spectra provide only molecular weight information. This might be sufficient if TSP is used only as a selective universal LC detector in order to screen samples for the possible presence of scheduled compounds (target compound analysis). For an unequivocal identification, other ionization modes (filament-on, discharge) and collision-induced dissociation will be necessary.

Unfortunately, TSP mass spectra are dependent on the eluent composition as well as on instrumental conditions and are therefore less suitable for retrieval purposes.

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CONFERENCE ON DISARMAMENT

CD/CW/WP.356
6 August 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

UNITED STATES OF AMERICA

Challenge Inspection Procedures for Declared Facilities

A requested perimeter for a challenge inspection of declared facilities may capture two types of areas:

(1) areas within declared facilities which are subject to recurring routine inspections (Articles IV, V and VI), and

(2) areas within declared facilities which are not subject to routine inspection (Article III, paragraph 1(C)).

This paper proposes procedures for these two situations.

PERIMETER DETERMINATION

For all declared facilities (Articles III, IV, V, and VI), the following procedures would apply:

-- If the requested perimeter is contained within or conforms with the declared perimeter, the declared perimeter shall be considered the final perimeter, with one exception: if agreed by the challenged State Party, the final perimeter may be made smaller to conform with that requested by the challenging State Party.

-- The challenged State Party shall transport the inspection team to the final perimeter as soon as practicable, but in any case shall ensure their arrival at the perimeter no later than 12 hours after specification of the site by the challenging State Party.

-- Inspectors shall have access to the final perimeter and a 50 meter band for the purpose of conducting monitoring and sampling activities as specified for the final perimeter in CD/CW/WP.352, Part III.II.F. At the discretion of the challenged State Party, the band could run inside, outside, or on both sides of the declared perimeter.

ACCESS WITHIN FINAL PERIMETER

A. For facilities declared under Articles IV, V and VI, the following procedures are proposed:

-- Upon arrival at the final perimeter, access shall be granted following the pre-inspection briefing and discussion of the inspection plan which shall be limited to the time frame specified in Part I.V.C. of the Protocol on Inspection Procedures.

-- For facilities with facility agreements, access and activities within the final perimeter shall be unimpeded within the boundaries established by the agreements.

-- For facilities without facility agreements, negotiation of access and activities shall be governed by the applicable general inspection guidelines or model agrements established under the Convention.

-- Access greater than that granted for inspections under Articles IV, V and VI shall be managed in accordance with procedures in CD/352, Part III, paragraphs III.B. 3 and 4.

B. For CW development facilities declared under Article III, paragraph 1(C):

-- At the perimeter, negotiations will be conducted and managed access commenced within (...) hours of arrival at the final perimeter. If access is restricted or denied to areas or structures not related to chemical weapons, the challenged State Party shall make every reasonable effort to satisfy the compliance concern.

CONFERENCE ON DISARMAMENT

CD/CW/WP.357
8 August 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

UNITED STATES OF AMERICA

Measures to Ensure Universality

Introduction

The United States places the utmost importance in achieving universal adherence to the Chemical Weapons Convention (CWC). As promised by President Bush in his May 13 initiative, we are proposing provisions which provide incentives for states to join the Convention.

The following provisions encourage universal adherence by limiting trade in scheduled chemicals and materials to States Parties only. We are also proposing that States Parties establish export/import monitoring regimes as suggested in the Austrian paper (CD/1062) based on the precedent set by the 1988 Narcotics Convention.

Add to Article VI - Activities Not Prohibited Under the Convention

"Chemicals listed in schedules 1, 2A, 2B, or 3, and equipment and technology used to produce them listed in (...) shall only be exported to, or imported from, other States Parties. These obligations shall take effect not later than the deadlines listed in this paragraph, and shall continue indefinitely thereafter:

"(a) For chemicals listed in schedule 1, upon entry into force of this Convention;

"(b) For chemicals listed in schedule 2, three years after entry into force of this Convention;

"(c) For chemicals listed in schedule 3, five years after entry into force of this Convention;

"(d) For equipment and technology used to produce such chemicals listed in (...), five years after entry into force of this Convention."

"States Parties shall make arrangements with non-States Parties for international inspections equivalent to those applicable to States Parties during the time periods before these restrictions take effect."

Add to Article VII - National Implementation Measures

"Each State Party shall establish and maintain a system to monitor exports and imports of chemicals listed in schedules 1, 2A, 2B or 3 and equipment and technology used to produce such chemicals listed in (...)."

Add to Section on Preparatory Commission, para 6 (d)

"6. The Commission shall:

(d) undertake, inter alia, the following tasks on subjects requiring immediate attention after the entry into force of the Convention:

> "...preparation of a list of equipment and technology used to produce chemicals listed in schedules 1, 2A, 2B or 3, as set forth in Article VI."

CONFERENCE ON DISARMAMENT

CD/CW/WP.358 */
13 August 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

UNITED KINGDOM OF GREAT BRITAIN AND NORTHERN IRELAND

PROPOSALS FOR ESTABLISHING THRESHOLDS IN THE CHEMICAL WEAPONS CONVENTION: SCHEDULE 2B

1. Introduction

1.1 The Chemicals listed in the Schedules in the Chemical Annex of the current rolling text pose, to varying degrees, risks to the CW Convention. Facilities handling such chemicals will thus be subject to the routine monitoring provisions of Article VI.

1.2 All production of Schedule 1 chemicals above 100g must be declared both nationally and for each facility. Appropriate limits for different types of facility are set (in Annex 1 to Article VI) to the quantities of these compounds which can be held or produced. In contrast production of Schedule 2 and 3 chemicals need only be declared once a particular threshold has been attained.

1.3 As there are marked differences between the potencies of different chemicals, and consequently the weights which would constitute militarily significant capabilities, there is a case for having declaration thresholds reflect these differences. Lower thresholds would apply to the most toxic compounds and higher ones for those less potent. Schedule 2B accommodates chemicals of risk to the Convention with a wide range of effectiveness and such an approach has been suggested several times during the negotiations.

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*/ Re-issued for technical reasons.

2. Aim

2.1 The paper carries this suggestion forward and describes a procedure whereby different thresholds for declaration of existing and potential Schedule 2B compounds could be set. If thought necessary the same procedure could be applied to set declaration thresholds for the toxic (non-key precursor) chemicals of Schedule 3 and for precursor chemicals on Schedules 2A and 3 by using the effective dose of the Schedule 1 (or 2B) chemical to which the precursor related. It could also be applied, if thought useful, to determine maximum permitted holdings (within the 1 tonne aggregate) of Schedule 1 chemicals at a single small scale facility.

3. Militarily Significant Quantities

3.1 The risk a particular compound poses to the convention depends upon a number of criteria. However the threshold for the weight of a substance which should be declared and subject to the Convention's provisions should be related to the minimum quantity which is likely to be militarily significant.

3.2 Calculations based on simulated battlefield models (Annex A) indicate that regardless of the delivery system (and the potency of the agent) a quantity of about a billion (10^9) times the effective dose of a substance is required for one attack under average meteorological conditions to cover an area within which a typical military unit might operate (0.5 to 2.0 km^2).

3.3 In the case of the nerve agents a billion times the lethal dose (about 1mg per man) affords a figure of 1 tonne. By contrast for botulinum toxin which has recognised potential as a chemical weapon (but not yet included in the Schedules) the lethal dose is probably about 5µg per man. Multiplying this by a billion (10^9) affords 5 kg. (Even a million million (10^{12})

doses, sufficient for 1,000 attacks, is equivalent to only 5 tonnes.) These widely disparate figures of 1 tonne and 5 kg reinforce the suggestion for setting declaration thresholds in a way which takes account of the potency of the agent.

4. Determination of Effective Dose

4.1 To implement such an approach, one needs a way to assess the effective dose of candidate substances for the Schedule. The standard LD_{50} toxicity test (with all its limitations) is not necessary for this purpose and is being increasingly replaced with a fixed dose procedure requiring far fewer animals. Each chemical is assigned to one of several agreed toxicity categories; it is not necessary to determine the exact toxicity or effectiveness of each substance. Declaration thresholds would thus be related to categories and not to the precise toxicity of individual chemicals.

4.2 Although the fixed dose procedure is recommended for assessing the effective dose of a chemical, details of species and routes of administration should be left to the judgement of those presenting the data. The upper and lower limits of category 1 would ensure that any recognised, relevant source of data for, say, botulinum and similarly potent toxins would fall within it. In fact data provided whereby they did not would itself arouse suspicion.

4.3 The Table shows five possible categories along with the associated declaration thresholds for actual or putative Schedule 2B chemicals. (The toxic, dual purpose chemicals of Schedule 3 are shown for comparison.) The suggested lower limits for each toxicity category run from zero (in theory, 1µg per man in practice) to 500mg per man. These afford declaration thresholds (based upon the factor of a billion) ranging from 1kg to 100 tonnes. A figure of 500mg/man (equivalent to a declaration threshold of 500 tonnes) is indicated for the cut-off at the lower limit as chemicals less toxic than this are unlikely to pose a risk as chemical weapons.

TABLE

POSSIBLE CATEGORIES FOR DECLARATION OF SCHEDULE 2B CHEMICALS

CATEGORY	LOWER LIMIT OF TOXICITY CATEGORY	THRESHOLD BASED ON THE BILLION DOSE CRITERION (see text)	EXAMPLE CHEMICALS
1	Zero in theory, 1µg/man in practice	1 kg	Botulinum toxin* Substance P*
2	0.5 mg/man	500 kg	BZ* Chloropicrin^^
3	10 mg/man	10 tonnes	Phosgene^^ PFIB^ Amiton^ Hydrogen cyanide^^
4	100 mg/man	100 tonnes	Apomorphine* Cyanogen chloride^^
5	500 mg/man	no need for declaration	

^ Schedule 2B compounds. ^^ Schedule 3 compounds.

* Potential Schedule 2B compounds.

4.4 Using this approach Schedule 2B chemicals would be declared when the weight produced attains one billion times the lower limit for the respective toxicity category. The effective doses for Amiton and PFIB (chemicals in the Schedule at present) fall between 10 and 100 mg/man. This would place them in Category 3 and they would thus be declared when their production exceeded ten tonnes; ie 10^9 (1 billion) x 10 mg = 10,000 kg = 10 tonnes.

5. Summary and Conclusions

5.1 A procedure is proposed for determining thresholds for declaration for Schedule 2B chemicals. Such thresholds will vary from chemical to chemical on the basis of their effectiveness, and hence the risk to the convention. It is recommended that the threshold determinations be based on 10^9 times the effective dose and that a chemical be placed in a toxicity category using a fixed dose procedure.

ANNEX A

CALCULATION OF A MILITARILY SIGNIFICANT QUANTITY

A1. A crucial factor in selecting any threshold is to determine the militarily significant quantity of an agent. Different chemical weapons may be delivered by different systems (eg artillery shell or rockets, aircraft bombs or as sprays). Good models exist for predicting the average area which will be covered to a given level in a single attack with each of these systems.

A2. The results of the simulations carried using a range of scenarios indicate that under "average" meteorological conditions (4 ms^{-1} wind speed and neutral stability) and regardless of the delivery system, a quantity of the order of one billion (10^9) times the effective dose is required to cover an area of between 0.5-2Km^2. This is an area within which a typical military unit would operate. Calculations have also been carried out using 10^8 effective doses of agent (for example by reducing the numbers of munitions by a factor of 10) and show that 10^8 effective doses would only cover about 0.1 km^2 under the same conditions.

A3. 10^9 doses therefore represents the approximate quantity of agent required for a single attack under most conditions. However under conditions which are favourable to the attacker, a significant effect may be caused by as little as 10^8 doses.

A4. Adopting this billionfold criterion for setting thresholds therefore only permits a potential aggressor to hold or carry out undeclared production of sufficient material for less than about 10 attacks even under the most favourable conditions and in most cases for no more than a single effective attack.

ANNEX B

POSSIBLE EXTENSION TO PRECURSORS

B1. The use of the toxicity of a precursor chemical to determine its declaration threshold is not appropriate and a modification to the procedure is required for those on Schedule 3, Schedule 2A and for DF and QL on Schedule 1.

B2. The simplest way to deal with this problem is to relate the values of the thresholds to the effectiveness of the agent(s) to which the chemical of concern is a precursor. (A similar approach is taken in declaring binary weapons for destruction; Annex to Article IV. Sector III. Binary Weapons. Paragraph 1). The quantity of precursor required to make 10^9 effective doses of agent would thus be used to assign the precursor to one of the categories in the Table for declaration purposes even though it is based upon a chemical conversion ratio rather than toxicity directly. If each chemical on Schedule 2A, the precursor members of Schedule 3, DF and QL are treated in this light all except a few fall into Category 2 (see table), which covers chemicals for which 10^9 effective doses range from 500kg to 10,000kg. (The exceptions are some VX precursors.) As all other precursors would fall into Category 2 the less stringent threshold would be most appropriate. All precursors would therefore have the same declaration threshold of 500kg, (or holding limit, if considered desirable, in the case of DF and QL).

B3. In calculating the weight of agent produced from a given weight of precursor an 80% theoretical yield is appropriate for each reaction step. This is a typical figure and actual yields range from 70 to 90+%.

USA	CD/CW/WP.359	Report of the third United States trial inspection exercize NOT REPRODUCED (see WP volume)	Also issued as CD/1100 14.8.91

* * *

Germany	CD/CW/WP.360	Report on a trial challenge inspection at a large chemical plant site NOT REPRODUCED (see WP volume)	Also issued as CD/1101 15.8.91

* * *

Germany	CD/CW/WP.361	Report on an international trial challenge inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1102 15.8.91

* * *

CONFERENCE ON DISARMAMENT

CD/CW/WP.362
19 August 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

DISCUSSION PAPER ON

SCHEDULES AND GUIDELINES

presented by the Friend of the Chair on Technical Matters (A.J. Meerburg)

During the 1991 Session of the Ad-Hoc Committee, consultations were held on parts II and III of the Annex on Chemicals, i.e. the Schedules and the Guidelines for the Schedules (pages 57 to 64 of doc. CD/1046). No conclusions were reached as yet. For further consideration the following Discussion Paper is presented. Changes with respect to doc. CD/1046 are indicated by *italics*. deletions by .... .

GE.91-62315

II. GUIDELINES FOR SCHEDULES OF CHEMICALS

A. Guidelines for Schedule 1

The following criteria .... shall be taken into account *[singly or in combination]* in considering whether a chemical should be included in Schedule 1:

1. (a) it has been developed, produced, stockpiled or used as a chemical weapon as defined in Article II;

[or]

(b) it poses otherwise a high risk to the objectives of the Convention by virtue of its high potential for use for activities prohibited *under* the Convention because one or more of the following conditions is met:

- it possesses a chemical structure closely related to that of other toxic chemicals listed in Schedule 1 and has, or can be expected to have, comparable properties;
- it possesses such lethal or incapacitating toxicity as well as other properties that might enable it to be weaponized and used as a chemical weapon;
- ..it may be used as a precursor in the final technological stage of production *[in a simple chemical conversion process]* of a toxic chemical listed in Schedule 1, regardless of whether this stage takes place in facilities, in munitions or elsewhere;..

[and]

2. it has little or no use for purposes not prohibited under the Convention.

B. Guidelines for Schedule 2 part A ..

The following criteria shall be taken into account in considering whether a precursor to a Schedule 1 chemical should be included in Schedule 2 part A:

1. it may be used in one of the chemical reactions at the final stage of formation of a chemical listed in Schedule 1 *or Schedule 2 part B:*

1/ ....

2. *it* .... poses a significant risk 1/ to the objectives of the Convention by virtue of its importance in the production of a chemical listed in Schedule 1 *or Schedule 2 part B;*

..3. *it* is not produced in large commercial quantities for purposes not prohibited *under* the Convention. 2/..

C. Guidelines for Schedule 2 part B ..

The following criterion shall be taken into account in considering whether a toxic chemical which is not included in Schedule 1 should be included in Schedule 2 part B:

it poses .... a significant risk to the objectives of the Convention *because it possesses such lethal or incapacitating toxicity as well as other properties that might enable it to be weaponized and used as a chemical weapon.* 3/ ..

D. Guidelines for Schedule 3 ..

The following criteria shall be taken into account when considering whether a *toxic* chemical or a precursor ...., not listed in other Schedules, *s*hould be included in Schedule 3:

1/ The view was expressed that the degree of the risk of a chemical is determined on the basis of the contribution made by a precursor to the formation of the structure, or on the basis of the role it plays in determining the toxic properties of a Schedule 1 chemical.

2/ The question of the applicability of a quantitative criterion requires further discussion, taking into account, inter alia, the aims of the *verification* measures stipulated in Article VI *and Annex 2 to Article VI, and the likelihood of meeting these aims through the verification regime for Schedule 2 chemicals.* ...

3/ ....

3/ A view was expressed that the consideration of this guideline should be continued taking into account that the present formulation is questionable as far as it reproduces a criterion for Schedule 1.

4/ ....

5/ ....

CD/1046, page 63, Appendix I

Toxic chemical:

1. *(a)* it has been stockpiled as a chemical weapon;

   [or]

   (b) it .... poses *otherwise* a risk to the objectives of the Convention *because it possesses such lethal or incapacitating toxicity as well as other properties that might enable it to be weaponized and used as a chemical weapon* 1/;

[and]

2. *it* is produced in large commercial quantities 2/ for purposes not prohibited *under* the Convention .... .

Precursor .... :

1. it .... poses a risk to the objectives of the Convention by virtue of its importance in the production of one or more chemicals listed in Schedule 1 *or Schedule 2 [B]* .... ; ..

[and]

2. *it* is produced in large commercial quantities 2/ for purposes not prohibited *under* the Convention .... .

....

1/ *A view was expressed that the consideration of this guideline should be continued taking into account that the present formulation is questionable as far as it reproduces a criterion for Schedule 1.*

2/ The question of a quantitative criterion, possibly including a numerical threshold, requires further discussion.

2/ ....

3/ ....

CD/1046, page 64, Appendix I

III. SCHEDULES OF CHEMICALS 1/

A. Schedule 1

1. O-Alkyl (≤C10, incl. cycloalkyl) alkyl (Me, Et, n-Pr or i-Pr)-phosphonofluoridates ...

   e.g. Sarin: O-isopropyl methylphosphonofluoridate (107-44-8)
   Soman: O-pinacolyl methylphosphonofluoridate (96-64-0)

2. O-Alkyl (≤C_{10}, incl. cycloalkyl) N,N-dialkyl (Me, Et, n-Pr or i-Pr) phosphoramidocyanidates ...

   e.g. Tabun: O-ethyl N,N-dimethylphosphoramidocyanidate (77-81-6)

3. O-Alkyl (H or ≤C_{10}, incl. cycloalkyl) S-2-dialkyl (Me, Et, n-Pr or i-Pr)-aminoethyl alkyl (Me, Et, n-Pr or i-Pr) phosphonothiolates and corresponding alkylated and protonated salts...

   e.g. VX : O-ethyl S-2-diisopropylaminoethyl methyl phosphonothiolate (50782-69-9)

4. Sulphur mustards:

   2-Chloroethylchloromethylsulphide (2625-76-5)
   bis(2-chloroethyl)sulphide: Mustard Gas (H) (505-60-2)
   bis(2-chloroethylthio)methane (63869-13-6)
   1,2-bis(2-chloroethylthio)ethane: Sesquimustard (Q) (3563-36-8)
   1,3-bis(2-chloroethylthio)-n-propane (63905-10-2)
   1,4-bis(2-chloroethylthio)-n-butane
   1,5-bis(2-chloroethylthio)-n-pentane
   bis(2-chloroethylthiomethyl)ether
   bis(2-chloroethylthioethyl)ether: O-Mustard (T) (63918-89-8)

1/ The ultimate composition of these schedules depends, inter alia, on the final guidelines for the Schedules, on the to be agreed verification regime with respect to the chemical industry, on actual production levels of certain chemicals and on the thresholds for declaration and verification to be agreed for schedule 2 B. This means that chemicals may at a later stage in the negotiations be added to, transferred between or removed from the Schedules. Further consideration also needs to be given to the specific verification requirements with respect to toxins.

A view was expressed that the composition of the Schedules should be based solely on the criteria contained in de guidelines for the Schedules.

....

CD/1046, page 57, Appendix I

5. Lewisites:

Lewisite 1: 2-chlorovinyldichloroarsine	(541-25-3)
Lewisite 2: bis(2-chlorovinyl)chloroarsine	(40334-69-8)
Lewisite 3: tris (2-chlorovinyl)arsine	(40334-70-1)

6. Nitrogen mustards:

HN1: bis(2-chloroethyl)ethylamine	(538-07-8)
HN2: bis(2-chloroethyl)methylamine	(51-75-2)
HN3: tris(2-chloroethyl)amine	(555-77-1)

7. 3-Quinuclidinyl benzilate (BZ) 1/ 2/ (6581-06-2)

8. Saxitoxin 3/ (35523-89-8)

9. Ricin 3/

10. Alkyl (Me, Et, n-Pr or i-Pr) Phosphonyldifluorides ..

e.g. DF: methylphosphonyldifluoride (676-99-3)

11. O-Alkyl (H or $\leq C_{10}$, incl. cycloalkyl) O-2-dialkyl (Me, Et, n-Pr or i-Pr)-aminoethyl alkyl (Me, Et, N-Pr or i-Pr) phosphonites and corresponding alkylated and protonated salts ..

e.g. QL: O-ethyl O-2-diisopropylaminoethyl methylphosphonite (57856-11-8)

.....

1/ *The view was expressed that this chemical should be included in Schedule 2 part B because of its production (as an intermediate in captive use) for purposes not prohibited under the Convention.*

2/ *A view was expressed that this item should be replaced by:*

3 Quinuclidinyl esters of (2-phenyl-2-(phenyl, cyclohexyl, cyclopentyl or cyclobutyl)-2-hydroxyacetic acids and their methyl, ethyl, n-propyl and iso-propyl esters

e.g. 3-Quinuclidinyl benzilate (BZ)

3/ The placement of toxines on the Schedules requires further consideration. A view was expressed that relevant toxins should be considered for inclusion in Schedule 2 part B, for example, in a separate section with lower thresholds for declaration and verification compared with other chemicals on that Schedule. Another view was expressed that different toxins could be included in different Schedules in accordance with the guidelines for those Schedules.

....

12. .. Chloro Sarin: O-isopropyl methylphosphonochloridate 1/ (1445-76-7)

13. Chloro Soman: O-pinacolyl methylphosphonochloridate 1/ (7040-57-5).

14. 3,3-Dimethylbutan-2-ol (pinacolyl alcohol) 1/ (464-07-3).

B. Schedule 2 part A

1. Chemicals, *except for those chemicals listed under Schedule 1*, containing a phosphorus atom to which is bonded one methyl, ethyl or propyl (normal or iso) group but not further carbon atoms ....

e.g. Methylphosphonyl dichloride
Dimethyl methylphosphonate

Exemption:
Fonofos : O-ethyl S-phenyl ethylphosphonodithioate 1/2/ *(944-22-9)*

2. N,N-Dialkyl (Me, Et, n-Pr or i-Pr) phosphoramidic dihalides

3. Dialkyl (Me, Et, n-Pr or i-Pr) N,N-dialkyl (Me, Et, n-Pr or i-Pr)-phosphoramidates

4. Arsenic trichloride 3/ (7784-34-1)

5. 2,2-Diphenyl-2-hydroxyacetic acid 4/ (76-93-7)

6. Quinuclidin-3-ol .. (1619-34-7)

....

....

1/ A view was expressed that this chemical should be included in Schedule 2 part A.

2/ *A view was expressed that the question of exemptions needs further consideration.*

3/ *A view was expressed that this chemical should be included in Schedule 3.*

4/ *A view was expressed that* if item 7 on Schedule 1 is expanded into a group, a corresponding expansion should be considered for items 5 and 6 on Schedule 2 part A. Item 5 could, e.g., then include:

2-phenyl-2-(phenyl, cyclohexyl, cyclopentyl or cyclobutyl)-2-hydroxyacetic acids and their methyl, ethyl, n-propyl and iso-propyl esters,

and item 6 could, e.g., include:

3- or 4-hydroxypiperidine .... .

7. N,N-Dialkyl (Me, Et, n-Pr or i-Pr) aminoethyl-2-chlorides and corresponding *protonated salts* .. ..

8. N,N-Dialkyl (Me, Et, n-Pr or i-Pr) aminoethane-2-ols and corresponding *protonated salts* .. ..

Exemptions:
N,N-dimethylamino ethanol and corresponding protonated salts 1/ *(108-01-0)*

N,N-diethylamino ethanol and corresponding protonated salts 1/ *(100-37-8)*

9. N,N-Dialkyl (Me, Et, n-Pr or i-Pr) aminoethane-2-thiols and corresponding *protonated salts* .. ..

10. Bis (2-hydroxyethyl)sulphide (thiodiglycol) .. (111-48-8)

....

C. Schedule 2 part B

1. Amiton : O,O-Diethyl S-(2-(diethylamino)ethyl) phosphorothiolate and corresponding alkylated and protonated salts (78-53-5)

.2. PFIB : 1,1,3,3,3-pentafluoro-2-(trifluoromethyl)-1-propene 2/ (382-21-8).

..

..

..

1/ *A view was expressed that the question of exemptions needs further consideration.*

2/ The view was expressed that further consideration is needed on the whole question of the handling of by-products that pose a risk to the Convention.

D. Schedule 3 ..

1.	Phosgene	(75-44-5)
2.	Cyanogen chloride	(506-77-4)
3.	Hydrogen cyanide	(74-90-8)
4.	Trichloronitromethane (chloropicrin)	(76-06-2)
5.	Phosphorus oxychloride	(10025-87-3)
6.	Phosphorus trichloride	(7719-12-2)
7.	Phosphorus pentachloride	(10026-13-8)
8.	Trimethyl phosphite	(121-45-9)
9.	Triethyl phosphite	(122-52-1)
10.	Dimethyl phosphite	(868-85-9)
11.	Diethyl phosphite	(762-04-9)
12.	Sulphur monochloride	(10025-67-9)
13.	Sulphur dichloride	(10545-99-0)
14.	Thionyl chloride 1/	(7719-09-7)
15.	*Triethanolamine 1/ 2/*	*(102-71-6)*
16.	*Ethyldiethanolamine 1/ 2/*	
17.	*Methyldiethanolamine 1/ 2/*	
18.	*Fonofos 1/*	*(944-22-9)*
19.	*N,N-dimethylamino ethanol and corresponding protonated salts 1/*	*(108-01-0)*
20.	*N,N-diethylamino ethanol and corresponding protonated salts 1/*	*(100-37-8)*

..

1/ Views were expressed that this chemical should be removed from the Schedules.

2/ A view was expressed that the question of placement of these chemicals in the Schedules, including the feasibility of moving them from Schedule 3 to Schedule 2 part A, needs further consideration.

AHCCW	CD/CW/WP.363	Draft Report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament	21.8.91

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* * *

CONFERENCE ON DISARMAMENT

CD/CW/WP.364
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Ad Hoc Committee on Chemical Weapons

UNITED STATES OF AMERICA

A Chemical Weapons Convention

Staffing and Cost Estimates for a Technical Secretariat

INTRODUCTION

Several studies have been done on the composition, organization and operational requirements necessary to establish, implement and monitor a Chemical Weapons Convention (CWC).(1) Clearly, costs will depend on several factors, i.e., such as, decisions involving alternative verification schemes and the structuring of administrative bodies.

One aspect of the overall costs associated with establishing and implementing a Chemical Weapons Convention (CWC) is the operating administrative costs. Discussions concerning costs should be addressed parallel to other areas of the negotiations. Such discussions should not dictate desired implementation procedures, rather, the approach to administrative requirements is yet another factor to be considered.

In addition to administrative considerations, there are other elements of costs of a CWC that will need to be addressed, such as establishing the preparatory commission, national implementation costs, and national contributions to a CWC. This paper does not focus on any of the other elements in the belief each is important enough to warrant separate consideration. This paper is an attempt to look at the various administrative operating costs for a Technical Secretariat to a CWC. To assist in putting this in perspective a representative organizational chart has been developed. In developing staffing requirements, national trial inspection reports, US/Soviet bilateral visits and experience, as well as CD/1046 were used.(2)

(1) Estimation of Tasks For Size of and Necessary Resources for a Technical Secretariat Under a CWC - Dr. S. Johan Lundin, 19 June 1989; Inspection Costs for a Multilateral Chemical Weapons Convention - Institute for Defense Analyses, June 1990; The CWC and the International Inspectorate A Quantitative Study - University of Saskatchewan and External Affairs and International Trade, Canada, August 1990; Verifying the Projected Chemical Weapons Convention - A Cost Analysis - Herbert Beck, 1989; Size and Structure of A Chemical Disarmament Inspectorate - Netherlands, March 1984 (CD/445).

(2) Cut-off is January 1991.

COST FACTORS

The cost of implementing/monitoring a Convention will be a function of a number of parameters. Some -- such as the cost of monitoring storage facilities during the destruction period -- will vary greatly depending on the number of facilities, while some -- such as the cost of maintaining a data management facility -- will consist largely of a capital investment that is relatively less sensitive to marginal changes in workload.

The number of chemical weapons production, storage and destruction facilities, and the number of civil chemical industry facilities potentially capable of producing chemical weapons or their precursors, will largely determine the number of inspectors and the amount of monitoring equipment necessary to ensure verification of a CWC.

The number of facilities will also determine the extent of the Technical Secretariat staff needed to examine proposed verification schemes. For each type of facility, the cost of inspection will depend on:

-- The type of inspection: i.e., whether continuous, (resident inspectors), or occasional (visiting inspectors).

-- The extent and duration of an inspection -- the more intrusive it is, and the longer it is, the more time-consuming and manpower-intensive it will be.

-- The frequency of inspection, which depends in part on whether it is conducted on a periodic or random basis.

Other factors to be considered include automatic remote monitoring equipment where in certain instances reduced costs could result by shifting verification tasks from inspectors to automatic monitoring equipment with data recorded at a central facility. Support costs will be affected by such factors as the location of the administrative headquarters, the size of the staff required, and whether to construct a laboratory to analyze samples taken during inspections, or whether to contract this work with commercial laboratories.

THE TECHNICAL SECRETARIAT

The Technical Secretariat, as agreed to in Article VIII.D will be the operating arm of the international institution established to implement the provisions of the Convention.

The annual costs incurred by the Technical secretariat, following start-up and capitalization, will include:

-- Provide interface with national organizations, to receive and transmit reports, etc.

-- Review, process, analyze and report on declarations submitted by States Parties. The processing of declarations submitted by the States Parties will be an early and continuing cost to the Technical Secretariat. Initial declarations could require significant staff effort. Annual declarations will require continued technical and administrative effort at a moderate level.

-- Routine inspections; each declared facility subject to routine inspection will also require an "initial inspection". This will also include the need to negotiate a Facility Agreement between the Inspectorate and the facility to establish the content and scope of the subsequent inspections.

-- The frequency of inspection is partially defined in the rolling text, but will vary, inter alia, based on the type of equipment used in the inspection process. The use of unattended monitoring equipment could reduce the frequency of on-site inspections for a given level verification.

-- Inspections requested by States Parties.

-- Facilities of a specific type, i.e., CW stockpile, CW production, commercial schedule 2, etc. The number of inspected sites will be determined by the definitions and descriptions contained in the Convention.

-- Size and composition of the inspection team and duration of the inspection. Recent cost estimates have assumed inspection teams numbering from 3 inspectors for 3 days to 10 inspectors for 5 days for the same Schedule 2 production facility. The factor of 5 variation can only be reduced by definition of the inspection procedures for each type of facility. The type and amount of equipment available to the inspection team will also strongly influence both the required size of the team and the duration of the inspection. Until specific pieces of equipment and their characteristics are specified, it will not be possible to quantify this factor with precision.

-- Capital equipment used by the inspectors will cause acquisition and operation costs to be incurred by the Technical Secretariat. Definition of the types of equipment to be employed is necessary to quantify these costs. The equipment used in the inspection process is interrelated with the required frequency of inspection, the size of the inspection team, and the duration of each inspection. The inspection procedures adopted are the key to both understanding these relationships and estimating the direct and indirect costs of inspection equipment to the Technical Secretariat.

-- Analytic support for inspections requirements. This relates to the chemical analysis of samples collected by the inspectors and also data analysis conducted to support the planning and/or interpretation of inspections. The key information necessary to quantitatively assess the costs associated with analytic support are the procedures by which the different types of inspection will be conducted. The use of on-site equipment may reduce (or possibly increase) the need for analytic support; definition of inspection procedures will improve the accuracy of the cost estimates.

A number of recent efforts have attempted to define the inspection workload by the Convention. Even though many issues remain to be resolved, efforts are beginning to identify the staffing cost of a Technical Secretariat.

Several countries, participants in the Conference on Disarmament (CD), have conducted National Trial Inspections (NTIs) of military and commercial facilities which would be subject to inspection under the terms of the Convention. These NTIs are intended to examine the characteristics of various

types of inspections and define the scope of the required effort. It should be noted, however, that unlike the NTIs the international inspection team would require additional inspection procedures and support activities (e.g., translation and interpretation services).

Recent studies have attempted to estimate the scope and cost of the Technical Secretariat. While numerous assumption and estimating factors will need to be evaluated to ensure consistency between estimates, the general level of agreement among the studies argues that expected annual costs for implementation of the Convention can be specified within ±50%.

THE TECHNICAL SECRETARIAT

This section presents a suggested Secretariat organization, staffing projections and rationale. (See Attachment I for suggested Organization Chart.)

EXECUTIVE OFFICE

	PROJECTED STAFFING		
	MANAGER	PROFESSIONAL	TECHNICAL CLERICAL
Executive Office	5*	10**	10
TOTAL	5	10	10

*Includes Director General, Deputy Director General, and three Associate Directors General.
**Includes executive and special assistants.

* *

MAJOR FUNCTION(S): To oversee the business of the Technical Secretariat, report to the Executive Council on the activities of the Technical Secretariat, and assist the Conference of States Parties and the Executive Council in the performance of their duties.

ACTIVITIES:

1. Provide overall leadership, management, and representation of the organization.

2. Prepare monthly, quarterly and annual reports compiled from all Directorates under their organization and forward them to the Executive Council. Oversee special projects.

3. Provide liaison for the needs and requirements of the Executive Council and the Conference of States Parties and disseminate tasking to the various Directorates under them.

BASIS OF STAFFING ESTIMATE:

The Executive Office will be the central, controlling body for the Technical Secretariat. With an organization of approximately 1,225 people, it is estimated the Director General will require one deputy and three associate directors in order to adequately manage personnel, along with professional and clerical support.

ADMINISTRATIVE DIRECTORATE

	PROJECTED STAFFING			
	MANAGER	PROFESSIONAL	TECHNICAL	CLERICAL
Directorate Manager	1	1		1
Medical	1	3		1
Personnel Office	1	10	2	4
Travel Office	1	3	4	2
Legal Office	1	5	2	3
General Services	1	3	3	2
TOTAL	6	25	11	13

* *

MAJOR FUNCTION(S): To ensure the smooth operation of all ancillary responsibilities of the Technical Secretariat.

ACTIVITIES:

1. Prepare contracts and letters of consent from all States Parties. (Legal)

2. Hire all Technical Secretariat personnel. (Personnel)

3. Arrange the official travel requirements of inspectors and other personnel. (Travel)

4. Maintain an approved list of inspectors, by country. (Personnel)

5. Assist the Technical Secretariat in negotiating inspection agreements. (Legal)

6. Maintain a file of applicable national laws and provide advice and assistance to inspectors. (Legal)

7. Ensure that necessary operating supplies and furnishings are available to staff personnel. (General Services)

8. Maintain medical dispensary for Secretariat personnel; provide medical assistance/information to inspection teams; administer innoculations. (Medical)

BASIS OF STAFFING ESTIMATE:

This estimate is based on typical corporate structures for supervising an organization of the size envisioned and comparable existing international organizations.(1)

(1) References throughout this paper to "typical corporate structures" refers to work done on overall corporate structure and does not refer to any specific corporation. References to international organizations includes, but not exclusively, IAEA, NATO and OECD.

COMPTROLLER DIRECTORATE

	PROJECTED STAFFING			
	MANAGER	PROFESSIONAL	TECHNICAL	CLERICAL
Directorate Manager	1	1		1
Finance and Accounting Office	1	4	3	2
Payroll Office	1	2	2	2
Budget Office	1	2		2
TOTAL	4	9	5	7

* *

MAJOR FUNCTION(S): To ensure that all financial obligations (contributions) are met by the States Parties as well as oversee the financial operations of the Technical Secretariat.

ACTIVITIES:

1. Pay employees.

2. Prepare and manage the budget of the Technical Secretariat.

3. Coordinate and manage all capital outlay.

4. Maintain records of States Parties contributions, obligations, etc.

BASIS OF STAFFING ESTIMATE:

It is assumed this Directorate would be highly automated, using the latest computer and accounting technology and is based on a typical corporate structure for staffing.

SECURITY DIRECTORATE

	PROJECTED STAFFING			
	MANAGER	PROFESSIONAL	TECHNICAL	CLERICAL
Security Management	1	1		1
Building Security	1	5	20	1
Personnel Security	1	10	3	1
Document Security	1	4	5	1
TOTAL	4	20	28	4

* *

MAJOR FUNCTION(S): To provide building, personnel and document security against unauthorized use or disclosure.

ACTIVITIES:

1. Provide building security against unauthorized entrance of personnel and acts of aggression.

2. Provide personnel security against entrance by unauthorized personnel as well as obtain clearances and approval by States Parties for inspectors.

3. Provide document security against dissemination of confidential business information to unauthorized personnel.

4. Provide training and security briefings for Technical Secretariat; keep inspectors and others aware of any threats pertaining to official travel.

BASIS OF STAFFING ESTIMATE:

Security management will consist of the manager, one assistant and one clerical person to administer the routine office operations.

Building security will be a 24-hour-day, 7-day-a-week operation. This will require one manager to oversee operations and other duties in the building. The five professional staff members would be in charge of the various shifts and augment the guard staff during times of emergency. Based on three shifts and forty hours per week, it is estimated that twenty personnel would be needed for this function. This would provide approximately five personnel per shift. However, the size of the guard staff would be dependent on the size of the building.

Develop and maintain security files on Technical Secretariat personnel. This would include background investigations periodic follow-ups and secure computer support. Clearance of personnel will be a key measure by which confidential information can be completely acquired and safely maintained.

INSPECTION DIRECTORATE

	PROJECTED STAFFING			
	MANAGER	PROFESSIONAL	TECHNICAL	CLERICAL
Directorate Manager	1	5	3	1
Inspectors	1	581	314	1
Technical Systems	1	3	2	
Analytical Laboratory	1	10	24	
Equipment Division	1	9	12	
Training Division	1	10	5	
TOTAL	6	618	360	2

* *

MAJOR FUNCTION(S): To support the Technical Secretariat in its primary function of conducting inspections, and to provide analytic chemistry services.

ACTIVITIES:

1. Carry out all types of inspections as agreed to in the Convention.

2. Prepare required reports on all inspections.

3. Maintain capability to reliably analyze any samples collected by inspectors.

4. Create and distribute standards to qualify and provide quality assurance of participating laboratories.

5. Develop and validate standard methods of sampling.

6. Refine inspection procedures and train inspectors.

BASIS OF STAFFING ESTIMATE:

It was determined that inspectors would spend ninety (90) days per year (65 work days) in the field doing actual inspecting; 50 days planning and preparing for inspections (i.e., travel arrangements, information gathering, etc.); and 50 days in follow-up activities (i.e., report writing, etc.). The remainder of the time would be allocated to training and other activities.

A Management team would be established to ensure the proper allocation and control of personnel resources commensurate with the type of inspected facility. Ensure that inspections are executed in a timely and reliable manner, consistent with approved procedures and practices.

The Analytic Laboratory is staffed to allow operation of state-of-the-art automatic analysis instrumentation. It will develop procedures for testing and assure that these procedures are used in the participating laboratories. They will, however, do some analysis on samples collected by inspectors. It is not anticipated that the analytic laboratory will analyze all samples generated by the inspectors but rather assist participating laboratories in the workload.

The Equipment Division will test and evaluate proposed inspection equipment as well as upgrades to existing equipment. It will also certify the inspection equipment used by the inspectors. In addition to States Parties proposing specific equipment, this Division will make recommendations for equipment based on its own R&D work.

The Training Division will be responsible for generating all curricula materials as well as training the instructors and inspectors to ensure uniform inspection procedures. This will involve initial, follow-on and refresher training in all types of inspection; i.e., storage, production, destruction, use and commercial chemical facilities. This Division will provide initial, follow-on and refresher training in equipment use, records keeping, report writing, etc. It will also provide safety training appropriate to the proposed inspection. The Training Division would also be responsible for ensuring that established procedures are followed in the field by observing randomly selected inspections.

It was determined that a medical person should be part of an inspection team to deal with emergency situations. (This person would be a qualified nurse/corpsman.)

INFORMATION SYSTEMS DIRECTORATE

	PROJECTED STAFFING			
	MANAGER	PROFESSIONAL	TECHNICAL	CLERICAL
Directorate Manager	1	2		1
Communications	1	3	4	2
Central Computer Center	1	10	8	1
Language Services	1	20		6
Library	1	3	4	
Public Affairs	1	4		4
TOTAL	6	42	16	8

* *

MAJOR FUNCTION(S): Organize and catalog all information collected on inspections as well as provide information services to States Parties. Maintain liaison with national authority.

ACTIVITIES:

1. Establish a central computer center and a distributed micro-computer network for collection and dissemination of information on inspections.

2. Provide translation capability for all stored documents and a multilingual access to electronic and hard copy materials.

3. Establish a library of information on all aspects of chemical warfare (hard copy - archive) as well as a cross reference filing system.

4. Collect, organize and integrate data, such as reports from States Parties, from multiple sources.

5. Provide periodic reporting to support inspection activities.

6. Maintain informational archives to support current and future Technical Secretariat functions.

7. Maintain liaison with national authorities for routine day-to-day activities, such as reports, etc.

8. Develop and maintain an international communications system supporting the Technical Secretariat.

9. Develop and maintain public affairs information and dissemination.

BASIS OF STAFFING ESTIMATE:

It is assumed this Directorate would be a highly automated operation using the latest computer technology. Computer operations and other services will support the entire Technical Secretariat and especially the Inspection Directorate.

ORGANIZATIONAL STAFFING SUMMARY

	PROJECTED STAFFING			
	MANAGER	PROFESSIONAL	TECHNICAL	CLERICAL
Executive Office	5	10		10
Administrative	6	25	11	13
Comptroller	4	9	5	7
Security	4	20	28	4
Inspectorate	6	618	360	2
Information Systems	6	42	16	14
TOTAL	31	724	420	50

Combined Total for all Directorates and the Executive Office:

TOTAL 1,225

NOTE: *Managers are defined to include the Director General and Deputies, Heads of Directorates and Heads of Divisions. Heads of Section are included in Professional Staff.*

COST ESTIMATES

This section presents the estimates used in projecting the cost assessments of the Technical Secretariat. Cost estimates are stated in current (1991) US dollars.

TECHNICAL SECRETARIAT
CENTRAL OFFICE EQUIPMENT COSTS
($US)

EQUIPMENT	COST
Analytic Chemistry Laboratory	9,500,000
Computing Equipment[1]	
Central Office	2,000,000
Inspectorate (field lap-tops)	415,000
Library/Reference Materials	500,000
Inspectorate	
Safety (@$500 per person)	490,000
TOTAL	**12,905,000**

(1) Assumes mini-computer at $1,000,000, plus 100 microcomputers with LAN at $10,000 per unit.

TECHNICAL SECRETARIAT
OUTSIDE LABORATORY COSTS
($US)

Utilizing outside existing laboratories for testing and analyzing samples from inspections.

Initial cost of equipment and materials set up in an existing laboratory building	4,000,000
Annual costs thereafter for service contracts and expendable supplies	500,000

Staffing for each laboratory would include:

Manager	1
Laboratory Staff	12
Clerical	1
TOTAL	14

Contract cost per manyear is approximately $120,000
Contract Labor per year Total: 1,680,000

Annual costs for service contracts, expendable supplies, and labor, per laboratory:

TOTAL 2,180,000

This paper does not include a discussion of capital costs, but an exception has been made in this instance to show estimates for setting up a facility in an outside existing laboratory. Estimates, based on U.S. costs, are for equipment, laboratory space, and personnel required for an off-site trace analysis laboratory. Initial equipment and materials costs of $4 million is based on setting up a facility within an existing laboratory. The $4 million figure has not been included in the total estimated administrative operating expenses. The $500 thousand per year per laboratory operating expenses covers service contracts and expendable supplies. The $120,000 per manyear is derived from current US government contract costs. Approximately 62,000 square feet of floor space would be needed to accommodate the instrumentation, data systems, and offices. This would also include a small, separate, secure area for storage of CW agent standard reference solutions.

TECHNICAL SECRETARIAT
FIELD EQUIPMENT REQUIREMENTS
($US)

INSPECTION	FIELD SAMPLING/ ANALYSIS	MONITORING/ SEALS	COMMUNI-CATIONS	NO. OF TEAMS	COST PER INSPEC-TION TYPE
CW Weapons Stockpile	120 (a,c,d)	70 (70f; 35 sites)	20	6	3,290,000
CW Weapons Destruction	20 (a,d)	50 (3e;5f; 35 sites)	20	12	1,080,000
Former Production Fac	120 (a,b,d)	10 (10f; 35 sites)	5	2	600,000
Permitted Schedule 1	10 (d)	20 (2e;5f; 20 sites)	5	1	415,000
Commercial Schedule 2	120 (a,b,d)	30 (2e; 200 sites)	5	13	7,625,000
Commercial CW Capable	120 (a,b,d)		5	19	2,375,000
Challenge Inspections	220 (a,b,c,d)	10 (10f; 10 sites)	20	3	820,000
			TOTAL		16,205,000

Field Sampling/Analysis (Thousands $US)		Monitoring/Seals (Thousands $US)		Communications (Thousands $US)	
a. CW Detection Equipment	10	e. Process	15	g. Phone/FAX/ Data	5
b. Portable GC/MS	100	f. Entry/ Exit	1	h. On-Site Radio	15
c. Munitions Testing	100				
d. Sample Collection/ Transportation	10				

TECHNICAL SECRETARIAT
ESTIMATED TRAVEL COSTS
($US)

CLASS OF TRAVELER	ANNUAL COST PER TRAVELER	NUMBER OF TRAVELERS	ANNUAL COST
Executive/Manager	20,000	31	620,000
Professional Staff	45,000	724	32,580,000
Technical Staff	45,000	420	18,900,000
Clerical	20,000	5	100,000
		TOTAL	52,200,000

Assumes each inspector travels on inspections ninety (90) days per year. Travel costs of $500 per day are incurred, made up of $200 subsistence and $300 for commercial air fare. Other travel reflects attendance at conferences, symposia, training, etc.

TECHNICAL SECRETARIAT
LABOR COST
($US)

LABOR	NUMBER	DIRECT SALARY	POST ADJ. OVERHEAD[1]	TOTAL
Executive/Manager	31	2,892,623	1,446,296	4,338,919
Professional	724	48,038,211	24,019,032	72,057,243
Technical	420	17,253,803	8,626,853	25,880,656
Clerical	50	1,204,548	602,319	1,806,867
Totals	1,225	69,389,185	34,694,500	104,083,685

TOTAL	104,083,685

(1) Post adjustment costs include such items as relocation costs and allowances; overhead includes such items as insurance benefits, payroll taxes, expendable supplies and maintenance.

LABOR COST BY DIRECTORATE
(US$)

	DIRECT SALARY	POST ADJ. OVERHEAD	TOTAL
Executive Office	1,338,434	669,212	2,007,646
Administrative Directorate	2,089,168	1,044,578	3,133,746
Comptroller Directorate	986,247	493,121	1,479,368
Security Directorate	2,093,999	1,046,997	3,140,996
Inspectorate	59,378,605	29,689,195	89,067,800
Information Systems	3,502,732	1,751,397	5,254,129
Total	**69,389,185**	**34,694,500**	**104,083,685**

TECHNICAL SECRETARIAT
COST SUMMARY
($US)

EQUIPMENT COSTS (Per Year)[1]	1,843,500
FIELD EQUIPMENT COSTS (Per Year)[2]	3,241,000
OUTSIDE LABORATORIES (Per Lab/Per Year)[3]	2,180,000
TRAVEL COSTS	52,200,000
LABOR COSTS	104,083,685
TOTAL	163,548,185

As stated elsewhere, this paper does not address capital costs,[4] however, it is worth noting that if the decision is made to construct a facility to house the Technical Secretariat, an estimated $50 million US would have to be added to the "start up" costs, amortized over an agreed number of years, adding to the overall operating costs. The $50 million estimate is based on current rates to construct, finish, and furnish a facility to accommodate 1,225 people.

(1) Assumes a seven year life expectancy for equipment.

(2) Assumes a five year life expectancy for field equipment.

(3) Annual cost estimate is for one outside laboratory, should there be more than one lab, this cost figure would increase by that magnitude, in turn increasing the total costs.

(4) Following generally accepted practice in international organizations where taxes are not applicable, depreciation of capital equipment is not a factor, and thus has not been included in this paper.

TECHNICAL SECRETARIAT
INSPECTION TEAMS
TRAVEL COSTS
($US)

Type Inspection	Duration[1]	Team Size	Cost Per[2] Inspection ($US)
Permitted Schedule 1	4 days	11	22,000
Commercial Schedule 2	7 days	11	38,500
Commercial CW Capable	5 days	7	17,500
Challenge Inspections	12 days	21	126,000
Former CW Production	4 days	16	32,000
CW Stockpile	9 days	21	94,500

* *

Assuming a continuous presence at $500 per day for one year per site (a constant presence could result in reduced travel and related costs, i.e., leased quarters):

CW Destruction	365 days	33	6,022,500

(1) Includes Estimated Travel

(2) Travel and Per Diem Costs; See Page 19 for Equipment Costs.

(3) Duration and cost will depend whether there is a continuous presence and if so, if staff are "assigned" on a permanent or temporary basis; whether or not quarters and incentive pay are provided. Further work is needed.

TECHNICAL SECRETARIAT
TEAM COMPOSITION

Using CD/1046 as the basis, an estimate of the number of inspections possible annually with the number of inspection teams required to conduct them would be as follows:

Permitted Schedule 1	20 sites	1 team
Commercial Schedule 2	200 sites	13 teams
Commercial CW Capable	200 sites	19 teams
Challenge Inspections	10 sites	3 teams
Former CW Production	35 sites	2 teams
CW Stockpile	35 sites	6 teams

* *

CW Destruction	35 sites	12 teams

It is estimated a team would be composed[1] of the following:

Chemical Weapons Experts
Chemical Engineers
Analytic Chemicsts
Industrial Auditor
Interpreters
Instrument Technicans
Medical
Security

(1) It is recognized that in some instances team composition and size may vary depending on circumstances.

SAMPLE INSPECTION COSTS(1)
($US)

TYPE INSPECTION	TEAM SIZE	NUMBER OF TEAMS	NUMBER OF SITES	TOTAL COSTS
Permitted Schedule 1	11	1	20	1,465,900
Commercial Schedule 2	11	13	200	10,167,900
Commercial CW Capable	7	19	200	4,557,800
Challenge Inspections	21	3	10	3,096,600
Former CW Production	16	2	35	2,718,300
CW Stockpile	21	6	35	5,713,900
Exclusive of Destruction			TOTAL	27,720,400

* *

At this point, it is difficult to estimate the costs for inspection of CW destruction since little is known about the number of destruction sites and the length of operations at each site.

Costs per site per year:

CW Destruction	33	1	1	8,982,900

* *

Assuming a continuous presence at a CW destruction site for a three year campaign, approximately one-third of the destruction sites would be continuously staffed during the first ten years of treaty operations.

Average annual costs, assuming a total of 35 sites, each operating for three years:

CW Destruction	33	12	12	107,794,800
Including 12 Destruction Sites			TOTAL	135,515,200

(1) Costs include: Annualized Field Equipment for all teams per inspection type; Travel Costs; Salaries of Inspection Team.

NOTES:

-- Page 18: Outside laboratory cost estimates were provided by the U.S. Army Chemical Research, Development and Engineering Center.

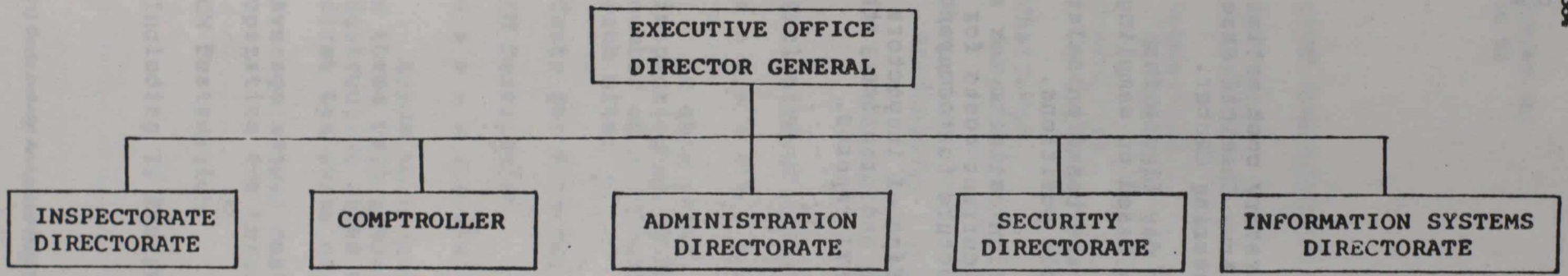
-- Page 20: The $500 per day figure for travel/subsistence is based on sampling.

-- Page 21: Labor costs are based on salary scales for the IAEA for comparable positions.

-- Page 23: The $50 million estimate for a new facility is based on current costs for construction and completing a structure for occupancy.

-- Scheduling and staffing of inspectors was drawn from IAEA experience and practice, other on-going CW discussions and NTI reports.

TECHNICAL SECRETARIAT



- On-site Inspectors
- Technical Instrumentation
- LABS: Sampling/ Analysis
- Equipment
- Assistants
- Training

- Payroll Office
- Accounting and Finance

- Personnel
- Travel
- Medical
- General Services
- Legal

- Building
- Personnel
- Document

- Language Services
- Library
- Communications
- Data Base Management
- Public Affairs

ATTACHMENT I

TECHNICAL SECRETARIAT
SAMPLE STAFFING OF INSPECTORS

TYPE OF INSPECTION	CW EXPERTS	CHEMICAL ENGINEER	ANALYTIC CHEMIST	INDUSTRIAL AUDITOR	INTER-PRETER	INSTRUMENT TECH	MED	SEC
Permitted Sch 1 Prod.	1	5	1	0	2	1	1	0
Cmcl Sch 2	0	4	1	2	2	1	1	0
CW Capable	0	2	1	1	1	1	1	0
Challenge Inspections	3	4	2	1	2	2	1	6
Former Production Facility	3	8	1	1	2	0	1	0
CW Stockpile	10	0	1	1	2	0	1	0
CW Destruction	7	10	5	2	4	1	3	1

ATTACHMENT II

CONFERENCE ON DISARMAMENT

CD/CW/WP.365
23 August 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

R O M A N I A

Information regarding Romanian export controls of precursors, equipment, plants or components therefor which could be used in the manufacturing of chemical weapons

In the implementation of the non-proliferation policy of the Government of Romania and based on the Government's decisions on the exports and imports regime, the Minister of Foreign Affairs, the Minister of National Defence, the Minister of Industry and the Minister of Trade and Tourism issued jointly Order no.40 on the control of exports which could contribute to the proliferation of weapons of mass-destruction and of the missiles carrying such weapons, published on July 8, 1991 in the official national document, containing all laws and other regulations, "Monitorul Oficial al Romaniei".

In accordance with the provisions of this Order, exports and imports for reexporting of chemical weapons precursors, equipment, plants or components therefor, listed in the Annex, which is an integral part of Order no.40, are subject to the terms and conditions for obtaining a licence, provided in Order no.49 of December 15, 1990 of the minister of trade and tourism.

When applying for licences for export or import for reexporting of the items listed in the Annex, economic agents shall ensure the strict compliance with the international obligations undertaken by Romania regarding non-proliferation and export control.

The Order provides that the Ministry of Foreign Affairs, the Ministry of Trade and Tourism, the Ministry of National Defence and the Ministry of Industry shall mutually advise and consult on the export applications for the chemical weapons precursors, equipment, plants or components therefor listed in the Annex, so as to ensure the compliance with the international commitments undertaken by Romania in the non-proliferation field.

Also under the provisions of Order no.40, the above - mentioned ministries and, as the case may be, other field ministries, shall periodically review, on the basis of existing records and customs statistics, the import and export licences for the purpose of taking the measures provided for by the international understandings that Romania has agreed to.

The Order came into force on the date of its publication in "Monitorul Oficial al Romaniei".

ANNEX

LIST OF PRECURSORS AND EQUIPMENT, PLANTS OR COMPONENTS THEREFOR WHICH ARE SUBJECT TO EXPORT CONTROLS FOR PREVENTING CHEMICAL WEAPONS PROLIFERATION

I. Precursors:

1. Thiodiglycol
2. Phosphorus Oxychloride
3. Dimethyl Methylphosphonate
4. Methyl Phosphonyl Difluoride (DF)
5. Methyl Phosphonyl Dichloride (DC)
6. Dimethyl Phosphite (DMP)
7. Phosphorus Trichloride
8. Trimethyl Phosphite (TMP)
9. Thionyl Chloride
10. 3-Hydroxy-1-methylpiperidine
11. N,N-Diisopropyl-(beta)-Aminoethyl Cloride
12. N,N-Diisopropyl-(beta)-Aminoethane Thiol
13. 3-Quinuclidinol
14. Potassium Fluoride
15. 2-Cloroethanol
16. Dimethylamine
17. Dimethyl Ethylphosphonate
18. Dimethyl N,N-Dimethylosphoramidate
19. Dimethyl Phosphite
20. Dimethylamine Hydrochloride
21. Ethyl Phosphinyl Dichloride
22. Ethyl Phosphonyl Dichloride
23. Ethyl Phosphonyl Difluoride

24. Hydrogen Fluoride
25. Methyl Benzilate
26. Methyl Phosphinyl Dichloride
27. N,N Diisopropyl (beta) Amino/Ethanol
28. Pinacolyl Alcohol
29. O-Ethyl 2-Diisopropylaminoethyl Methylphosphonite (QL)
30. Triethyl Phosphite
31. Arsenic Trichloride
32. Benzilic Acid
33. Dimethyl Methylphosphonite
34. Dimethyl Ethylphosphonate
35. Ethyl Phosphinyl Difluoride
36. Methyl Phosphinyl Difluoride
37. 3 Quienolidene
38. Phosphorus Pentachloride
39. Pinacolone
40. Potassium Cyanide
41. Potassium Bifluoride
42. Ammonium Bifluoride
43. Sodium Bifluoride
44. Sodium Fluoride
45. Sodium Cyanide
46. Tri-ethanolamine
47. Phosphorus Pentasulphide
48. Di-isepropylamine
49. Diethylaminoethanol
50. Sodium Sulphide

II. Equipment, plants or components therefor:

1. Plants that are suitable, directly or through minor modifications, for the production of the chemical weapons or precursors, and work with reactors with a volume of 0.1 m^3 or more, and that are made of very corrosion-proof metals (such as high-grade steels with a share of more than 35% of alloy components or nickel-based alleys, tantalum, titanium and zirconium materials, silicon casts, Hastelloy, Monel), or of very resistant plastics (fluorcarbon polymers) or of glass, graphite or ceramics materials or that is coated or enamelled with these materials;

/...

2. Components which are suitable and have been modified especially for the plants listed in head (1):

- pumps for chemical plants, instrument panels, agitators with sealed shafts, pipes ready to be installed, especially suitable for the protection of the discharge of highly toxic substances;

- reactors with a volume of 0.1.m^3 and more (except enamelled reactors with up to 4 enamel layers);

- heat exchangers;

3. Especially modified components, as follows:

- filling and capping machines for the chemical weapons precursors specified in Chapter I above into containers;

- combustion equipment with combustion chamber temperatures of more than 1,000°C or catalytic combustion over 350°C and subsequent flue gas treating equipment;

- exhaust devices with high flow rates at 3,600 m^3/h and more, or locally effective exhaust devices with subsequent treating equipment for the aforementioned toxic substances;

- waste water treating equipment for the aforementioned toxic substances;

- gas warning devices and their especially designed major components for the aforementioned toxic substances with detection limits below 50 ppb;

- equipment for safety sluice devices for plants for the aforementioned toxic substances.

USA	CD/CW/WP.366 Rev.1	Report on the fourth United States trial inspection exercize NOT REPRODUCED (see WP volume)	Also issued as CD/1107 Rev.1 23.8.91

* * *

CONFERENCE ON DISARMAMENT

CD/CW/WP.367
7 October 1991

ENGLISH
Original: RUSSIAN

Ad Hoc Committee on Chemical Weapons

UNION OF SOVIET SOCIALIST REPUBLICS

MAIN TECHNOLOGICAL ASPECTS OF THE DESTRUCTION OF CHEMICAL WEAPONS

(An approach proposed by Soviet experts)

Hitherto, problems involved in the destruction of chemical weapons have usually been settled in the various countries within the framework of national programmes mainly designed for the destruction of defective munitions unsuitable for use or of obsolete CW agents kept in storage containers. As a result of progress at the talks on general and complete chemical disarmament, aspects of the technical solution of this problem are increasingly attracting attention beyond national frontiers and becoming a subject of interest for all countries in the world community. The complexity of finding a solution to the task is due, to a large extent, to the variety of types of CW agent, ranging from phosphorus-containing to sulphur-containing and arsenic-containing substances that differ in physical, chemical and toxicological properties, and also to the specific features of the design of chemical munitions.

All this variety of substances and types of munitions makes it necessary to develop, as far as possible, universal methods of destruction applicable to all forms of CW agents and munitions. In every specific case, the decisive criterion for the technological aspects of the process is that they should ensure to the utmost safe conditions of work and the protection of the environment. Meanwhile, the technology itself must take into account the specific features of work with supertoxic substances and comprise tried and tested techniques and approaches, not forgetting irreplaceable practical experience in destroying chemical weapons at the experimental stage of the work. This last circumstance is of particular importance, since the experience gained makes it possible to develop and put into practice measures to ensure safe working conditions and the protection of the environment.

These measures, taking into account the fact that working with extremely toxic chemicals is involved, include sanitary, hygienic, fire-protection and ecological standards, regulations covering technical supervision and also legal obligations anchored in legislation. It is precisely this regulatory basis that is the main requirement when developing technological processes for destroying chemical weapons.

The existing technology in the USSR is based on the use of a two-stage process for destroying chemical weapons.

In the first stage sarin, soman, VX and mustard gas are detoxified and reaction products of low toxicity are obtained, i.e. one of the tasks is completed - the chemical warfare agent is irreversibly transformed into a product unsuitable for further use for warlike purposes. Meanwhile it must be emphasized that the process is discontinuous, thus making it possible to localize any emergency and restricting the amount of toxic material being processed at any one time to a strictly limited minimum. Tc this must be added that each operation in detoxification must be monitored.

The main reagent used for destroying sarin, soman and mustard gas is monoethanolamine, whereas for VX the detoxification agent is a formula based on ethylene glycol and orthophosphoric acid in a ratio of 1:1.

Trace amounts of the chemical warfare agents are found in the reaction product obtained, at a level of 1.10^{-5} mg/ml. It should be noted that the reaction product consists of a complex, multicomponent system. For that reason the quantitative titration of the agent at a level of 10^{-5} per cent is quite a laborious and lengthy task, so that while the detoxification process is completed in one hour, analysis to a sensitivity of 1.10^{-4} mg/ml requires a further 40-90 minutes.

In plants for dismantling chemical munitions, items may also be processed one by one, thus considerably reducing the scale of any possible emergency. At this stage the munition is opened, the CW agent is removed under vacuum and the inner surface of the container body is decontaminated. Meanwhile, when munitions are being destroyed that contain sarin, soman or mustard gas the degassing agent used is the basic reagent for the detoxification reaction - monoethanolamine; this makes it possible in each dismantling process to degas the inner surfaces of the contaminated fittings and the tubes and pipes, thus making it easier to determine the correct amounts of reagents to be used.

The second stage of the process is the incineration of the low-toxicity products resulting from detoxification of the CW agent and also the burning of the thoroughly degassed munition casings. Thus, tried and approved technical solutions are being used in the Soviet Union.

Experimental work was carried out on the method of direct incineration of the CW agents, but the method was not adopted, since it requires a very high degree of reliability in the equipment used in order to exclude even the tiniest discharge of highly toxic substances in any emergency situation in the furnaces or in the decontamination plant, and to ensure that is extremely difficult and expensive. Nor is it possible to be satisfied with chemical detoxification alone. For that reason a double screen was created, to guarantee both the completeness of the destruction and, as a result of the selected sequence of stages, its safety.

This design of the technological process is extremely reliable. It has been tried out and tested in the course of scientific research and experimental design work on the destruction of defective munitions and of various forms of chemical munitions piecemeal or in small batches. In the

course of this work some thousands of chemical artillery shells and self-propelled missiles, mines, aerial bombs, rocket devices and rocket nose cones containing almost 300,000 kg of toxic substances of all kinds were destroyed. The destruction took place without any accident or anything out of the way occurring.

The ecological reliability and effectiveness of the technological processes was confirmed and more precise knowledge gained of a number of important parameters; the reliability and efficiency of ecological monitoring with the means of analysis developed was also confirmed.

In 1987 in Shikhany the representatives of countries participating in the Geneva talks saw a demonstration of a mobile complex and the destruction of an aerial bomb charged with sarin.

In accordance with the traditional arrangements for carrying out such work, the establishment of high-capacity plant was preceded by the setting-up of a pilot scheme in the city of Chapaevsk. It was proposed to use four continuous belts for the munition dismantling process in order to try out the technological processes for destroying most types of munitions and all types of organophosphorus CW agents. The capacity of the pilot plant ranged from 100 to 500 tons per 100 working days, depending on the size of the munitions concerned.

Work in this plant was limited to tests in simulated environments. The efficiency of the main units and assemblies and of the systems for managing and monitoring the environment was demonstrated.

The problem of destroying lewisite and substances based on it must be regarded from a somewhat different point of view.

When it was a matter of destroying organophosphorus CW agents, their destruction led in the final analysis to the formation of inorganic compounds of phosphorus which are vitally important elements in the natural cycle. It is precisely in that fact that lay the possibility of further work to utilize the mineral salts for commercial purposes.

When, however, arsenical compounds are concerned, special factors arise connected with the fact that practically all compounds of arsenic are toxic and consequently national record-keeping is required at all stages where arsenic is present.

For that reason, when processes were being developed in the USSR for destroying lewisite the pathway chosen was chemical binding of arsenic to form low-toxicity material for burial.

The process developed for destroying lewisite and mustard gas-lewisite formulas is based on carrying out detoxification with a melt of elemental sulphur and forming a water-insoluble polymer which is subsequently buried.

However, the problem of destroying lewisite can be regarded also from another point of view, that of arsenical raw material supplies, since arsenic forms something of the order of 30 per cent of the CW agent concerned.

In this connection also, there is some interest in the process of transforming lewisite by chlorination into arsenic trichloride, which is subsequently converted into pure arsenic for commercial use.

Thus, we have available at the moment sufficiently reliable and well-developed processes for dismantling practically all forms of munitions and detoxifying all varieties of CW agent.

It should be noted that the technological processes recommended today for destroying chemical weapons are the result of thorough analysis and experimental study both of traditional techniques and of a number of alternative methods of detoxifying CW agents. Among the traditional methods are the quite well-known techniques for degassing CW agents based on the use of chlorinating agents, alkaline formulas, oxidants, etc.

Among the alternative methods considered in the USSR, mention must also be made of thermochemical and photochemical destruction of CW agents in low-temperature plasma; thermochemical destruction based on adiabatic compression; thermal destruction of CW agents in a closed space; direct incineration of CW agents; destruction of chemical weapons by nuclear blasting; destruction of CW agents in deep boreholes with the help of the natural magma; use of thermal energy sources, etc. Undoubtedly these possibilities are of interest even as regards the destruction of industrial waste. However, in such a specific field as the destruction of chemical weapons they are either insufficiently universal in their application or fail to meet requirements.

CONFERENCE ON DISARMAMENT

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Ad Hoc Committee on Chemical Weapons

UNION OF SOVIET SOCIALIST REPUBLICS

ENVIRONMENTAL ASPECTS OF THE DESTRUCTION OF CHEMICAL WEAPONS

(An approach proposed by Soviet experts)

The destruction of chemical weapons prompts a measure of concern on the part of people living in the immediate vicinity of CW storage facilities and in the areas where CW destruction facilities are located. There is a need for guarantees of the safety of the destruction methods adopted, the irreversibility of the decontamination processes and the dependability of surveillance of destruction at all stages. The urgency of this problem is also underlined in the bilateral agreement between the USSR and the United States, which provides that each party will, during the destruction of its chemical weapons, give the highest priority to ensuring the safety of persons and the protection of the environment.

In this way, safety issues extend beyond national borders and constitute a matter of interest for other countries within the world community.

In such circumstances, "commercial confidentiality" becomes of secondary importance in the face of the parties' efforts to check whether correct technical choices have been made for the purpose of guaranteeing the safety of actual operating conditions for CW destruction facilities. It is this approach which gives rise to the need for the fullest possible exchange of information with specialists on such key matters as methods and specific technologies for CW destruction, safety measures, and protection of people and the environment.

The main aim of such cooperation is a more multifaceted approach to environmental issues, the selection and evaluation of the safest technological processes and the improvement of measures to monitor facility activities.

In this way, the key criterion for evaluating the destruction process is the need to ensure to the utmost safe conditions of work and the protection of the environment.

In the view of Soviet experts, these requirements are best met by a two-stage, discontinuous process of destruction of chemical weapons, with the mandatory implementation of the following measures:

zoning of process stages according to level of danger, with corresponding changes in air pressure;

organization of automatic control in all sectors through which the munitions pass, with the halting of operations if regulatory standards are breached;

dismantling of munitions in special boxes equipped with exhaust ventilation and contact air cleaners;

reduction of the number of sources of impacts by means of decontamination of munition casings and tubes and pipes immediately after removal of the CW agent;

use of a vacuum to transport CW agents and decontaminants along pipes;

use of a system to monitor the movement of the munition at the processing stage, as well as the quantity of products removed during dismantling as they reach the destruction stage;

constant surveillance of the air in the dismantling units, ventilation outlets, production areas, industrial area and health protection zone.

These measures will be incomplete if steps are not taken to deal with ways and means of organizing analytical and environmental monitoring of the CW destruction processes.

Analytical monitoring embraces all stages of CW agent destruction and decontamination of containers. It includes:

qualitative and quantitative analysis of CW agents awaiting destruction;

verification of the completeness of CW agent destruction;

quantitative analysis of the content of CW agents in circulating decontaminants and intermediate and final wastes;

verification of the completeness of decontamination of surfaces of munitions, equipment and protective gear.

In the process of analytical monitoring every possible effort is made to rule out the possibility that the operational personnel will come into contact with chemically harmful substances.

This is achieved through the following measures:

Automatic taking of samples from containers used in the process;

Airtight packaging of samples and delivery to the laboratory by pneumatic tube conveyor;

Opening of containers using a manipulator, preparation of samples for analysis and their transfer through an airlock to the analysts' workstations.

Monitoring of the completeness of destruction of CW agents is accompanied by laboratory analysis of components of the environment against national health standards.

Of key importance in selecting a method for destroying chemical weapons is an evaluation of the technical safety of the processes involved and the environmental safety of the destruction facilities as a whole. The evaluation consists of an analysis of all stages of the movement of chemical munitions, together with a forecast of possible hazard situations and their consequences in terms of environmental contamination.

The system for forecasting hazard risks is complex and includes, together with technical and environmental aspects, economic, psychological, biological, sociopolitical, international and other factors.

These factors are each considered independently. The use of technical means of forecasting hazard situations is a vital element in the atmospheric monitoring system.

However effective and reliable the destruction process may be, and however up-to-date the measures taken to ensure safe operation, a CW destruction facility is by its nature a potential source of environmental contamination.

In this context, monitoring of chemical pollution is a tool of objective monitoring of the safe implementation of CW destruction processes, independently of the technologies adopted, as well as a tool for monitoring and reporting on the development of possible hazard situations.

As far as the destruction processes under review are concerned, the basic functions of the atmospheric monitoring system consist of:

Constant surveillance of processes of munition dismantling and destruction, focusing on the presence of CW agent vapours in the air of plant premises, to permit timely notification of the appearance of local sources of contamination as a result of breaks in the process or equipment sealing failure;

Surveillance of gaseous and atmospheric discharges;

Monitoring of the air against established maximum permissible concentrations.

These functions are performed using various items of gas analysis equipment, each of which has a different function and operating principle, but which complement one another, thus enhancing the reliability and trustworthiness of the information obtained.

Detection equipment makes it possible to collect, process and display the information received from the various sectors of the process, and is functionally linked with the warning, notification and cut-off system.

At the heart of this surveillance system are automatic gas detectors using the biochemical method of analysis to a sensitivity at the level of the maximum permissible concentrations of organo-phosphorus CW agents, and also ionization sensors with a sensitivity of between 10^{-2} and 10^{-3} mg/m, but with a reaction time of 2-3 seconds, which is compatible with the speed at which hazard situations develop and permits maximum speed in identifying and locating a source of air pollution.

Together with stationary environmental monitoring equipment, portable apparatus for observing local sources of air contamination or observing contamination of surfaces is proposed for use, as is a mobile chemical laboratory to analyse impacts on various environmental objects.

In order to ensure safety in operations in the industrial zone of CW destruction facilities, the operating personnel are provided with protection gear which affords reliable protection against the possible impact of CW agents in the vapour and droplet phases. The protection capacity is 200 mg/dm^2. Work shifts in protection gear should not exceed four hours.

After completion of operations, the operating personnel in protection gear undergo obligatory rinsing with decontaminating fluids, followed by contamination checks in detection chambers.

To put into effect these approaches to ensuring the safety of people and the environment, more concrete and technical measures have been drawn up which have been tested in experiments involving the destruction of virtually all types of CW agents.

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Ad Hoc Committee on Chemical Weapons

UNION OF SOVIET SOCIALIST REPUBLICS

Complex for the destruction of faulty chemical munitions (KUASI)

Kuasi is designed for the destruction in the field conditions faulty chemical munitions filled with sarin, soman, VX and containing no explosives.

The complex provides safe destruction of chemical munitions with minimum pollution of the environment by both chemical warfare agents and their destruction products in accordance with the standards effective in the USSR.

Productive capacity of the complex in the agent to be destructed	- up to 90 kg/hr
Staff personnel (without supporting services)	- 17 pers
Maximum power	- 200 kWt
Current, frequency	- alternate, 50 Hz
Supply voltage	- 380/220 V
Complex total mass	- 66.3 t
Time of deployment	- 10 hr
Site dimensions	- 100 x 100 m

The complex consists of the following:

SPECIAL TECHNOLOGICAL EQUIPMENT

- chambers for draining munitions of different types - 3
- reactor of chemical agent detoxication - 1
- units of technological shut-off fittings - 2
- unit of vacuum support - 1
- sets of assembly parts, accessories and spare equipment

AUXILIARY EQUIPMENT

- mobile chemical laboratory - 1
- chemical reconnaissance car - 1
- unit of liquid waste incineration - 1
- mobile compressor station - 1
- mobile diesel power station - 1
- tank truck - 2
- lift truck of 5 t load capacity - 2
- truck with two trailers for transportation of technological equipment - 1

MEANS OF ANALYTICAL AND GAS CONTROL

- automatic gas alarm PAS-83 - 1
- express-detector NR05 - 2
- local contamination detector - 2
- gas liquid chromatograph "Tsvet-550" - 1

The complex can be moved to the destruction work site under its own power (with the use of two truck tractors), by railway and air transport.

In the area of future operations a preliminary survey is carried out with the aim of providing safe destruction, organizing site preparation, guard and examination of local background of the environment.

EXPERIENCE OF KUASI OPERATION

In the period 1980-1990 the destruction of more than 4,000 munitions of various calibres, filled with sarin, soman and VX, was carried out with the use of KUASI. Total mass of disposed chemical warfare agents is ca 200 t.

KUASI TECHNOLOGY

KUASI technology is based on the method of chemical agent detoxication by suitable reagents followed by incineration of produced reaction masses.

Operations of munition draining (opening, chemical agent evacuation, body decontamination) are carried out in one of the special air-tight chambers (depending on the munition calibre), in which constant negative pressure is maintained. The agent is evacuated to the detoxication reactor by means of vacuum, produced in it, through the siphon introduced into the opening drilled in the munition body. Then, the munition body is filled with ethanolamine that is transferred to the detoxication reactor. After monoethanolamine evacuation the munition body and the draining chamber is washed by the decontamination solution, that is then pumped to one of the tank trucks (ARS). After washing the decontaminated munition body by 1 per cent alkali solution (with the definite stand and analysis of washing solution) the draining chamber is opened, and the munition body is transferred to roasting into the combustion furnace. In this furnace the destruction of previously decontaminated capping, packing and other combustible materials is carried out. Spent alkali solution is pumped out to the tank truck, designed for receiving the reaction mass. To the preset amount of the chemical agent, accumulated in the detoxication reactor, a reagent is added up to the norm, the contents is mixed and heated to the required temperature and then is kept for 30-45 minutes. The samples of the obtained reaction masses are analyzed for residual content of the chemical agent. When analysis results conform to the preset standards, the reaction mass is discharged to ARS tank trucks, where it is diluted with an additional quantity of water or alkali solution (ratio 1.1), previously used for reactor cooling.

Diluted reaction mass and decontamination solutions are destructed in the liquid waste incineration unit.

REQUIREMENTS FOR MUNITIONS AND CONTAINERS RECEIVED FOR DESTRUCTION

Chemical munitions and agents containers, received for destruction should meet the following requirements:

- absence of explosives and pyrotechnical devices;
- absence of excessive agent vapour pressure;
- leak-proofness and absence of body contamination.

For meeting these requirements, entry control of munitions and containers should be organized. It consists of two stages.

At the first stage, the munitions are subject to primary sorting on the storage area and are prepared to transfer for destruction.

Munitions and containers, after the primary sorting and preparation, are marked with indicator paints (welds and filling unit).

The second stage is carried out on a destruction site for the purposes of registrating and revealing leaking munitions and containers after their transfer.

SAFETY AND MEDICAL ASPECTS

One of the principal requirements for chemical weapons destruction plant is ensuring the safety of personnel involved in plant operation and any local civilian population.

Technological process of chemical weapons destruction, its equipment design, accident prevention system provide the necessary plant safety level, i.e. safety standards should be met for operating area ($2* 10^{-5}$ mg/m^3 for sarin) and for atmosphere air of populated areas ($2* 10^{-7}$ mg/m^3 for sarin).

In regular performance of technological destruction process, i.e. when sarin vapours concentration are equal or lower than maximum allowable concentrations (MAC), its vapour concentration in the atmosphere (taking into account vented air purification efficiency 0.9999) would be essentially lower than MAC values for populated areas.

In emergency situations, e.g. spilling sarin out of technological equipment or munitions to be destructed to trays of technological room, at a period of accident liquidation about 0.5 hour, sarin vapour concentration (taking into account vented air purification) at a distance 3 km would be $2.5* 10^{-11}$ mg/m^3 i.e. 10,000 times less than MAC value for populated areas.

In emergency situations during loading and unloading 0.05-1 t sarin filled munitions in CW destruction plant, contamination zone depth with casualty toxic dose (0.055 mg.min/l) would be not more than 1 km.

During incineration of sarin detoxication reaction masses in a furnace as a part of CW destruction plant, waste gases of the following compositon are generated:

P_2O_5	C = $2.87* 10^3$ mg/m^3
HF	C = 813 mg/m^3
NO_2	C = 50.4 mg/m^3
CO	C = 300 mg/m^3

The values of maximum ground level concentrations in different distances from emission source, as well as their comparison with emission standard for populated area atmosphere, are contained in the table below.

Substance	Concentration in a distance 1 500, 2 000 and 3 000 m, mg/m^3	MAC values (USSR) mg/m^3	Other accepted standards for atmosphere air quality, mg/m^3
P_2O_5	1 500 m - 0.209 2 000 m - 0.123 3 000 m - 0.061	0.01	0.1
HF	1 500 m - 0.059 2 000 m - 0.035 3 000 m - 0.017	0.005	0.05
NO_2	1 500 m - 0.0036 2 000 m - 0.002 3 000 m - 0.001	0.085	0.1
CO_2	1 500 m - 0.0218	1	10

Thus, the size of sanitary protective zone for CW destruction plant, taking into account forementioned information, should be about 2,000-3,000 m.

In case of necessity to meet USSR emission standards for populated areas in relation to P_2O_5 and HF, the device of gas waste cleaning is supplied in addition to liquid waste incineration unit.

The plant location area is declared to be a prohibited one, within its limits the suitable guard is organized and environmental control is carried out periodically.

The territory adjacent to the main technological building, with facilities placed on it, is a technical site, on which standards and regulations for harmful chemical productions are effective and required air monitoring is carried out. On the technical site a motor vehical decontamination station is foreseen; two vehicles - ARS - 14 tank trucks, filled with decontamination solution (5 per cent NaOH) and water as well as fire truck for fire extinguishing are in permanent readiness to liquidate accidents. A special team of the taking over shift conducts elimination of emergency situation, and a special crew carries out fire extinguishing, both are equipped with the isolating skin and respiratory protective equipment and are ready to cooperate in emergency elimination.

ORGANIZATIONAL AND TECHNICAL SAFETY MEASURES

In the CW destruction plant, the engineering, technological process design and equipment arrangement ensure maintenance of the following main principles of destruction process organization:

- realization of agent chemical detoxication in mild conditions without possibility to convert the agent into combat state;

- periodicity of agent detoxication process with limitation of quality of the agent under processing, what provides localization of possible emergency situations;

- agent transport through pipings by vacuum;

- realization of munition opening, agent evacuation and body decontamination in special chambers with negative pressure 5-10 mm of water column in relation to the environmental atmosphere;

- chemical decontamination of munition body and container internal surfaces before their thermic treatment;

- placing the plant parts, where agent spilling is possible, in closed rooms, equipped with ventilation with air cleaning (on absorbent filters);

- stack emission cleaning (by means of absorbent filters);

- limitation of reaction mass incineration capacity of the plant, that provides the harmful contaminant absence above MAC values in the air on sanitary zone boundary;

- moving the stack emission point off the ground in a distance, which excludes all harmful contaminant concentrations arising on the sanitary zone boundary, on the ground level above MAC values for populated areas.

SAFETY MEASURES FOR WORKERS

The plant may be operated by persons being at least 18 years old, healthy and specially trained.

Before taking over the shift the personnel is subject to special medical examination, and receives necessary instructions.

Immediately before the agent destruction operation the personnel puts on the isolating skin protective equipment and filtering gas masks, followed by their checking in the gas mask fumigation chamber.

All operations relative to entry control, munition and container opening and decontaminating, as well as to sampling agents and its chemical detoxication products out of technological facilities, are carried out by personnel having to wear full individual protective equipment (IPE).

L-1 protective suit in the absence of damage may be used, after proper treatment, up to 20 times (depending on operating conditions).

IPE, which are unfit for use, whose present use cycle is over, or contaminated with agent vapours or drops are destroyed by incineration after preliminary decontamination.

In the time of operation the personnel watches closely the cleanness and condition of IPE and, if required, carries out its partial or total decontamination.

After completion of operation, the personnel, before taking off IPE, carries out its total decontamination followed by water washing. Taking off IPE before its decontamination is prohibited.

Before taking off IPE, its cleanness is controlled with ILS-85 (ILS-1) instrument. Upon detection of the IPE contamination, IPE is additionally decontaminated.

After taking off IPE, the personnel is subject to sanitary treatment and medical control in the medical supervision point.

This point is equipped with all means of emergency medical treatment of chemical casualties as well as an ambulance to evacuate a patient to the hospital.

In addition to the point, there are first aid means for chemical casualties (antidotes) in the special first-aid set, placed in the technological building and AL-4M mobile laboratory.

CHEMICAL AGENT DETECTION AND MONITORING SYSTEM

The monitoring system - it is a set of measures and instruments used for control of CW agents. It is one part of the system ensuring the safety of the total incineration system from the point of view as of personnel and of the environment.

The monitoring system consists of three levels of control:

The first level - internal control, which includes:

- qualitative and quantitative analysis of CW agents which has to be destructed and, if necessary, the identification of them;
- quantitative determination of the CW agents in liquid and solid wastes, which have to be incinerated.

The first level of control is based on the methods of sample preparation, methods of quantitative analysis used in laboratory and on semi-quantitative express test methods. It may be as local control and in mobile chemical laboratory (AL-4M). The chemical, biochemical and GC methods are used for this a control.

The limit of detection for CW agent type "G" in different technological liquids is $1 * 10^{-4} - 1 * 10^{-3}$ mg/cm^3. The time necessary for one determination is 30-60 min.

The second (intermediate) level - of workplace ambient monitoring, which includes:

- ambient air monitoring in the main industrial part of the system;
- ambient air monitoring in the auxiliary part of the system;
- monitoring of the contamination of the surface as of the technological equipment and of protective individual clothing.

The workplace ambient monitoring of is based on the use of automatic gas analysers, portable testing boxes for indicating of local contamination, laboratory quantitative methods of CW agent analysis of air and surfaces, and semi-quantitative express test methods. This monitoring is carried out as at the most potentially dangerous areas and in the mobile chemical laboratory (AL-4M) with the help of biochemical method and the limit of detection corresponds to the maximum allowable concentration of CW agent in the workplace.

Automatic gas analysers are used in the rooms where the most potentially dangerous work is carried out (the draining of CW agent from containers and the detoxication of them, the decontamination of the bodies of munitions and etc.) by the personnel, which has to be all the time in protecting clothing.

The principle of the standard gas analyser, (PAS-83) which is included into the set of the equipment of the system is based on the ionization of compounds being present in air.

The limit of the detection for "G" type CW agent - $(1-5) * 10^{-2}$ mg/m^3 and the time constant is about 5 sec.

Three sensing elements are supplied with every gas analyser.

Express tests with the help of the kit of fine chemicals (NR05, NR06) are used if it necessary to determine low concentrations of "G" type CW agent. The time necessary for the determination is 2-5 min.

The portable automatic indicator of local contamination (type ILZ) is used when it is necessary to find out the source of leakage of CW agent and to control the contamination of the different surfaces. The limit of the detection for sarin in this case is $(1-5) * 10^{-2}$ mg/m^3.

The time constant, depending on modification of the instrument, is 2-30 sec. Express analysis of the solutions, which were formed after washing out of contaminated surfaces, can be made, if necessary, with the help of the kit of fine chemicals (NR14).

The more sensitive gas analysers are used for air control in the auxiliary rooms and outdoor areas:

- automatic portable gas analyser of biochemical type, model SBM-1; the limit of the detection for sarin is $(1-5) * 10^{-5}$ mg/m^3 and time

constant - about 7 min; it is necessary to use the kits of fine chemicals (NR02 and NR03) with this analyser, it can be used at the temperature from +5° C to +35° C;

- automatic stationary gas analyser (ASK) which can be used from -40° C to +50° C; the limit of the detection (for sarin) is (1-5) * 10^{-5} mg/m^3, this type of analyser is used at the perimeter of the technological area.

The express test or quantitative analysis of air is carried out in the auxiliary rooms or technological areas with the help of biochemical method, if necessary; the limit of the detection in this case corresponds to maximum allowable concentration.

The third (external) level of monitoring - environmental control, includes:

- control of vent emission;
- control of stack emission;
- control of ambient air on the sanitarian protective board;
- control of the environment.

The environmental control is based on the use of gas analysers, express test kits, methods of sample preparation and methods of the laboratory quantitative analysis. This control is fulfilled as in the field and in the mobile chemical laboratory (AL-4M) after samples have been supplied to it. The chemical and biochemical methods are used in this case and their limit of the detection corresponds to maximum allowable emissions and maximum allowable concentrations for populated areas.

Automatic gas analyser model ASK is used for control of vent emissions. It is necessary to recharge this analyser with new kit of fine chemicals (NR02 or NR03) once per three days.

The regular express control is possible using kit of fine chemicals (NR05 together with NR06).

The control of stack emission is fulfilled regularly with the help of methods of quantitative analysis the limit of detection of which is on the level of maximum allowable emissions.

The gas analysers for control of main components of stack emission (such as NO_x, CO, etc.) can be supplied, if necessary.

The control of ambient air on the board of sanitarian protective zone can be fulfilled, if necessary, with the help of corresponding methods of quantitative analysis.

The limit of the detection in this case is on the level of maximum allowable concentrations for populated area.

The control of plants, soil and other environmental objects can be fulfilled with the help of corresponding methods of analysis, if necessary.

The taking of air samples in required places is made with the help of portable device (model MP-1) or according to the corresponding methods.

Appendix 1

AUTOMATIC INSTRUMENTS FOR AMBIENT AIR CONTROL

Technical characteristics and price	Model of the instrument and sampling devices					
	PAS-83	JLZ-85	JLZ-1	ASK	SBM-1	MP-1
1. The designation	Alarm in case of accident	The determination of leakage	The determination of leakage	Environmental control	The control of safety	Sampling
2. The limit of the detection for GB, mg/m^3	$(1-5)*10^{-2}$	$(1-5)*10^{-2}$	$(1-5)*10^{-2}$	$(1-5)*10^{-5}$	$(1-5)*10^{-5}$	-
3. The time constant	to 5 sec	to 2 sec	to 30 sec	(30 and 45 min)	to 7 min	1,3,10,15 min
4. Kits	-	-	-	NR02, NR03	NR02, NR03	solid solvents and liquid absorbents
5. The range of temperatures in which the instrument can be used, °C	-10 ÷ +25	-20 ÷ +40	-40 ÷ +40	-40 ÷ +50	+5 ÷ +35	-10 ÷ +40
6. Power supply, V.	220+/-22 (220+/-20)	220+/-20 6(accumulator)	127/220+/-20 6(accumulator)	220+/-22	220+/-22 25(battery)	220+/-33 27(battery)

Technical characteristics and price	Model of the instrument and sampling devices					
	PAS-83	JLZ-85	JLZ-1	ASK	SBM-1	MP-1
7. Power consumption, wt.	about 100	about 50	about 50	about 500	about 200	about 200
8. The time of continuous operation without recharge, hours	continuous	not less than 8	not less than 6	45 and 72	not less than 6	2-6
9. The dimensions of the control and alarm units, correspondingly	375x130x171 520x390x198	130x80x70 270x120x210	820x60x150 225x100x250	-	395x160x315	398x202x208
10. The weight of the control unit, kg	3* 5 15	total 5	total 4.5-7.5	30 20	13(without batteries)	13(without batteries)
11. The numbers of instruments being used in the project	2	2	2	5	3	2
12. The approximate price, thousands dollars	20	15	10	40	25	20

Appendix 2

MOBILE CHEMICAL LABORATORY AL-4M

The mobile chemical laboratory (AL-4M) is intended to use for the determination of CW agents in different samples in field conditions.

This laboratory as a part of incineration system KUASI allows to organize all technological and environmental control during the destruction of CW agents.

The standard set of laboratory equipment used in this laboratory provides the opportunity for:

- qualitative and quantitative determination of CW agents in samples of water, food, air, soil, plants, forage and on the surfaces of equipment and munitions;

- physico-chemical investigation of CW agent's samples, taken from the different munitions (shells, ammunitions, etc.);

- selective control of the degree of decontamination of munitions, weapons, military equipment, etc. and ground;

- investigation of the quality of chemicals used for decontamination and selective control of these compounds.

The standard set of equipment of the mobile chemical laboratory (AL-4M) also allows to carry out:

- qualitative and quantitative analysis of CW agents to be destructed;

- control of the decomposition degree of CW agents being destructed, of the decontamination degree of munition bodies and of different surfaces;

- control of CW agent content in different technological liquids and in wastes to be incinerated;

- the determination of decontamination materials quality and of components of mixtures being used for the decontamination;

- express control of ambient air for content of CW agents and quantitative determination of their content in the environment.

The high selective and high sensitive determination of the traces of CW agents being destructed in products of decontamination and identification of CW agents and the determination of their purity degree is possible when GC model "Tsvet 550" with AFID and FPD is used as option to equipment of mobile chemical laboratory AL-4M.

When such instruments and equipment as SBM-1, ILZ-85, MP-1, NR-14, etc. are used as options to the equipment of AL-4M the automatic control of ambient air, the determination of the sources of local contamination of different surfaces and express control of ultralow concentrations of nerve gases with the help of test kits is possible in the environment.

If the mobile chemical laboratory is used in conditions of hot climate it is necessary to use an air-conditioner, small refrigerator and to locate this laboratory under the roof or camouflage net (the square is about 100 m^2).

Technical characteristics of the mobile chemical laboratory AL-4M

1. The limit of detection of "G" type CW agents:

in ambient air - $1* 10^{-4}$ - $1* 10^{-6}$ mg/m^3 (biochemical method)

in the reaction mixtures and liquid waster, and water

- $1* 10^{-4}$ - $1* 10^{-7}$ mg/cm^3 (biochemical method)

- $1* 10^{-4}$ - $1* 10^{-5}$ mg/cm^3 (GC method)

on the surface of the equipment, munitions and protective clothing

- $1* 10^{-4}$ mg/dm^2 (biochemical method)

2. The throughput of the laboratory during 10 hours operation:

- about 20-100 samples (depending on the sample type when CW agents and other chemicals are analysed).

3. The power consumption is about 10 kwt.

4. The laboratory consists of two departments:

- analytical one for carrying out biochemical analysis (on the base of truck)

- the department which is used for sample preparation and carrying out GC and chemical analysis (on the base of the trailer).

5. The total weight of the laboratory is about 11 tons (the weight of the analytical department is about 7.5 tons and of the sample preparation department - 3.5 tons); the total length of the laboratory is about 12 m. The dimensions of the laboratory: the analytical department - 7100*, 2440*, 3300 and the sample preparation department - 5600*, 2180*, 2120.

6. The maximum speed on highway - 50 km/hour.

7. The safety requirements for the personnel are fulfilled even when the external air is contaminated by CW agents. It is possible owing to the filtration with FVUA-100N-12. The maximum allowable concentrations are given in the instruction manual for this laboratory.

Appendix 3

THE INDICATING ENAMEL HV-5145

The enamel HV-5145 is a suspension of the indicating compound, pigments and fillers in the solution of polymer resin with the plastificator in the organic solvent. This enamel is designated for detecting leaks of the "G" type CW agents from chemical munitions when they become not leakproof during storage or transportation. It is applied with the help of pneumatic spraying or brush on places of the surface of munitions (welds, sealed assemblies) where leakage may take place. The enamel is applied over the anticorrosive coating or just on the surface of the metal.

The principle of functioning of the enamel is based on the selective indicator solubility when the CW agent contacts with the indicating coating.

The colour of the indicating coating is grey. When the drops of the "G" type CW agent fall on this coating, the bright fuchsia (violet-red) colour has to appear. This colouring appears in case of "GB" in 3-5 seconds at 20° C and in 1-2 seconds at 50° C. This time for "GF" is 15-30 seconds and 1-2 seconds, correspondingly.

The limit of the detection for the "G" type CW agents, when the indicating enamel is used, is about 0.1 mg.

The lower limit of the temperature, at which the indicating enamel can be used, is -40° C for "GB" and -15° C for "GF".

The time, necessary for the detection in these cases, increases to 0.5-5 hours.

The indicating coatings HV-5145 are very selective. They do not change their colour and do not fail under the influence of the high humidity (even 100 per cent), rainfall, drops of gasoline, toluene, white spirit, ethanol, lubricants, solutions of ammonium hydroxide and sodium hydroxide. The vapours of hydrogen chloride, nitrogen oxides, sulphur dioxide and ammonium do not change properties of this coating even when their concentration is about 1 mg/l. It is only necessary to exclude the occasional falling of drops of such active solvents, as acetone and dimethylformamide, on this coating.

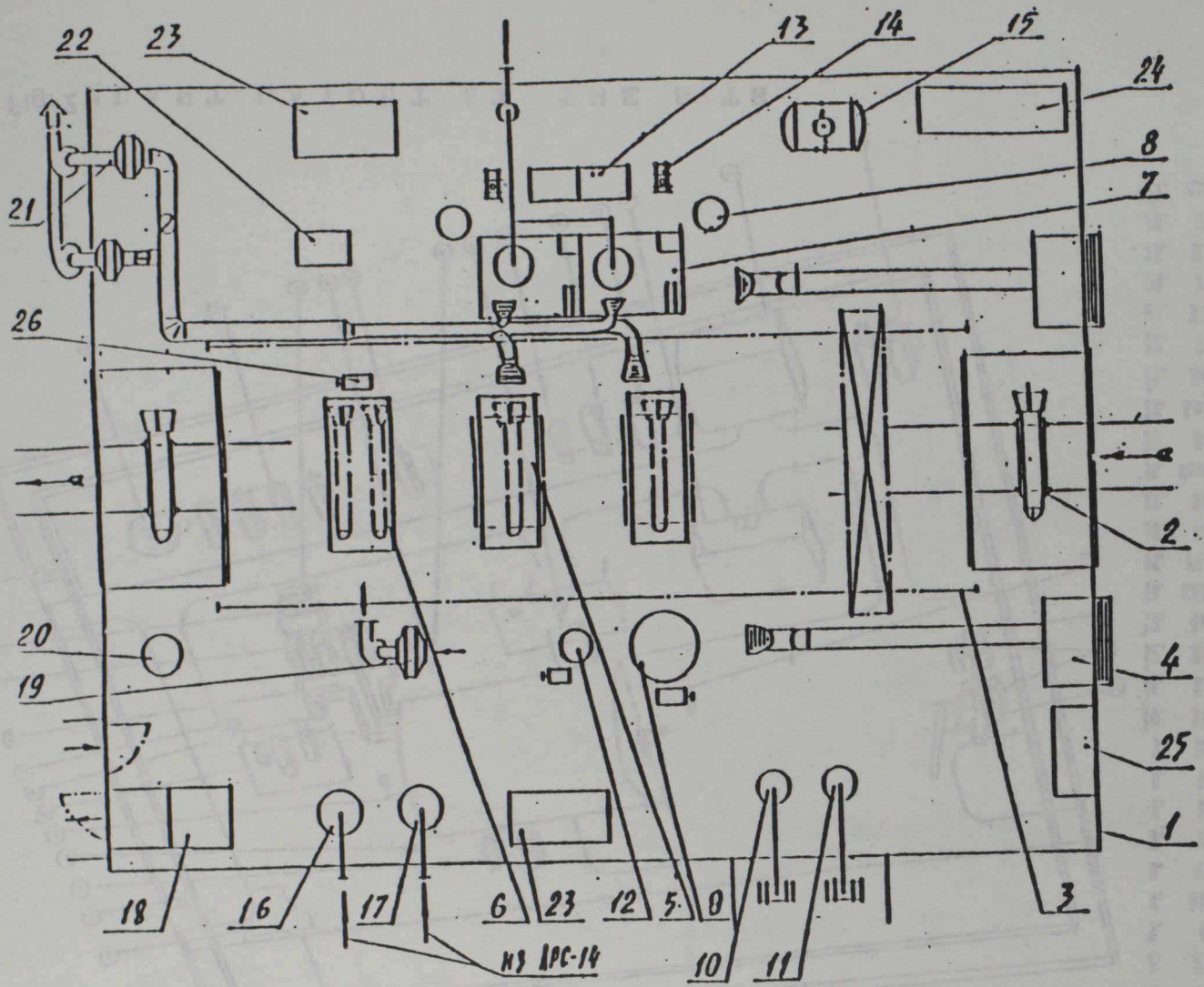
The time, necessary for drying the indicating coating when the thickness of it is 20-30 mcm, is not more than 15 min. at 20-25° C.

The adhesion of this coating on the oily, polyvinyl chloride and anticorrosive coatings is reliable.

Coating of munitions with the enamel is carried out usually in rooms or chambers provided with the suction-and-exhaust ventilation. It may be carried out at the outdoor area under a roof too.

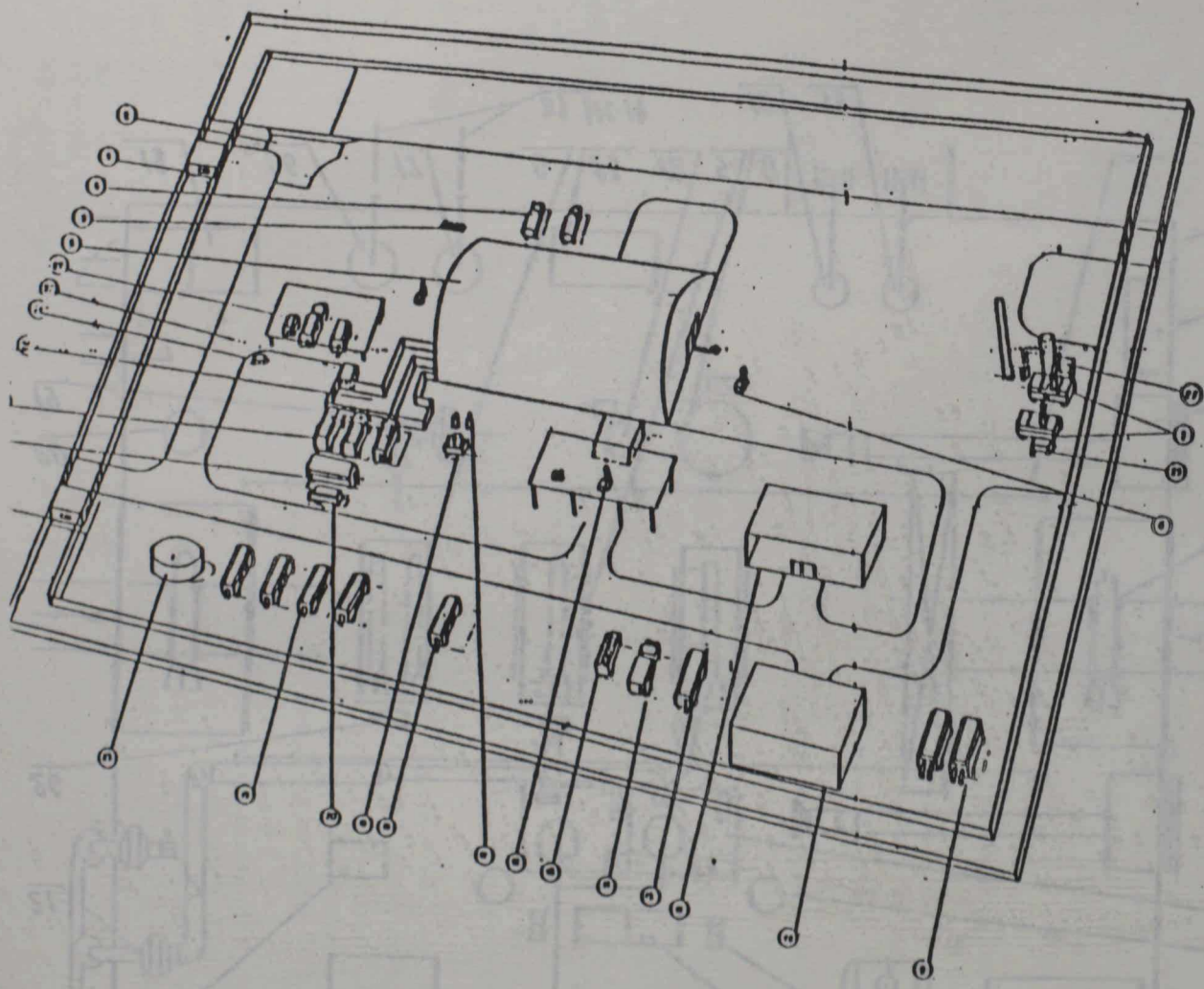
The enamel HV-5145 is a fire hazardous substance. The fire extinguishing means are the same that are usually used for organic solvents: felting, sand and carbon dioxide type fire extinguisher.

Keeping in mind, that the enamel is produced in accordance with single orders, the price of it is $28 per kilogramme.



1. Technological building
2. Industrial truck
3. Crane jib Q – 3 tc
4. Air conditioner
5. Drain chamber
6. Alkaline chamber
7. Detoxication reactor
8. Intermediate vessel
9. Circulation vessel for the solution
10. Pressure vessel for the solution
11. Pressure vessel for the reagent
12. Dischange vessel for the reagent
13. Vacuum unit
14. Spray separator
15. Emergency vessel
16. Pressure vessel for water
17. Pressure vessel for alkali
18. Shower for the individual protective equipment
19. Cleaning system
20. Drain vessel for wastes
21. System of local suctions
22. Compressor
23. Fire prevention board
24. Maintenance area
25. Electric board
26. Pump

Fig. 1 EQUIPMENT LAYOUT
I.N THE TECHNOLOGICAL BUILDING



1. Check point
2. Medical supervision
3. Area of drained pieces for shipment
4. ARS - 14 truck
5. Technological building
6. Truck loader
7. Incineration unit
8. Mobile laboratory
9. Mobile power station
10. Store for spare equipment & accessories
11. Raw material store
12. Reagents store
13. Amenity rooms
14. Maintenance shop
15. Fire - fighting vehicle
16. Control unit
17. Bucking plant
18. Decontamination shower station
19. Medical unit
20. Ambulance
21. Medical supervision
22. Tent for gas mask fumigation
23. Fire - fighting vessel with water
24. Passage gallery
25. Mobile compressor
26. Vessel for wastes
27. Cleaning unit (if required)

Fig. 2 PLANT LAYOUT AT THE SITE

CONFERENCE ON DISARMAMENT

CD/CW/WP.370
9 October 1991

Original: ENGLISH

Ad hoc Committee on Chemical Weapons

GERMANY

(Western Group CW Co-ordinator)

VERIFICATION IN CHEMICAL INDUSTRY UNDER ARTICLE VI

Informal Discussion Paper

1. Working Group B of the Ad hoc Committee on Chemical Weapons has focused in 1991 on the question of verification in chemical industry under Article VI. The course of discussions in Working Group B as well as working and discussion papers tabled in 1991 suggest that a substantial revision of the relevant provisions in the Rolling Text will be necessary to find a consensus approach. Work done in the interim is reflected on page 129 of CD/1108 of 27 August 1991. However, no comprehensive picture of a revised verification system for chemical industry has emerged as yet.

2. The German Delegation, in its capacity as Western CW co-ordinator, undertook a series of consultations in the Western Group on the question of how to revise the verification system under Article VI. On the basis of these consultations this paper was prepared to facilitate drafting work in Working Group B.

3. The main thrust of the paper is to provide an illustrative structure (in treaty language) for a revised verification system in chemical industry. The paper builds on the two existing Annexes 2 and 3 to Article VI (Western delegations concluded that such an approach, although not necessarily their preferred one, might be most likely to find consensus). Following this approach, the paper sets forth a range of options on a number of key questions. These options, although not comprehensive, reflect not only views of Western Delegations but also proposals from outside the Western Group. Further options could easily be included.

4. Footnotes attached to current Annexes 2 and 3 of Article VI (cf. pages 120-27 of CD/1108) are not addressed in this paper.

GE.91-62579

ARTICLE VI

It is suggested that the text of Article VI itself in its new version as contained in CD/1108 on page 129 (former B/7/Rev.2) remains basically unchanged except for its last paragraph, which should be amended as follows:

> "9. For the purpose of on-site verification, each State Party shall grant to the inspectors access to facilities as required in the Annexes to this Article, in accordance with the Protocol on Inspection Procedures and the Annex on the Protection of Confidential Information."

References to Article VI in this working paper relate to the new version as contained in CD/1108 on page 129 (the numbering of the paragraphs in Articles VI old and new is different).

DEFINITIONS

(Cf. paper CW/FCTM/PC/11 of 3 September 1991; placement in the Convention to be decided later)

1. Definitions related to activities of a facility

"Production" of a chemical means the formation of a chemical through chemical reaction, including rearrangement, or through biochemical reaction.

"Biochemical reaction" means a chemical reaction involving a biological medium (i.e. living organisms).

"Processing" of a chemical means a physical process, such as formulation, extraction and purification, in which the chemical is not converted into another chemical.

"Consumption" of a chemical means its conversion via a chemical reaction into another chemical.

"Discrete organic chemical" means any organic chemical compound, identifiable by chemical name, structural formula and, if assigned, Chemical Abstracts Service registry number.

2. Definitions related to the organizational structure of the chemical industry

"Facility" in the context of Article VI means any of the industrial sites defined below.

"Complex" ("Combine") means an area comprising two or more autonomous plant sites.

"Plant site" ("Works", "Factory") means the local integration of one or more plants, with any intermediate administrative levels, which are under one operational control and includes common infrastructure, inter alia:

- administration and other offices
- repair and maintenance shops
- medical centre
- utilities
- central analytical laboratory
- research and development laboratories
- central effluent and waste treatment area
- warehouse storage.

"Plant" ("Production facility", "Workshop") means a relatively self-contained area, structure or building containing one or more units with auxiliary and associated infrastructure, which could include, inter alia:

- small administrative unit
- storage/handling areas for feedstock and products
- effluent/waste handling/treatment area
- control/analytical laboratory
- first aid service/related medical unit
- records associated with the movement into, around and from the site, of declared chemicals and its feedstock or product chemicals formed from it, as appropriate.

- **"Unit"** ("Production unit", "Process unit") means the combination of those items of equipment, including vessels and vessel set up, necessary for the production, processing or consumption of a chemical.

ANNEX 2 TO ARTICLE VI

Régime for Chemicals on Schedule 2 Parts A and B and Facilities related to Such Chemicals

I. DECLARATIONS

The Initial and Annual Declarations to be provided by a State Party under paragraphs 3 and 4 of Article VI shall include:

A. Declarations of aggregate national data

1. Aggregate national data for the previous calendar year on the quantities produced, processed, consumed, imported and exported of each chemical listed in Schedule 2, as well as a quantitative specification of import and export for each country involved.

2. Such quantities shall be calculated on the basis of ... (to be elaborated:
- in case of production, processing and consumption on the basis of individual facility data above which threshold?
- in case of foreign trade on the basis of individual export and import transactions above which threshold?)

B. Declarations of plants

1. General

Declarations are required for:

(a) All plants that produced, processed or consumed during any of the previous 3 years or are anticipated to produce, process or consume in the next year

- more than 1 tonne of a chemical listed in Schedule 2 A, or

- more than [...] of a chemical listed in Schedule 2 B. 1/

(b) Plants that produced at any time [since 1 January 1946] [during the 15 years prior to the entry into force of the Convention] a chemical in Schedule 2 for chemical weapon purposes.

1/ The question of thresholds for chemicals listed in Schedule 2 B as well as the question of adding toxins to Schedule 2 B need further consideration.

2. Declarations on past activities

For each plant, declarations shall include the following information on Schedule 2 chemicals as well as on the plant itself (as well as any other information considered appropriate):

Chemical(s)

(a) The chemical name, common or trade name used by the plant, structural formula, and Chemical Abstracts Service Registry Number (if assigned).

(b) The total amount produced, processed, consumed, [imported and exported] in the previous calendar year or, in the case of the initial declaration as required in Article VI paragraph 3, in each of the three previous calendar years.

(c) The purpose(s) for which the chemical(s) are produced, processed or consumed:

(i) processing and consumption on-site (specify product type);

(ii) sale or transfer within the country (specify, if to other domestic industry, trader or other destination with an indication, if possible, of final product type);

(iii) direct export (specify which country);

(iv) other - specify.

Plant

(d) The name of the plant and of the owner, company, or enterprise operating the plant.

(e) The precise location of the plant (including the address, location of the plant site, location of the plant within the plant site including the specific building and structure number, if any).

(f) The main orientation (purpose) of the plant.

(g) Whether the plant is dedicated to producing, processing or consuming the listed chemical or is multi-purpose.

(h) The approximate production capacity of the plant [for the declared Schedule 2 chemical(s)].

(j) Which of the following activities are performed with regard to the Schedule 2 chemicals:

(i) production;

(ii) processing;

(iii) consumption;

(iv) other - specify (e.g. storage).

3. Notifications of anticipated activities

The notifications relating to anticipated activities as required in paragraph 1 shall follow the same format as provided for in the preceding paragraph. In addition, the anticipated time period(s) of production, processing or consumption are to be notified.

C. Procedural Provisions

Each State Party shall submit, when the Convention enters into force for it,

1. initial declarations not more than 30 days later (Article VI, paragraph 3),

2. annual declarations relating to past activities by the end of March for the preceding calendar year, starting in the year which follows the year of entering into force,

3. annual notifications relating to anticipated activities by the end of October for the following calendar year. Subsequently planned notifiable activities in the same reporting year shall be notified not later than [5] days before this additionally planned activity begins. The first annual notification is due by the end of the first October during which the Convention has been in force.

D. Information to States Parties

The list of plants declared under this Annex together with the information provided under paragraphs [...] shall be transmitted by the Technical Secretariat to all States Parties within 30 days after declarations have become due.

II. VERIFICATION

1. General

(a) International on-site verification provided for in paragraph 6 of Article VI shall, under this Annex, be carried out by the Technical Secretariat through routine inspections of those of the declared plants which produced, processed or consumed during any of the previous 3 years or are anticipated to produce, process or consume in the next year

- more than 10 tonnes of a chemical listed in Schedule 2 A, or

- more than [...] of a chemical listed in Schedule 2 B.

(b) The draft programme and budget of the Organization to be submitted by the Executive Council pursuant to Article VIII paragraph 20(e) shall contain, as a separate item, an indicative draft programme and budget for verification under this Annex. [..] % of the resources available for verification under Annexes 2 and 3 of Article VI shall be devoted to verification under this Annex.

(c) The Technical Secretariat shall

(i) perform initial inspections of declared plants in accordance with paragraph 2 below.

(ii) select plants for routine inspections in accordance with paragraph 3 below.

2. Initial Inspections

Each plant specified in paragraph 1 (a) above shall receive an initial inspection as soon as possible but preferably not later than [3] years after entry into force of the Convention.

3. Routine Inspections

(a) Having received the initial inspection, each plant specified in paragraph 1 (a) above shall be subject to routine inspections.

(b) In selecting particular plants for inspection, the Technical Secretariat shall:

(i) give due consideration to the risk to the objectives of the Convention posed by the relevant chemical, the characteristics of the plant and the nature of the activities carried out there;

(ii) take into account, on the basis of subsequent declarations, such operational modifications of plants it deems relevant.

(iii) choose the particular plant to be inspected in such a way as to preclude the prediction of precisely when the plant is to be inspected.

(iv) not inspect one plant more than twice per year.

4. Inspection Aims

The general aim of inspections shall be to verify that activities are in accordance with obligations under the Convention and with the information provided in declarations on individual plants. Particular aims of inspections at plants declared under this Annex shall include verification of:

(a) consistency with declarations of levels of production, processing or consumption of Schedule 2 chemicals;

(b) the absence of non-declared chemicals listed in Schedules 1, 2 or 3 above thresholds for declarations;

(c) non-diversion of chemicals listed in Schedule 2 for purposes prohibited under the Convention.

5. Inspection Procedures

Inspections shall be carried out in accordance with agreed guidelines and other relevant provisions of the Protocol on Inspection Procedures and the Annex on the Protection of Confidential Information.

(Note: the relevant provisions in the Protocol on Inspection Procedures remain to be elaborated in sufficient detail to obviate the need for individual facility agreements [unless a facility agreement for a specific facility is requested by the State Party concerned or by the Technical Secretariat].)

ANNEX 3 TO ARTICLE VI

Régime for Chemicals on Schedule 3, Facilities Related to Such Chemicals, and Other Facilities Relevant to the Objectives of the Convention

I. DECLARATIONS

The Initial and Annual Declarations to be provided by a State Party under paragraphs 3 and 4 of Article VI shall include:

A. Declarations of aggregate national data

1. Aggregate national data for the previous calendar year on the quantities produced, [processed,] consumed, imported and exported of each chemical listed in Schedule 3, as well as a quantitative specification of import and export for each country involved.

2. Such quantities shall be calculated on the basis of ...
(to be elaborated:
- in case of production, [processing] and consumption on the basis of individual facility data above which threshold and in which ranges?
- in case of foreign trade on the basis of individual export and import transactions above which threshold?)

B. Declarations of [plant sites] [plants]

1. General

Declarations are required for all [plant sites comprising one or more]

(a) plants that produced, [processed] or consumed [at any time since entry into force] [during the previous year] or are anticipated to produce in the next year more than 30 tonnes of a chemical listed in Schedule 3.

(b) plants that produced at any time [since 1 January 1946] [during the 15 years prior to the entry into force of the Convention] a chemical in Schedule 3 for chemical weapons purposes.

(c) plants that produced [at any time since entry into force] [during the previous year] or are anticipated to produce in the next year more than [..] tonnes of a discrete organic chemical, except those that only produce chemicals containing only carbon and hydrogen and those that only refine petroleum. 1/

1/ Further consideration needs to be given to the specific declaration and verification requirements with respect to facilities using biochemical reactions or extraction from natural sources, and which are "capable" of producing more than [...] of listed chemicals.

[(c) plants that have a production capacity of more than [..] tonnes of a discrete organic chemical containing the elements phosphorus, fluorine or sulphur [or those involving the processes of phosphorylation, fluorination or sulphurylation,] identical to those chemicals included in Schedule 1 as well as in Schedule 2.]

2. Declarations on past activities

(a) Declarations required under paragraph 1 (a) above shall include the following information on the Schedule 3 chemical(s):

(i) The chemical name, common or trade name used by the facility, structural formula, and Chemical Abstracts Service Registry Number (if assigned).

(ii) The approximate amount of production, [processing] and consumption of the chemical in the previous calendar year, expressed in the ranges: up to 100 tonnes specified to the nearest 10 tonnes, up to 1,000 tonnes specified to the nearest 100 tonnes, and above 1,000 tonnes specified to the nearest 1,000 tonnes.

(iii) The purpose(s) for which the chemical(s) are produced, [processed] or consumed.

(b) Declarations required under paragraph 1 (a), (b) or (c) shall include the following information on the plant site and its plants:

(i) The name of the plant site and of the owner, company, or enterprise operating the plant site.

(ii) The precise location of the plant site including its address.

(iii) The number of plants within the plant site which fall under the definitions of paragraph 1 (a), (b) or (c) above.

(iv) Within the plant site the number of plants which are declared under Annex 2 to Article VI.

[(v) The name of the plant(s) declared under this Annex and of the owner, company, or enterprise operating the plant(s) if different from the information provided for the plant site under subparagraph (i) above.

(vi) The precise location of the plant(s) within the plant site including the specific building and structure number, if any.

(vii) The main orientation (purpose) of the plant(s).

(viii) The approximate production capacity of the plant(s).]

3. Notifications of anticipated activities

The notifications relating to anticipated activities as required in paragraph 1 shall, with the exception of a reference to [processing and] consumption, follow the same format as provided for in the preceding paragraph. [In addition, the anticipated time period(s) of production are to be notified.]

C. Procedural Provisions

Each State Party shall submit, when the Convention enters into force for it,

1. initial declarations not more than 30 days later (Article VI, paragraph 3),

2. annual declarations relating to past activities by the end of March for the preceding calendar year, starting in the year which follows the year of entering into force,

3. annual notifications relating to anticipated activities by the end of October for the following calendar year. [Subsequently planned notifiable activities in the same reporting year shall be notified not later than [5] days before this additionally planned activity begins.] The first annual notification is due by the end of the first October during which the Convention has been in force.

D. Information to States Parties

The list of [plant sites] [plants] declared under this Annex together with the information provided under paragraphs [...] shall be transmitted by the Technical Secretariat to all States Parties within 30 days after declarations have become due.

II. VERIFICATION

1. General

(a) International on-site verification provided for in paragraph 6 of Article VI shall be carried out by the Technical Secretariat through routine inspections at facilities 1/ declared under this Annex.

(b) The draft programme and budget of the Organization to be submitted by the Executive Council pursuant to Article VIII paragraph 20 (e) shall contain, as a separate item, an indicative draft programme and budget for verification under this Annex.

1/ The term facility is used here and in the following subparagraphs only provisionally; it should be replaced eventually by either "plant site" or "plant".

(c) The selection of facilities for inspection shall be performed by the Technical Secretariat on the following basis:

(i) Each State Party has the right annually to nominate [at least [5]] [different] facilities for inspection. The maximum number of facilities a State Party may nominate for inspection shall be decided annually by the Executive Council on the basis of estimates provided by the Technical Secretariat in the context of setting an overall annual quota of nominations for inspections. This number shall be commensurate with the verification budget and the number of State Parties.

[(ii) A State Party may transfer some or all of its nomination quota to the Technical Secretariat.]

(iii) Nominations for inspection by States Parties shall be communicated to the Technical Secretariat before the beginning of the year for which the inspections are proposed. The Technical Secretariat shall ensure that the identity of the facilities nominated and the proponents are not revealed.

***

Optional subparagraphs (iv) and (v):

First option

(iv) To fulfil the annual quota of nominations set by the Executive Council in accordance with subparagraph (i) above, an additional number of facilities will be added to the nominations of States Parties. These will be selected on a purely random basis from facilities declared under this Annex [and not nominated by States Parties].

(v) The Technical Secretariat shall then, from the combined nominations, randomly select the facilities to be inspected in such a way as to preclude the prediction of when the inspection takes place.

Second option

(iv) Should the combined number of different national nominations be smaller than the overall annual quota set by the Executive Council in accordance with subparagraph (i) above, an additional number of facilities declared under this Annex shall be added. These shall be nominated by the Technical Secretariat [on a strictly random basis.]

(v) The Technical Secretariat shall then, from the combined nominations, randomly select the facilities to be inspected in such a way as to preclude the prediction of when the inspection takes place.

Third option

(iv) Should the combined number of different national nominations be smaller than the annually available number of inspections, all facilities nominated by States Parties shall be inspected. [In addition, the technical Secretariat shall select [on a strictly random basis], from facilities declared under this Annex, such a number as to make full use of the verification budget.]

(v) Should the combined number of national nominations be greater than the annually available number of inspections, the Technical Secretariat shall, from the combined nominations, randomly select the facilities to be inspected in such a way as to preclude the prediction of when the inspection takes place.

***

(vi) Under this Annex, a State Party is not obliged to receive a greater number of inspections per year than two plus [5 %] of the number of its declarations under this Annex.

(vii) No facility shall receive more than two inspections per year under the provisions of this Annex.

2. Inspection Aims

(a) At [plant sites] [plants] declared under this Annex, the general aim of inspections shall be to verify that activities are in accordance with obligations under the Convention and with the information provided in declarations. In particular, it shall be verified that non-declared chemicals listed on Schedules 1, 2 or 3 are not present at the [plant site] [plant] in quantities above thresholds for declarations.

[(b) Inspections of plant sites declared under this Annex shall not lead to a duplication of the inspection régime provided for plants declared under Annex 2 to Article VI. However, such plants, if located within a plant site inspected under this Annex, may be inspected according to the provisions of this Annex.]

3. Inspection Procedures

Inspections shall be carried out in accordance with agreed guidelines and other relevant provisions of the Protocol on Inspection Procedures and the Annex on the Protection of Confidential Information.

(Note: the relevant provisions of the Protocol on Inspection Procedures remain to be elaborated in detail)

CONFERENCE ON DISARMAMENT

CD/CW/WP.371
11 October 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

ARTICLE IX

.....

Procedure for challenge inspections

This Working Paper contains the outcome of the open-ended consultations conducted by the Chairman of the Ad Hoc Committee on this issue. It is intended to serve as the basis for further elaboration of the Article.

****

1. Each State Party has the right to request [the Executive Council for] an on-site challenge inspection of any facility or location in any other State Party */ for the sole purpose of clarifying and resolving any questions concerning compliance with the provisions of the Convention, and to have this inspection conducted anywhere without delay by an inspection team designated by the Director-General of the Technical Secretariat and in accordance with the Protocol on Inspection Procedures.

Each State Party is under the obligation to keep the request within the scope of the Convention and to provide in the request all appropriate information [evidence] [supporting the suspicion of non-compliance] [on the concern regarding compliance] with the Convention as specified in the Protocol on Inspection Procedures. [Each State Party shall refrain from unfounded requests, care being taken to avoid abuse.] The challenge inspection shall be carried out for the sole purpose of determining facts relating to compliance.

---

*/ It is understood that issues relating to "jurisdiction and control" in the context of challenge inspections need to be considered further.

2. For the purpose of verifying compliance with the provisions of this Convention, each State Party shall permit the Technical Secretariat to conduct on-site challenge inspections pursuant to paragraph 1.

3. Pursuant to a challenge of its facility or location, and in accordance with the procedures provided for in the Protocol on Inspection Procedures, a State Party has:

- the right and the obligation to demonstrate its compliance with the Convention and, to this end, to enable the inspection team to fulfil its mandate;
- the obligation to provide access within the requested site for the sole purpose of establishing facts relevant to the request; and,
- the right to take measures to protect sensitive installations, and to prevent disclosure of confidential information, not related to the Convention.

4. The requesting State Party [has the right to] [may, subject to the agreement of the inspected State Party,] send a representative to observe the conduct of the inspection. The inspected State Party shall [then] grant access to the observer in accordance with the Protocol on Inspection Procedures.

5. The requesting State Party shall present a request for an on-site challenge inspection to the Director-General of the Technical Secretariat. The Director-General shall [notify] [transmit the request immediately after its receipt to] the inspected State Party [not less than 12 hours prior to the planned arrival of the inspection team at the point of entry]. Contemporaneously, the members of the Executive Council [and all the other States Parties] shall be informed about the request.

6. The Director-General of the Technical Secretariat [subsequent to the decision of the Executive Council] shall issue a mandate for the conduct of the inspection. The mandate shall be the request referred to in paragraph 1 put into operational terms, and shall conform with the request.

7. The inspection shall be conducted in accordance with Part III or, in the case of alleged use, in accordance with Part IV of the Protocol on Inspection Procedures. */ The inspection team shall be guided by the principle of conducting the inspection in the least intrusive manner possible, consistent with the effective and timely accomplishment of its mission.

8. The inspected State Party shall assist the inspection team throughout the inspection and facilitate its task. If the inspected State Party proposes [,in exceptional cases,] [,pursuant to Part III, Section III.B of the Protocol on Inspection Procedures,] arrangements to demonstrate compliance with the Convention, alternative to full and comprehensive access, it shall make every [reasonable] effort, through consultations with the inspection team, to reach agreement on the modalities for establishing the facts with the aim of demonstrating its compliance. [In case of any prolonged disagreement, the Executive Council shall be immediately seized of the problem.]

9. The final report shall contain the factual findings as well as an assessment by the inspection team of the degree and nature of access and cooperation granted to the inspectors and the extent to which this enabled them to fulfil their mandate. The Director-General of the Technical Secretariat shall promptly transmit the final report of the inspection team to the requesting State Party, to the inspected State Party, to the Executive Council and to all other States Parties.**/ The Director-General shall further transmit promptly to the Executive Council the [assessment] [view(s)] of the requesting State Party, the view(s) of the inspected State Party, and the view(s) of other States Parties which may be conveyed to the Director-General for that purpose, and then provide them to all States Parties.

---

*/ A view was expressed that the provisions regarding investigations of alleged use of chemical weapons should be placed in a separate section under this Article.

**/ It was suggested that the Director-General should express his views on the matter.

10. [When requested by any State Party,] the Executive Council shall meet within 48 hours [following the presentation of the final report of the inspection team] to review [and assess] the situation [, decide on, inter alia, whether any non-compliance with the Convention or abuse of the right to challenge inspection has occurred] and consider any appropriate further action necessary to redress the situation and to ensure compliance with the Convention, including [sanctions and other] specific proposals to the Conference of the States Parties. [It may also assess whether a request was in conformity with the obligations contained in paragraph 1 to keep the request within the scope of the Convention.] At such a meeting, the requesting State Party and the inspected State Party shall have the right [are invited] to participate. The Executive Council shall inform the States Parties (Conference of the States Parties?) of the outcome of its meeting.

CONFERENCE ON DISARMAMENT

CD/CW/WP.372
11 October 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

SWITZERLAND

Report on the Second Swiss Trial Inspection

Introduction

On 6-7 November 1990, Switzerland conducted an inspection at a government-owned armaments plant in accordance with the Draft Chemical Weapons Convention (hereafter Draft Convention) of the Conference on Disarmament in Geneva. The aim of this exercise was to test the regulations for the conduct of a challenge inspection. The object of the inspection was a munitions factory which was to be examined with respect to suspected chemical weapons activities, especially those which might involve producing and stockpiling such weapons.

The trial challenge inspection clearly demonstrated that a whole series of arrangements are required on the part of both the inspectors and the challenged State in order that such inspections may be carried out efficiently and within a reasonable time span. These include the establishment of an easily transportable and effective monitoring infrastructure, as also arrangements for the reception and escort of the international inspectors by the challenged State. It must also be taken into account that, in practice, there will always be fewer inspectors available than are required to handle, in a completely satisfactory manner, the various tasks of the challenge inspection, for example the temporary securing of the perimeter of the site being verified. In many cases the inspectors will be obliged to rely on the cooperation of the challenged State.

The trial challenge inspection also showed that the regulations and procedures for the conduct of a challenge inspection listed in article IX of the Draft Convention are generally adequate. Apart from the question of the desirability of incorporating a tour of the site in the briefing of inspectors, the draft apparently contains no real gaps.

In practical terms, however, many of the measures and procedures envisaged may prove impossible to implement because of the limited time and material available. Above all, the trial challenge inspection showed that items such as securing the perimeters of inspected sites, sampling, translation of

documents provided by the plant and working out a comprehensive inspection plan could sometimes have a substantial effect on the length of the inspection and the number of inspectors required. The amount of practical cooperation which may be required of the challenged State may also be considerable. Apart from interpreters and permanent escort personnel, secretarial services and administrative and organizational support staff may have to be provided.

Interesting experience was also obtained in the matter of confidentiality, in which it appeared that procedures such as "managed access" can help to prevent the misuse of inspections. However, the amount of time required for working out and applying such procedures should not be underestimated. Due to the pressure of time, it frequently occurred that only a part of the tests and examinations contained in the inspection plan could be carried out.

Taking samples, dismantling of munitions for inspection and analysis proved to be especially time-consuming.

Preliminary remarks

Challenge inspections are single examinations carried out at short notice at the request of a contracting State by an international chemical weapons inspectorate. In contrast to the challenge inspection system laid down in the Vienna CSCE Document on Security and Confidence-Building Measures, the Draft Convention does not provide for the inspection to be carried out by the contracting State which makes the request. The latter may, however, be represented by an observer, whose freedom of movement must not be unduly limited.

In principle, challenge inspections may take place at any place and at any time. Although the current Draft Convention does not allow the challenged State to refuse a request, it includes regulations governing the inspection procedures which might help to prevent the misuse of inspections. These include the "managed access" system which was applied and tested during the trial challenge inspection.

The aim of the trial challenge inspection was to examine the practicability, completeness and appropriateness of the regulations contained in the Draft Convention. An additional indirect aim was to begin preparations for the implementation of chemical weapons challenge inspections in Switzerland and to draw the attention of the responsible authorities to the possibility of such challenge inspections.

Starting position

Assumptions

The trial challenge inspection was based on a number of concrete suspicions:

1. The suspicion that non-declared substances, as defined in Schedules I, II and III, which could be used for the production of chemical weapons, were being stockpiled in violation of the Draft Convention.

2. The suspicion that chemical weapons munitions were being manufactured, using equipment located at the site being inspected.

3. The suspicion that chemical weapons munitions were being stockpiled in violation of the Draft Convention.

The elements of suspicion were selected in such a way that it might have been assumed for the purposes of the trial challenge inspection that chemical weapons were being stockpiled or even to some extent manufactured in the sites being inspected - even though of course such a possibility did not exist. In addition, at the planning stage special efforts were made to take into account all the possible functional and operational structures of the plant being inspected. The intention here was to find out which procedures would be particularly promising for the inspection of military installations.

General aspects and remarks

Technical equipment and instruments

In selecting the measuring instruments to be used it appeared necessary to take into account the working and operational standards applied in the site being inspected. In this connection, the availability of easily transportable and effective measuring equipment which corresponded to the most severe safety standards and which could in consequence be used in all inspections would be of great advantage. In this way it would be possible to carry out analyses and other measurements and examinations rapidly and in sufficient number.

Securing the perimeter of the site

If possible the perimeter of the site being inspected should be secured before the beginning of the inspection, so as to ensure as far as possible that no removal of evidence could take place. This would mean that all entry and exit points to and from the site being inspected should be kept under control. In addition, flows of goods and transport systems within the site being inspected should be examined as to the possibility of removing evidence. Of particular importance here would be registration and control of flows of goods from buildings which might be of special importance for the inspection. The decision as to whether securing the perimeter of a site being inspected is necessary or adequate should in all cases be left to the inspectors themselves.

During the test challenge inspection it was clearly shown that the number of persons required to secure the whole perimeter of the site being inspected would in most cases exceed the resources of the international inspectorate.

In the event that securing the perimeter is considered necessary, either the site will have to be evacuated or the inspectors will have to rely on the site's own monitoring system by bringing this under their control. As a rule, military installations possess an effective and easily controlled system for securing the perimeter.

It is also possible, as was successfully established during the trial challenge inspection, to secure particularly suspect premises on an individual basis by means of measures such as sealing and permanent guarding until the necessary examinations have been carried out.

It may also be noted with respect to the practical procedure that the first measures to secure the perimeter must be taken immediately the inspectors arrive. The time spent in explaining the reasons which have led to the inspection should not provide the State being inspected with an opportunity to remove suspicious materials or to shift them around within the site. Particular attention should be given to the identification and registration of all points of access to underground structures.

During the preparations for the test challenge inspection it also became apparent that it would be helpful if the team of inspectors could include a specially trained expert for security questions. The inspection team would therefore be in a better position to resolve the logistical and organizational problems of securing the perimeter of the site being inspected immediately after its arrival.

Determining the perimeter of the site

When an inspection is announced the approximate location of the site to be inspected is generally known. In consequence, an inspector must immediately establish the perimeter of the site to be inspected as soon as he arrives at the site. It is assumed in the Draft Convention that the perimeter of the site includes all suspected buildings and structures. But in the case of the military installation inspected this did not apply. The buildings and structures which were relevant to the inspection were widely scattered. Some munitions stores were located up to 20 km from the munitions factory. In planning the trial challenge inspection, it was necessary to clarify the reasons for this large distance between factory and store:

- Operational safety

  Dangerous substances and highly explosive munitions are normally stored in buildings separated from each other by a minimum distance.

- Confidentiality

  Technically sophisticated and consequently valuable munitions are normally kept in high security buildings

- Problems of available space

  Available space problems which arise from the fact that there has to be a minimum distance between munitions manufacturing and storage sites are often solved by distributing storage, manufacturing and administrative buildings amongst a number of different sites.

In these circumstances, a purely geographical determination of the assumed site to be inspected would either lead to a much too narrow a definition of a site which is in fact scattered over a number of different

locations or to a much too wide a definition which would include installations belonging to third parties which have nothing to do with the site in question. This means that in every case functional as well as geographical criteria must be taken into account when determining the perimeter of the site. So no special importance needs to be attached to the fact that the sites identified are not located in a single geographical area.

Time-frame of the inspection

In principle the inspectors are free to determine the amount of time they require for a challenge inspection. However, in spite of the fact that there is no upper time limit, it may be assumed that in most cases a challenge inspection will not take more than two or three days. The trial challenge inspection was based on the assumption that the inspectors would be available for two days.

But in order that such a demanding and many-sided task could be completed within the space of a few days, it became apparent during the preparatory stage that in most cases very well-trained and well-equipped inspectors would be required. Apart from a high level of general experience, these would wherever possible require special knowledge in the general subjects of manufacture and storage of chemical weapons, as well as manufacture of conventional munitions.

Photographic and film equipment

In the trial challenge inspection it was assumed that the inspectors would be able to photograph and film suspect objects. It was known in advance, however, that, in principle, photography is prohibited in a military installation or is at least subject to rules of confidentiality, and that in certain circumstances this prohibition makes it difficult to use photography to provide evidence.

As with measuring instruments, video and photographic equipment must be specially approved for use in areas where there is an explosion danger. In consequence, preference should be given to mechnical cameras with highly sensitive films which can be used without flash.

Escort of inspectors

The Draft Convention envisages continuous in-country escort of inspectors. During the trial challenge inspection it was assumed that one person would be available for the escort of each inspector at any time. It is true that this presents the State being inspected with an organizational problem, but it also ensures that each individual inspector is given the greatest possible freedom of action. In the preparatory stage the question also arose whether the escorts of the inspectors had a part to play during the inspection itself or whether they should remain apart from the inspectors during their actual work.

Definitions

The Draft Convention includes a series of definitions to be used for the conduct of challenge inspections. On the basis of the practical results of the trial challenge inspection the question arose as to whether these definitions are in fact usable. The following conclusions were reached:

- The function of an assistant during an inspection must be clearly defined and must in all cases be limited to an auxiliary role. The definition contained in the Draft Convention is too vague.

- It would also be useful if the definition applied to the time necessary for an inspection should include the time required for an initial tour of the site.

Conduct of the inspection

Notification

According to the Draft Convention the State which requests the challenge inspection in another contracting State may itself decide whether to include in its request detailed information of its suspicions for the use of the international inspectorate. The Draft Convention also includes a provision that it is only when the inspectors arrive at the point of entry that they will inform the State challenged of the identity of the site to be inspected.

From the point of view of the challenged State it is certainly desirable that precise details about the site to be inspected and about the various elements of suspicion should be notified at the earliest possible moment, so that organizational and logistical preparations may be made for the reception of the inspectors and the conduct of the inspection. However, if the notification is made too early, an opportunity might in some circumstances be given to the challenged State to cease suspect activities at the site to be inspected and to cover up all traces of them. In order to prevent this happening, precise specification of the suspect elements should be left to as late a stage as possible to maintain a surprise effect.

During the trial challenge inspection there was no such surprise effect, since all those participating knew in which site the inspection was to take place. However, even in the absence of an actual trial of the initial phase of the challenge inspection it may be stated that a series of executive measures at the national level will always have to be taken in advance in order that the challenge inspection may be conducted without hindrance. Such measures should include:

- the training of specific expert personnel to escort the international inspectors throughout the period of the inspection;

- the setting up of a national control authority to ensure that the international inspectors are guaranteed the maximum possible freedom of action during the whole period of the inspection;

- simplified entry and exit formalities for the international inspectors and all measuring instruments which they may wish to bring with them.

Briefing

The inspection itself begins with a briefing in which the inspectors are provided with all necessary information by the challenged State about the site to be inspected. The Draft Convention provides a maximum of three hours for this briefing - which is not included in the time allowed for the actual inspection - as well as for the establishment of the inspection plan.

The primary purpose of the briefing in the case of a challenge inspection is to provide the inspectors with a general overview of the structure and operational organization of the site to be inspected. The trial challenge inspection showed clearly that the inspectors attach great value to this general briefing, since it is in this way that they are able to obtain a detailed idea of the structure of the site to be inspected.

During the trial challenge inspection the briefing was also used by the inspectors to provide detailed information about the suspect buildings and structures, followed by a comprehensive tour of the site established on the basis of these details. In view of the importance of the briefing and the tour the three hours provided for these purposes in the Draft Convention hardly seem sufficient.

During the trial challenge inspection the briefing was also used to provide the inspectors with documentary information about the operations, the buildings, the product groups and all ancillary activities of the site in question. It was also established that apart from a general overview of the buildings on the site documents and layouts on energy supply pipes and waste disposal systems are useful to the inspectors.

During the trial challenge inspection it was established that it would be useful if the following documentary information could be provided by the management of the site in question at the time of the briefing:

- a general plan of the site to be inspected;

- documents on the range of goods produced;

- documents on the management structure.

There is likely to be a basic language problem during the briefing. In normal circumstances documents will be available only in the language of the challenged State. And it may be assumed that it will probably not be possible to obtain an interpreter who will be in a position to provide an overview of the documents made available. In addition, during the trial challenge inspection the following points were established:

(a) To save time, only information directly relating to the inspection should be provided. This comprises information about the type of buildings and equipment, intermediate and end-products manufactured, security, administration and logistics (movement of goods, storage, etc.);

(b) For reasons of security and confidentiality it must be clearly stated which documents the inspectors may retain and which they must hand back at the end of the inspection. A check-list should be established to this end.

Inspection plan

The inspection plan establishes the programme of the inspectors. During the trial challenge inspection the inspection plan formed the basis on which the whole inspection was conducted. In the inspection plan it is particularly important that all fields of operation which may entail problems of security or confidentiality should be identified. Such fields include:

- arrangement, size, availabilty of energy equipment (distribution, manufacture, consumption);
- safety installations and special protective equipment for life-saving operations;
- special work instructions and testing regulations;
- flow charts and site plans.

During the trial challenge inspection the inspection plan also included elements designed to verify certain of the information provided by the challenged State regarding the functional structure of the site. In such cases inspection of the exterior of the building in question or a spot-check on the operations in question was sufficient.

If possible the inspection plan should also contain the intended course of the inspection, the order in which equipment is to be tested, as well as the length of time which is likely to be required for the inspection of individual installations. In this way the management of the plant will be better able to organize the visit, and interference in operational and manufacturing processes will be kept to a minimum.

It also became apparent that working out the inspection plan took a great deal of time, particularly in cases where alternative procedures had to be worked out.

Managed access

The term "managed access" refers to an agreement to be established between the inspectors and those responsible for the site to be inspected. This agreement should contain detailed provisions regarding the procedure of the inspectors and the achievement of the goals of the inspection.

Managed access is, in general, applicable in cases where two sets of problems arise. The first is during the inspection of equipment which, after closer investigation, turns out to be irrelevant to the suspect element in question. The second is where particularly sensitive buildings or equipment are involved. In such cases, managed access provides an alternative procedure

which takes into account the need for confidentiality regarding the site in question but which nevertheless permits the suspect elements to be verified with a high degree of certainty.

Protection of confidentiality covers matters such as the technology applied, organization, location, structure and overall content of the site in question. A basic difference should be made between military and civilian sites. Military sites are subject to a particularly high degree of confidentiality with respect to the following:

- location;

- the inner structure;

- the content, employment and purpose.

An exception to this general rule would be military production sites which can be considered as entirely equivalent to civilian plant sites of a similar nature with respect to confidentiality.

In general terms, military sites can be verified in the same way as civilian chemical plant sites. However, in the case of some types of military sites, particularly munitions stores, special inspection procedures centering on gathering circumstantial evidence must be worked out. But during the trial challenge inspection it was established that it is impossible to clarify a suspect element entirely or to provide final proof of prohibited activities by the use of circumstantial evidence alone, particularly in cases where the confidentiality of such evidence has to be provided with appropriate protection. Consequently, the system of managed access is applied specifically in cases where military confidentiality is required. In this connection it was established that the necessity of protecting confidentiality was least troublesome with respect to the location of buildings and specific installations. Indeed, in many cases, knowledge of the location of the buildings and specific installations of the site to be inspected was sufficient for an assessment to be made of both their specific military use and their structure. In almost all such cases inspection of the exterior of buildings enabled the inspectors to eliminate to their satisfaction the suspicion with respect to the manufacture and stockpiling of chemical weapons.

During the trial challenge inspection situations sometimes arose in which an inspection of the exterior of a building was not sufficient. The procedure established for such cases was as follows:

With respect to particularly sensitive aspects of the site being inspected (e.g. plans, store inventories, diagrams of installations, alarm and security dispositions) a step-by-step procedure was worked out, and as soon as the required result was attained those responsible for the site were informed that no further inspection would be necessary. In one individual case the inspectors did not require the production of documents and were satisfied with a very brief inspection.

Precisely in a country such as Switzerland, whose military infrastructure is mainly based on permanent installations, the issue of military confidentiality is of particular importance. Disclosure of the structure, plans and capacity of military installations would have far-reaching consequences and would certainly reduce the battle-readiness of the country's militia. In consequence, the suspect elements notified for inspection could be dealt with only by means of an exterior inspection of the various buildings and structures to be inspected and of the security dispositions available.

Collection of samples

The Draft Convention expressly permits the taking of samples. However, the trial challenge inspection showed that the problem of designating or sealing samples and other pieces of evidence requires more study and that systems worked out must be practiced in advance (X-ray and sampling procedures, etc.). Taking samples and making measurements require a great deal of time, and the site in question must cease operations while this is being done. (Stores may have to be sealed for several hours while measurements are taken). In the same way chemical containers and lists featuring trade names can make verification more difficult and may even be used to deceive the inspectors.

X-ray procedures

At a site in which, in normal circumstances, chemical weapons are neither manufactured nor destroyed, it is clear that no equipment for dismantling chemical munitions will be available. In consequence, alternative procedures must be found for the verification of suspect munitions. One possibility would be through X-ray treatment of any suspect munitions. By photographing a munition at a certain angle, for example, it is possible to determine from the X-ray picture whether it contains liquid, which would indicate the presence of chemical weapon substances. Any possibility of confusion in the X-ray procedure or a secret manipulation of X-ray pictures can be prevented by attaching special markers to those projectiles which are to be X-rayed. In normal circumstances X-ray equipment is available in munitions factories for quality control purposes. Since this procedure calls neither for dismantlement of the munition nor for chemical analysis of the contents of the munition, the principle of confidentiality is in large measure guaranteed.

Off-site analyses

In every inspection involving the taking of samples, a bottleneck arises with respect to the capacity for rapid analysis of any laboratory which may be available. According to the type of sample involved it may take up to one day per sample for the analysis and interpretation procedure to be completed. On average it may be assumed that six to eight different samples can be processed within the space of two days. Possibilities of confusion may be eliminated by the use of different types and sizes of container for different types of sample or by using more comprehensive markings. Also, wherever possible the samples should be split into at least four parts. Two samples are absolutely necessary for effective analysis, while one each should be kept for reference

and reserve. In the long term it will be necessary to standardize all procedures for taking, processing, splitting and securing all samples according to accepted methods.

During the trial challenge inspection it became apparent that problems involved in the taking of samples are for the moment largely unsolved. The Draft Convention does not contain detailed inspection procedures in this respect. This means that decisions regarding the taking of samples at present depend only on the personal assessment of individual inspectors. It would be desirable, however, that the main purpose of taking samples should be seen as part of a specific clarification procedure with respect to suspect elements. A very carefully considered and fairly restrictive decision-making process on sampling should be worked out, since both the actual taking of samples and the consequent analysis of unknown substances are extremely time-consuming.

With respect to photography during inspections, the trial challenge inspection made it apparent that polaroid cameras, together with still-video and other video equipment, make up the most appropriate instruments. Their advantage lies in the fact that the results can be inspected immediately. In the case of electronic equipment the question of duplicates arises. If necessary two exposures with the same setting must be taken one after another.

In order to safeguard the interests of the site in question, photographs will be taken only after discussions with those responsible for the equipment being inspected. The latter will have the right, in general terms, to propose the angle from which the photographs are to be taken and the precise items which are to be photographed, and they will also have the right to cover pre-determined parts of the site being photographed.

During the trial challenge inspection, various key operations were photographed. The procedure was as follows:

1. Two consecutive photographs were taken of each key operation with a polaroid camera.

2. Representatives of the challenged State and of the plant management then examined the photographs as to whether they contain relevant information and whether they contained a danger in terms of confidentiality. This meant that the photographs had to be submitted to the site management for approval.

3. The challenged State was given the right either to cover or to exclude from the photographs sensitive parts of the site which were not deemed relevant to the inspection. This was done through determining the angle from which the photographs were to be taken.

Questioning of personnel

The Draft Convention explicitly permits the questioning of site personnel. One of the problems so far unresolved refers to the questioning of site employees who are bound to secrecy. In any case, all employees must be informed about the reason and goal of the inspection before they are subject to questioning by the international inspectors. In consequence, the

inspectors may conduct interviews and interrogations only after discussions with the challenged State and after the employees in question have been fully informed.

Composition of the inspection team

The inspection team should include at least the following persons:

- at least five inspectors. The exact number of inspectors should be determined according to the size and complexity of the site to be inspected;

- one to two translators and possibly support staff (it will hardly ever be possible to rely on local writing aides and secretaries);

- as a general rule inspectors should always work in pairs.

In some circumstances it may be helpful to give the chief inspector a free hand in composing his subordinate teams in order to allow him the possibility of coordinating them as and how he wishes and of personally supervising areas of verification which he considers particularly critical. During the trial challenge inspection communication between the subordinate teams took place through the facility's own internal radio system. In addition, the inspectors were able to dispose of an office in the management area containing a telephone which they could use to make calls abroad.

Results

The trial challenge inspection demonstrated that the regulations and procedures for inspections contained in Article IX of the Draft Convention are quite sufficient. Apart from the desirability of incorporating a tour of the site in the briefing of inspectors by those responsible for the site, the Draft Convention contains no important gaps in this respect.

However, from the practical point of view many of the measures and proceedings envisaged may prove very difficult to implement because of the limited amount of time and material available. Above all, the trial challenge inspection showed that items such as securing the perimeter of the suspect site, sampling, translating documents provided by the plant and working out a comprehensive inspection plan could sometimes have a substantial effect on the length of the inspection and the number of inspectors required. The amount of time which has to be spent by the inspectors on questions of organization is substantial. Apart from interpreters and permanent escort personnel, secretarial services and administrative and organizational support staff may have to be provided by the challenged State.

Interesting experience was also obtained in the matter of confidentiality. In most cases it was possible for operational confidentiality to be combined with the requirements of the inspection. With respect to military confidentiality the managed access system proved sufficient in the trial challenge inspection, even though this increased the pressure on the inspectors and the inspection consequently lasted longer.

During the instrumental phase of the inspection it became apparent that the analysis and checking equipment used (video and other cameras) must always be seen to conform to the operational safety regulations before they can be used in the site in question without danger. In addition, however, there proved to be no difficulty in using testing and analysis equipment (X-ray equipment, etc.) available at the site for the requirements of the inspection, as long as the inspectors themselves possessed sufficient technical knowledge to operate them.

In order to conduct a complete and detailed challenge inspection, at least twice as much time would be required as was available during the trial inspection (particularly for working out the inspection plan, sampling, measuring, etc.). In a number of the buildings and structures to be examined, it was in fact impossible to carry out all the checks planned because of lack of time. It should also be noted that sampling, dismantling of munitions for inspection and analyses were particularly time-consuming. It also sometimes happened that inspection plans had to be changed suddenly due to unexpected incidents. A special problem turned out to be the extra time required for obtaining access to military sites. For reasons of security and confidentiality various difficult steps or phases, such as informing the responsible site or plant personnel and securing the perimeter, could not be left out. Obtaining sufficient trace evidence of chemical weapons substances is also time-consuming. In this connection the main problem to arise was whether much more would actually be achieved even if more time and more sophisticated material were available. One of the main aspects which should be examined with respect to extra time required is what would in fact be the value added provided by such an increase.

APPENDIX I

INSPECTION RESULTS

Object	Activity	Number of personnel	Allotted time	Begin	Observation
site 452	viewing	2	1/4 h	0800	use as declared, production or stockpiling of CW possible
building 890	viewing	2	1/2 h	0800	use as declared, intermediate stockpiling
building 783	viewing	2	1/2 h	0800	use as declared
Annex building munition assembly 785	viewing	4	1/2 h	1430	function as declared
laboratory L_1 4640	viewing (all rooms and foundations)	2	3/4 h	0900	underground building connections and overall situation, facility clearly serves production and stockpiling of munition no evidence of CW
munition warehouses 4616/4606	control of entries, viewing	4	1 h	1500	no deviation of random sample, no CW control?
chemical stocks	viewing, air sampling, overall view of stocks	2	1 h	0800	no suspect chemicals, safety installations would allow for stock-final results after analysis of samples
explosives foundry	viewing, detailed examination of detail	4	4 h	1330	use for CW only under extreme conditions (high risk) and corresponding adaptation of the facility, without considering the pending analyses, no evidence of past use for CW

APPENDIX I

INSPECTION RESULTS (continued)

Object	Activity	Number of personnel	Allotted time	Begin	Observation
phosphorus foundry 797	air tester sampling as needed	2	1/2 h	1130	clearly used for pouring phosphorus air sample indicates slight risk, probably false due to Phosphin
stores/ inventory	examination of sulphur content, search for CW precursors	4	1/2 h	1100	interference with production program, no objections, no CW-relevant amounts of sulphur
underground munition facilities	control, stockpiling (access to all rooms and spaces)	2	1 1/2 h	1300	only conventional stockpiling (issues of concern) dismantled munition clearly pointed/ explosive grenade
examination of grenade	X-ray, dismantling, possibly further examination	4	2 h	1000	explosive grenade, X-ray gave no indication of CW

APPENDIX II

SUGGESTED "CONTROL SHEET FOR INSPECTION SITE"

Identification

Classification No.

Building No.

Description

Official Designation of Use

Buildings

Facilities

Further Possible Use

Buildings

Facilities

Inspection Experience/Relevant Points

1

2

3

4

Assessment

APPENDIX III

PROVISIONAL INSPECTION REPORT

Inspection commission:

Time:

Area:

Inspection Plan

1. Viewing of site 452
2. Viewing of building 840
3. Viewing of building 783
4. Viewing of munition assembly 785/798
5. Viewing of all rooms and examination of use of L_1 including underground connections
6. Control of entries
7. Viewing of all storage rooms for chemicals, sampling
8. Explosives foundry, detailed viewing and clarification enquiry into alternate use, waste water and air-expelling devices, sampling
9. Phosphorus foundry 797, viewing, examination of waste air and waste water, sampling, deployment of CAM AP2C
10. Control of facility inventories for chemicals
11. Underground munition stores, control of content, access to all rooms, random sampling of a grenade
12. Examination of grenades, X-ray, delaboration, possibly further examination

APPENDIX III

PROVISIONAL INSPECTION REPORT (continued)

Results

Provided the analyses prove to be negative, the situation is as follows:

1. No relevant amounts of CW-precursors.

2. Provisional charging of munition with CW agents is only just conceivable at the explosives foundry but would be highly dangerous and entail corresponding adaptive measures.

3. Clearly the underground stores contain only conventional munition.

Addendum

List of samples

List of documents

List of accessories

APPENDIX III

Addendum 2

List of Samples

Designation	Type, Quantity	Sampling Site
11 12 13 14	air samples in XAD-2-tubes, each approx. 40 ml	building 4646, chemical stock #2, room on basement floor - only hermetically sealed, ventilated room
a_1 a_2 a_3 a_4	liquid chemical "soltene" direct from 800 l tank, approx. 2 ml each	building 4646, chemical stock #2, room on 1.basement floor
x_a x_b x_c x_d	(waste) water samples from surface treatment	building 4646, (former) phosphorus stock #3 vertical tank A_5 (10 m)
a_a a_b a_c a_d	sludge samples, approx. 20-50 g each, settling basin of waste water purification plant	adjacent to building 4630, (explosives foundry)
m_a m_b m_c m_d	ceiling coating with residues approx. 0.1-0.5 g taken from above the filling station	building 4630, (explosives foundry)

APPENDIX III

Addendum 2 (continued)

Documents

4 X-ray exposures R_1-R_4 *

5 Polaroid photographs A_1-A_4 *

List of respirator material

Alarm scheme for facility fire brigade

Extracts from munitions facility handbook

1-311 protective shoes

1-314 protective glasses

1-110 procedure in case of accidents

1-111 accidents one is obliged to report

Computer inventory of protective and working clothes (SMFA80-03)

Plan of building explosives foundry

Computer inventory of chemicals 7 November 1990 0641 h

Control print-out of article group # 03 000 000 - 03 050 200 1115 h

4 overall view maps of area of munitions facility

4 brochures munitions facility *

Inventory of stocks, buildings 406 and 416

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* May be retained by inspectors.

CONFERENCE ON DISARMAMENT

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Ad Hoc Committee on Chemical Weapons

UNITED KINGDOM OF GREAT BRITAIN AND NORTHERN IRELAND

Destruction of CW stocks, weapons and associated plant

(for the meeting on Technical Aspects of CW Destruction
7-11 October 1991)

1. OVERVIEW

1.1 The destruction of CW stocks, CW filled weapons and the plant associated with the production, bulk storage and weapon filling of CW materials, is a major task. The difficulties must be recognized of carrying out such a complex and hazardous operation, on any significant scale, in a safe and environmentally acceptable manner. It must also be recognized that whilst there is a clear need to protect the environment, the safety of those directly involved in the operation must be paramount. In this paper the general principles involved in such an operation are discussed.

1.2 Countries which have undertaken, or are attempting to undertake, the destruction of CW materials and facilities have found that it is first necessary to categorize the materials for disposal (e.g. bulk agents, agent filled shells, agent filled rockets, contaminated plant, etc.) and then develop procedures on a case-by-case basis. For example, whilst the overall policy may be to go for incineration as the primary means of destruction, the actual methodology to be used for each category of material will need to be determined individually.

2. DISPOSAL METHODS

2.1 Some of the methods available for the disposal of these materials, and their respective advantages and disadvantages, are reviewed in the following paragraphs.

2.2 Land burial

2.2.1 This is a quick, cheap solution; particularly for lightly contaminated items of plant and equipment. The risk of future problems can, if deemed necessary, be significantly reduced by first encasing the materials in a non-leachable package such as foamed concrete contained within an outer metal

container. Whilst land burial is less likely to be acceptable to the environmental lobby, it is nevertheless the current commercial practice in many countries for dealing with hazardous materials of a similar nature.

2.3 **Sea disposal**

2.3.1 The disposal of CW materials and weapons filled with these materials in deep ocean trenches was the solution adopted at the end of World War II. Problems with this technique are only known to have arisen where dumping was carried out in relatively shallow waters such as the Baltic. Dumps located at depths well below that at which fishing operations are routinely undertaken appear to pose few problems, if any.

2.3.2 However, the Oslo and London Dumping Conventions, to which the United Kingdom is a signatory, require such operations to cease by the end of 1992. Therefore, while sea dumping might well be the quickest and safest method for the disposal of certain CW munitions (especially those containing an explosive element) it is unlikely in the future to be seen as the preferred option.

2.4 **Open burning**

2.4.1 Open pit or tray burning has been used extensively in the past for the disposal of mustard. However, this results in a considerable release into the atmosphere of acid combustion products together with quantities of partial combustion products. This method of disposal therefore is generally no longer acceptable in countries which have environmental protection legislation. The major exception being where, owing to the hazardous condition of a chemical munition, open air explosive demolition/burning is considered the only safe disposal option.

2.5 **Chemical degradation**

2.5.1 The toxicity of most CW agents can either be eliminated or reduced to an insignificant level by chemical treatment. GB, for example, is rapidly hydrolyzed by treatment with a 10 per cent to 20 per cent aqueous solution of sodium or potassium hydroxide. All the nerve agents of current interest can be toxicologically neutralized in this manner. In the case of GD and GF however the hydrolysis rate is markedly slower, owing to lower solubilities. Thus United Kingdom practice, when destroying these agents, is to add ethanol to the decontaminant to overcome this problem. The major disadvantage of alkaline hydrolysis is the large volume of hydrolysate produced.

2.5.2 The USSR has developed procedures for the hydrolysis of nerve agents using monoethanolamine. The principal advantage of this method is that the hydrolysis products are largely organic in nature and therefore more suitable for treatment by incineration, if further degradation of the material (i.e. to rupture the carbon-phosphorus bond present in the hydrolysis product) is required (cf. para. 2.5.6).

2.5.3 Mustard can also be hydrolyzed satisfactorily using aqueous/alcoholic solutions of sodium or potassium hydroxide. A 10-20 per cent chlorine bleach solution is known to be equally effective.

2.5.4 Once an agent has been hydrolyzed, the question of disposal of the hydrolysate arises. Three possible solutions to this problem are discussed in the following paragraphs.

2.5.5 Sea disposal

Alkaline hydrolysis has been used in the United Kingdom for disposing of stocks of nerve agents. Each batch of hydrolysate was checked to confirm that no anticholinesterase activity remained. Batches were then diluted to reduce the fluoride ion concentration to below the required discharge limit and then discharged to the ocean via the normal effluent outfall. Whether or not this remains an acceptable solution is likely to depend upon the geographical location of the disposal site.

2.5.6 Incineration

The organic content of the hydrolysate can be further degraded by high temperature incineration. However, where sodium or potassium hydroxide has been used to effect the hydrolysis, the very low calorific value and high salt content of the product makes this a demanding and expensive solution. It also leaves the problem of disposing of the inorganic salts recovered from the incinerators effluent gas cleaning plant. Our view is that the potential hazard from the products of alkaline hydrolysis of nerve agents is low and further treatment by incineration is therefore unnecessary.

2.5.7 Concentration followed by land burial

In this approach the hydrolysate is first placed in open air evaporation pans and the liquid phase allowed to evaporate. The residual salts are then collected and buried in a properly constructed secure landfill (i.e. designed to contain any leachate 1/). In countries with high ambient temperatures this should be an attractive option. It is safe, cheap, energy efficient and, when carried out correctly, environmentally acceptable.

2.6 Incineration

2.6.1 The majority of CW agents are flammable (the notable exception being GB) and have reasonable calorific values. They lend themselves therefore to destruction by incineration. Even GB, although non-flammable, can be readily destroyed by this method. It has been demonstrated that in properly designed and operated plants very high destruction removal efficiencies can be obtained (99.99999 per cent removal) for these materials.

2.6.2 Direct, high temperature incineration was the process adopted for the disposal of the United Kingdom's remaining stocks of mustard. Small amounts of material recovered from old munitions, which continue to be occasionally found in the United Kingdom during construction and excavation work, are still processed by this method in the CBDE Porton hazardous waste incineration plant.

1/ Groundwater contaminated with water soluble products extracted from the buried waste.

No problems have been experienced with this process and the procedure complies with both existing and pending United Kingdom environmental legislation.

2.6.3 Whilst direct high temperature incineration is an equally effective method of destroying G agents, their higher vapour pressure and greater inhalation toxicity makes the operation more difficult. The problem is not in the actual incineration step, but rather in the handling and feeding of the waste to the furnaces and the protection of the operating staff. Even the best commercial hazardous waste incinerators provide only a limited degree of containment. They are simply not designed to cope with materials of this level of toxicity.

2.6.4 The capability to design incineration plants with the necessary level of containment for the safe handling of these materials is very limited. Practical experience in this area resides primarily within the United States of America. Plants of this type, by their very nature, require advanced and complex technology and as a result are extremely expensive. The alternative approach is to hydrolyze the G agent prior to incineration, thus reducing the need for such high levels of containment at the incineration step. However, as argued previously (para. 2.5.6), once the G agent has been hydrolyzed incineration may no longer be the optimum disposal solution.

2.6.5 Incineration plants of similar design to those at the Chemical and Biological Defence Establishment in the United Kingdom and the Defence Research Establishment Suffield in Canada have demonstrated that they can readily cope with mustard and mustard contaminated components. However, they do not have the level of containment necessary to deal with untreated nerve agents. Both these plants, although purpose built, are constructed from commercially available components. The provision of a plant or plants of this type should therefore not pose any major technical problems.

3. ENVIRONMENTAL ASPECTS

3.1 Whichever disposal method or methods are adopted, the aim must be to achieve appropriate high standards of safety and environmental acceptability. However, it must be remembered that these two requirements cannot be considered in isolation. The safest and most expedient disposal method may not necessarily be the most environmentally friendly. Whilst there is a clear need to protect the environment, the safety of the staff directly involved in the operation must be paramount.

3.2 In difficult and hazardous operations such as these it is essential that external parameters, such as environmental emission standards, are set at realistic and scientifically justifiable levels. Apart from making the operation both more expensive and protracted, unjustifiably high standards can actually increase the immediate risk to the personnel undertaking the work.

3.3 It will therefore be essential to decide what environmental standards are required before any final decision on disposal methods can be made. It should be recognized that environmental standards vary widely between different countries. For example, under United States environmental legislation CW agents are proscribed substances and their emission to the environment is effectively forbidden. Meeting this requirement, along with the problems

associated with transporting CW materials through States and across State lines, has been an overriding factor in the design of United States CW destruction facilities and has probably, to a large extent, determined the disposal options open to them.

3.4 The EEC approach is somewhat different. Although standards still vary between individual member States, the BATNEEC (Best Available Techniques Not Entailing Excessive Cost) and the BPEO (Best Practical Environmental Option) principles are the foundation on which environmental standards within the Community are being based.

3.5 This is the approach which has been adopted under the United Kingdom's new Environmental Protection Act (EPA). Whilst the EPA regulations set target limits for the more common pollutants (see table 1) operators of plants producing these substances are required to employ the BATNEEC principle; this means that if, in the Governments Pollution Inspectorates view, lower limits could be achieved without entailing excessive cost, they will be applied and must be met. As with similar United Kingdom legislation the question of what is and is not excessive cost will, where necessary, be determined in the law courts. Operators will also have to consider the impact of their process across the complete environmental spectrum (e.g. air, water, land, etc.) and apply the BPEO principle to determine the best overall environmental option.

Table 1 - Typical United Kingdom emission limits to the air for pollutants from high temperature incineration plants

Pollutant	Emission limit (mg/m^3)
Particulate matter	50
Heavy metals (total)	5
Sulphur dioxide	300
Hydrogen chloride	100
Hydrogen fluoride	5
Phosphorus compounds (Calculated as phosphorus)	10
Carbon monoxide	100
Total acidity	750
Organic compounds (excluding particulate matter)	20
Total free halogen	5

3.6 Under current United Kingdom legislation the release of CW materials and their degradation products is not specifically proscribed. As for other similar hazardous materials the regulations require that their potential human and environmental impact be identified, the risk assessed and appropriate action taken to reduce this risk to the minimum practicable level. Again what is practicable will, if necessary, be determined in a court of law.

3.7 In our view the EEC philosophy of BATNEEC and BPEO is a realistic and defendable approach to the setting of environmental standards for CW disposal operations.

4. DISPOSAL OPERATIONS

4.1 Once it has been decided which disposal methods are acceptable on safety and environmental grounds it will be necessary to determine the most appropriate destruction procedure for each category of CW material (eg. Bulk agent, filled munitions, contaminated plant and equipment and potentially contaminated plant and equipment). The major problems are not likely to arise with the final destruction step, but with the transport and preparation of the materials for destruction. These problems are likely to differ in severity for each of the different categories of material.

4.2 Bulk CW agent

4.2.1 If it is decided to destroy bulk stocks of agents at their current locations then the problems associated with their destruction, although by no means insignificant, are likely to be of a minor nature compared to those associated with the destruction of the CW munitions and CW production facilities. The principal factor in this situation will be providing the appropriate level of containment and protection for the operators undertaking the work. Procedures for doing this are well established and those countries which have produced these materials should have the necessary expertise.

4.2.2 However, if it is decided to move bulk stocks to a central location for destruction then the major problem will be one of safe transportation. It will be necessary, before undertaking such an operation, to agree and acquire appropriate packaging, determine acceptable routes and draw up emergency procedures to deal with any incidents that might arise.

4.3 CW munitions

4.3.1 CW munitions without an explosive or propellant component can be treated in a similar manner to bulk agent storage containers and therefore do not pose any significant additional problem. The situation with CW munitions which contain explosives or propellants on the other hand is entirely different. Destruction of these items in a safe manner poses major problems, particularly where the available disposal options are constrained by environmental considerations.

4.3.2 The historical solution to this problem has been to deep sea dump. However, if this option is not available then there are really only two basic approaches which can be used:

(a) The explosive component must be separated from the CW component and then each element destroyed by an appropriate safe method.

(b) A process capable of safely handling the combined hazard must be developed.

4.3.3 The United Kingdom, for the disposal of the occasional old munition, has adopted approach (a). Munitions are placed in a specially designed chemical containment facility, either drilled or cut open remotely, and the CW content then removed by personnel wearing full individual protective equipment. Recovered CW material is either treated chemically or incinerated in a high integrity incineration plant designed for the purpose. The explosive component is destroyed by open pit burning and the decontaminated metal components buried in a secure landfill.

4.3.4 The technique is safe, relatively cheap and no major problems have been experienced with it. However, it is slow and the disposal rate is fairly low. Where large numbers of similar munitions are involved the throughput could be greatly improved by the use of automation. Whilst the United Kingdom process has been designed in such a manner that an accidental explosion would not present any risk to the staff involved, an event of this nature would result in the release of a small quantity of CW material to the atmosphere. Appropriate safety distances and decontamination procedures are therefore essential.

4.3.5 Approach (b) has been followed in the cryogenic fracture process currently under consideration in the United States. In this process the complete munition is cooled in liquid nitrogen, to make it brittle, and then mechanically fractured into small pieces. These pieces are in turn fed to a high integrity incineration plant. The system has been designed to provide complete chemical and explosive containment.

4.3.6 Plants of this type require sophisticated technology and engineering. They involve a lengthy design, construction and commissioning period and are likely to be extremely expensive. However, they should provide a relatively high throughput and a high degree of safety and environmental protection.

4.4 CW production plant, filling and storage facilities

4.4.1 Once the bulk of the agent has been removed the problem of decontaminating the associated plant and equipment must be tackled. In order to do this safely and efficiently it is essential to have a detailed knowledge of the plant. An account of the dismantling of the United Kingdom nerve agent pilot plant is given in CD/856, 11 August 1988.

4.4.2 In a typical decontamination operation the first step would be to drain the facility down and remove as much of the CW agent as practicable. At this point it is essential that any potential points where liquid might be trapped or held up are identified and where possible dealt with. The plant would then be sealed, and completely filled with a 10-20% sodium hydroxide solution and left to stand for a lengthy period (ideally several weeks). On removal of the sodium hydroxide solution would be checked for the presence of any anticholinesterase activity and disposed of accordingly. The plant, although now free of gross contamination, may still contain traces of agent. Experience has shown that gaskets, seals and glands for example are still likely to be contaminated with agent.

4.4.3 It will therefore be necessary to dismantle the plant, starting at the top and gradually working down, piece by piece. All joints will need to be

broken, gaskets removed and potentially contaminated surfaces exposed. Where practicable the individual plant items would be submerged in decontaminant for at least 24 hours. The decontaminated components could then be buried in a secure landfill.

5. LOCATION OF DISPOSAL OPERATIONS

5.1 A decision will need to be made on whether to treat the CW materials at their individual locations or move them to one or two centrally located destruction facilities.

The optimum solution will depend on a number of factors:

(a) The location and condition of the respective CW sites and the services available at them.

(b) The difficulties associated with moving the materials safely across country.

(c) The destruction method selected. (Incineration plants for example are much more difficult to move from site to site than chemical treatment facilities.)

5.2 Whilst truly portable incineration plants are technically feasible, and a small number of units are known to exist in the United States of America and some other countries, they are not generally designed to handle materials of this nature.

5.3 Size and throughput are the major limiting factors with respect to portable incineration facilities. The size of major components, such as furnaces and gas cleaning systems, increases rapidly with increased throughput. Plants with a feed capacity in excess of 200 Kg to 300 Kg per hour of material are therefore not generally regarded as being readily portable.

5.4 All incineration plants need careful location, require ready access to significant amounts of power, fuel and water (if wet scrubbers are used) and need a high level of maintenance support. The situation with high integrity incineration plants suitable for handling CW materials is even more complicated and for these plants a fixed, carefully selected location, is likely to prove the safest and most effective solution.

6. SAFETY

6.1 As stated throughout this paper the task of destroying these materials and their associated production and storage facilities is going to be both difficult and hazardous. Whilst it may be possible to apply certain general principles to the overall programme, the actual destruction of each category of CW material and its associated facility will need to be tackled on a case by case basis. It will be essential to obtain detailed information about each facility before planning how its destruction might be achieved. In order to

keep the risk to the health of the destruction teams to a minimum the highest safety standards will be required. Safety will need to be built into each phase of the operation.

6.2 In the United Kingdom, work with hazardous substances of this nature is governed by the Control of Substances Hazardous to Health Regulations (COSHH). These regulations set out the safety principles which must be applied in order to protect the health of the people involved. They require that:

(a) The hazard to health of each substance be identified;

(b) An assessment of the risk they pose to the people involved. This must include both normal and abnormal operating conditions;

(c) Action be taken to reduce any identified risk to as low a level as can reasonably be achieved;

(d) Where protection from exposure is necessary this must be achieved, wherever practicable, by primary containment and the use of engineering controls. Dependence on personal protective equipment as the primary means of protection is only acceptable where control cannot reasonably be achieved by any other means;

(e) Routine atmospheric monitoring must be carried out for the substances of concern and records maintained;

(f) Where appropriate routine medical surveillance of staff must be undertaken;

(g) Staff must understand the hazards and be adequately trained to cope with them;

(h) Emergency procedures must be drawn up and their effectiveness regularly tested.

6.3 Whilst these regulations do not apply outside the United Kingdom, the principles they set down remain valid. They illustrate the type of approach necessary for the development of safe and effective working procedures. If the task is approached in this manner there is no reason why it should not be carried out successfully, with the minimum risk to the operators and the surrounding population.

7. CONCLUSIONS

7.1 The aim of this short paper is to review some of the options available for the safe disposal of stocks CW agents, munitions and production and storage facilities. It is intended as a discussion document and an aid to reaching an agreement on how the disposal of these materials might be achieved in a timely, safe, environmentally acceptable and cost effective manner.

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"OLD CHEMICAL WEAPONS" DISPOSAL

(Subject presented by Dr.Hermann Martens, NBC Defence Establishment Munster (WWD), at the meeting of Technical Experts on CW Destruction, Geneva, 7-11 October 1991)

Introduction

The term "old chemical weapons" (OCW) as used in this paper relates to chemical munitions with their fillings, which were produced during or before World War II. Due to external corrosion, unpredictable ballistic properties and fuze mechanisms and the extent of decomposition of chemical fillings, at least those OCW found in Germany are no longer of military use as chemical weapons (CW). However, since they represent a burden to the environment and a potential hazard, Germany decided to destroy the OCW recovered on its territory.

As a result of workshops held in the NBC Defence Establishment Munster (WWD) in 1984 and 1990, the Conference on Disarmament was informed on the issue of OCW accidentally or systematically recovered in Germany after World War II (cf. CD/518, dated 17 July 1984; CD/1026, dated 3 August 1990).

This paper describes in some detail the procedural steps of the OCW disposal concept elaborated and adopted. It also touches on some general aspects and the historical background.

1. General remarks

The undertaking in Germany relates rather to a "collection" of various OCW items than an "OCW stockpile". Most of these items, including captured OCW from other countries, were sporadically recovered in the past four decades.

Each of these items requires individual assessment by explosives ordnance disposal (EOD) personnel of its condition prior to destruction. Therefore, a high degree of automation aimed at speeding up certain steps in the disposal process, which would also reduce costs and improve the working conditions of the personnel, does not appear to be feasible at the present stage. Whenever possible, high-risk operations must be carried out under remote control.

A completion date of the disposal programme is difficult to predict, given the considerable uncertainty of the total amount of OCW that might still be hidden in the ground.

Only some of the experience gained in terms of the technology used, time frames and costs of the overall effort may apply to the destruction of large current CW stockpiles. However, Germany is ready to share its experience with other countries also faced with similar problems.

2. Types of OCW munitions and chemical fillings

A large variety of OCW munitions, and even storage tanks, produced within the 1915-1945 time frame, may be found. These include

- artillery shells
- mortar ammunition
- hand and rifle grenades
- land mines
- bombs
- spray canisters.

The wide spectrum of toxic chemicals then used as fillings in OCW munitions includes various preparations of sulphur mustard, nitrogen mustard, lewisite, other arsenicals (e.g. Adamsite, Clark 1, Clark 2), phosgene and tabun.
Sulphur mustard fillings in many cases contain arsenicals such as phenyl dichloroarsine or "arsine oil".
Only a small number of tabun shells have been recovered in Germany and were destroyed.

The recovered OCW items originate from introduced or experimental types of chemical munitions, which are increased in number and variety by items captured from various adversaries of the World Wars. Hence, OCW munitions were dispersed over large parts of Central Europe, due to both WW I operations and battlefields (Flanders) and the dislocation of chemical weapons to depots in World War II.

Former test sites and destroyed production or filling sites (like Munster), are believed to still have OCW buried in the ground, which include failure batches of viscous sulphur mustard in spray canisters.

3. Procedural steps of OCW disposal

3.1 Reconnaissance and locating

Prior to the scouring of the terrain for the presence of OCW, a comprehensive search of the archives and all other available sources, including evident facts and notes of eye witnesses, is required. Thorough evaluation of aerial infrared photographs has proven to be a suitable means. Irregularities of the surface in areas surrounding former CW production or filling facilities would indicate underground artifacts, such as hidden munitions.

Reconnaissance missions on the ground are carried out by EOD experts using magnetic probes or other metal detectors. These instruments are capable of locating metal parts up to a depth of 6 meters. Those spots giving a positive detector response are marked with flags and safeguarded to prevent unauthorized access.

3.2 Unearthing and identification

In order to recover and identify the artifact hidden in the ground, EOD experts cautiously clear away the soil until the item or the number of items can be recognized visually. At a later stage mobile X-ray equipment is used to further evaluate the internal structure of the munition. Following the preliminary assessment of the item as to whether or not it relates to OCW, its state of preservation and possible leakage, a decision is made on its transportability. Some OCW munitions have to be disassembled and decontaminated on-site.

Unidentified items are transported to the nearest demilitarization facility and opened for sample-taking and chemical analysis of the contents.

During the whole process of unearthing and identification, appropriate safety areas around the discovery points must be established and standardized operational procedures (SOPs) of individual protection applied.

3.3 Removal and transportation

Since OCW items are often found in places quite distant from each other, they have to be collected and transported to an intermediate storage site.
For transportation of OCW items rugged containers are needed which remain gas-tight under pressure and which are coated in a way as to allow the use of aggressive decontaminants. The transport containers must be approved by the federal authority for transportation of dangerous goods and explosives.

The planning of transportation comprises negotiations with federal state police departments and environmental agencies. Detailed agreement is required on the routes to be taken. In case of significant quantities, OCW transports must be secured by police and accompanied by trained personnel for detection, decontamination and medical support.

3.4 Intermediate storage

In order to facilitate further treatment and final destruction and for reasons of safety and clarity, the storage of OCW is organized in a way that identical munition items and heterogeneous ones are collocated. This, of course, results in a need for facilities for the safe intermediate storage of OCW, which have been removed from different areas of the region or other federal states.

The storage bunkers must meet a number of stringent standards with regard to over-pressure stability, fragment protection of walls and doors, gas-tightness in case of accidental release of toxic chemicals, chemical agent detection and alarm systems. Filtered air ventilation and automated fire-fighting installations including sprinklers are also needed to ensure occupational safety and the protection of the environment. In addition, a basin for the collection of waste water in eventual decontamination or fire-fighting missions is required.

The bunker, with its separate compartments, is structured in a way which keeps the various types of munitions segregated. Walls of adequate height are installed in order to prevent the propagation of eventual explosions to other munition staples.

The storage facility is subject to frequent inspections in accordance with safety and security regulations issued for its operation.

Plans for early warning and eventual evacuation of the population living in surrounding areas have to be established. These plans must be continuously updated taking into account changing results of risk analyses. Although very unlikely, the release of toxic chemicals as a result of explosions, fires or direct impact of an airplane cannot totally be ruled out.

3.5 Preparation of OCW for demilitarization ("demil")

The munitions excavated from the ground are mostly covered with dirt and rough layers due to corrosion. For this reason, prior to further treatment, they have to be cleaned using a high-pressure water jet.

Since the internal structure of recovered OCW differs considerably from item to item, x-raying of each of them individually is absolutely crucial, followed by a thorough evaluation of the negative film produced, which includes as accurate measurements as possible of all relevant features shown on the film. Only then are the EOD experts in the position to determine the detailed demil route, which is pertinent to the specific OCW item.

In order to reduce the internal pressure of volatile liquid or gaseous chemical fillings, the munition items are frozen in a final step prior to demil. Thus, agent spillings out of the shell can be avoided or at least limited when being opened.

3.6 Demilitarization

Demil of OCW is the mechanical disassembling of munition items, which includes the elimination of the fuzes, explosive charges (bursters) and the draining of chemical fillings. In most cases, for the execution of these particularly critical and hazardous steps, remote-controlled special demil tools and devices are required. These include defuzing tools, machines for drilling, milling and sawing and chopping benches.

In the event of explosions or release of toxic chemicals, the personnel operating these devices from a demil control bunker are protected against fragments, vapours and aerosols by thick concrete walls and an air filtration system of high efficiency.

The demil process is constantly monitored and video-recorded, which provides thorough documentation and retrospective evaluation of the entire operation.

The techniques that have been developed for the draining of OCW include

- the draining of liquids after piercing or drilling through the wall of the compartment;
- expansion of pressurized volatiles (e.g. phosgene) through a pipe system connected with an absorption column;
- removal of pasteous fillings using special tools, such as spatulas;
- removal of solid fillings from opened munitions by melting through hot steam injection.

Draining of chemical munitions bears a high risk of contamination not only of the demil devices themselves, but to all surfaces inside the demil chamber. Therefore, encapsulation of all parts not related to actual mechanical operation is required. Impermeable protective suits and respirators with filter canisters must be worn by personnel while working in the demil chamber on the preparation, maintenance or repair of demil devices.

At present, none of the aforementioned draining techniques is used in automated continuous operation. (In the case of large numbers of sufficiently uniform munitions, typical of current CW stocks, a highly automated demil line would be required and has been shown by the United States to be feasible.)

For thorough decontamination of all exposed surfaces inside the demil chamber a highly effective decontaminant, such as a decont emulsion introduced by the Federal Armed Forces, must be kept in readiness.

After any mission that could involve exposure to toxic chemicals, the protective equipment used has to be decontaminated and carefully inspected.
Furthermore, special first-aid medical support and transportation with ambulances must be ensured at any time while demil operations are being carried out.

4. Destruction of chemical agents

The German OCW disposal concept includes as a decisive step the incineration of the chemical agent fillings at high temperatures, which results in the complete mineralization of the chemicals. The combustion flue gases are scrubbed and stack emissions continuously monitored in a way as to ensure that stringent German environmental standards are met.

Prior to incineration, chemical agents and demil wastes as well as empty shells are intermediately stored in polyethylene (PE) containers.

The incineration plant near Munster is designed for a destruction capacity of approximately 70 agent tonnes per year.

An additional plant is currently being planned for the disposal of both soil contaminated with arsenicals and explosives mixed with toxic chemicals.

4.1 Incineration of chemical agents

The Munster incineration plant, after approximately 5 years of planning and construction, began full operation in 1980. It includes a batch-type double chamber furnace with unique features primarily designed for the destruction of viscous sulphur mustard. In two overlapping shifts or a 14 hour working day, up to 350 kg of sulphur mustard can be incinerated.
(Further technical and other data on the plant, including costs of construction and operation, are specified in Annex 1; the functional diagramme of the plant is shown in Annex 2).

Prior to incineration the chemicals are analyzed for the presence of arsenicals. Based on analytical results, batches are put together and the conditions for the most effective waste and effluent air treatment adjusted accordingly.

The re-opened PE storage containers are loaded on a heat resistent charging wagon which is then moved through a gas lock into the evaporation chamber. There, at 300°C and in an inert atmosphere (N_2+CO_2+H_2O), the chemicals are vapourized within 10 to 12 hours and the vapours introduced through an insulated duct into the main incineration chamber.

In this chamber, sulphur mustard is oxidized at temperatures between 1000°C to 1200°C, and within a reaction time of 2 seconds converted into sulphur dioxide (SO_2), hydrogen chloride (HCl), carbon dioxide and water.

The charging wagon, carrying unvapourized organic residues and metal parts, is then moved into the burn-out chamber. Here, the metal parts, e.g. shells, are annealed in air at 1000°C during 12 to 18 hours. The effluent air from this chamber is conducted through the main incineration chamber for final combustion of toxic components.

The charging area and displacement room are adjusted to an underpressure of 0.5 to 1.0 millibar below atmospheric pressure in order to prevent any toxic vapour from escaping to the environment.

4.2 Flue gas scrubbing

Flue gas from chemical agent incineration at first is cooled down to 80°C by injection of water into the cooling tower (quench). In passing two washing towers (scrubbers) arranged one after the other, the noxious gases SO_2 and HCl are eliminated with additional water; by simultaneous injection of sodium hydroxide (NaOH) a defined pH profile is achieved.

As a result of waste water neutralization, sulphur mustard is finally converted into harmless salts. The sodium sulfate and sodium chloride formed can be released into the municipal waste water system.
The scrubbed flue gas leaves the plant via the stack after removal of particulate matter by an aerosol separator. Arsenic trioxide, formed under incineration of materials containing arsenicals, is also removed from the flue gas in this process.

Sampling probes and analyzers for the continuous monitoring of the emissions of SO_2, HCl, hydrocarbons and dust (including traces of arsenic trioxide) are attached to the stack. The emission data are transmitted to recorders installed in the switchboard panel in the central operation control room. All recorded data have been far below the limits set by law for permitted emissions.

4.3 Precipitation of arsenic compounds

Arsenicals or mixtures containing arsenic require as an additional effort the collection of the entire washing water. This aqueous solution of mineralized arsenic(III) compounds is then subjected to oxidation and subsequent arsenic precipitation.

The mineralized arsenic(III) compounds are oxidized by permanganate to form arsenate(V). The addition of ferric chloride leads to a flocculation and dragging effect of the resulting ferric hydroxide, accompanied by the precipitation of ferric arsenate. This sludge-type material is dehydrated by a filtration step and the filter cake filled into plastic bags placed in 200 litre steel barrels.

4.4 Waste product disposal

The filter cakes containing arsenic eliminated from the washing water, as well as solid wastes with residual arsenic, if found on the charging wagons, are finally deposited in an old underground salt mine.

The purified filtration waters are disposed of into the municipal sewer system. However, because of their high salt concentration, a waste water evaporator will be required in the future.

Conclusion

The disposal of OCW recovered on German territory represents a long-term effort, and the time required for its completion is hardly predictable.

Incineration is felt to be an appropriate method of destruction of OCW (and of current CW), since it leads to irreversible total mineralization of the toxic chemicals involved. (In a few exceptional cases, as in the case of phosgene, hydrolysis could be more effective.) The double-chambered incineration plant in Munster is likely to continue its operation on the long term. Due to the lack of uniformity and the extent of corrosion, most of the OCW items need to be handled individually in a time-consuming and costly process.

Procedures aimed at speeding up the disposal of OCW require further efforts of technological research and development and extensive testing. The incineration of OCW fillings using universal furnaces, such as a rotary kiln, seems to be feasible in a continuous process.

Manual techniques will widely continue to be employed in the searching, excavation, transporting and demil activities. However, improved physical and chemical methods of identification of types of munitions and fillings could facilitate and accelerate the demil steps.
In any case, considerable efforts in the areas of protection of personnel and decontamination will continue to be necessary.

Annex 1

A. Operational data

(1) Operating time:

14 h/day, 2 overlapping 8 hour shifts
(during weekends or holidays temperature is maintained by support firing)
200-250 days/year available for operation

(2) Throughput:

22-35 kg/h (relating to sulphur mustard)
capacity of charging wagon: 24 PE containers (30-l-barrels)

(3) Consumption:

fuel oil (EL)	50-200	l/h
electric power	4,500	kWh/day
tap water	2	m3/h
inert gas	400	Nm3/h
compressed air	60	Nm3/h

(4) Waste gas & emissions:

waste gas rate	2,800		m3/h
temperature	105		oC
height of stack	30		m
sulphur dioxide	< 5	(100)	mg/m3
hydrogen chloride	< 5	(50)	mg/m3
arsenic	0.2	(1)	mg/m3
nitrogen oxides	200	(250)	mg/m3
dust	< 1	(20)	mg/m3

(in brackets: limits permitted by law)

(5) Waste water:

rate		0.5	m3/h
salinity		5-40	g/l
arsenic	(adjusted to)	< 1 (1)	mg/l
pH value	(adjusted to)	8	

B. Materials destroyed since 1980

(1) chemical agents	75	tonnes
(2) contaminated materials, incl. munition scrap	441	tonnes

C. Materials in storage & ready for destruction

(1) At Munster-North Training Area:

chemical agents	115	tonnes
contaminated earth	30,000	tonnes

(2) In the German federal states:

chemical agents (mainly arsenicals)	30	tonnes
contaminated earth	1,000	tonnes

D. Costs

(1) Construction:

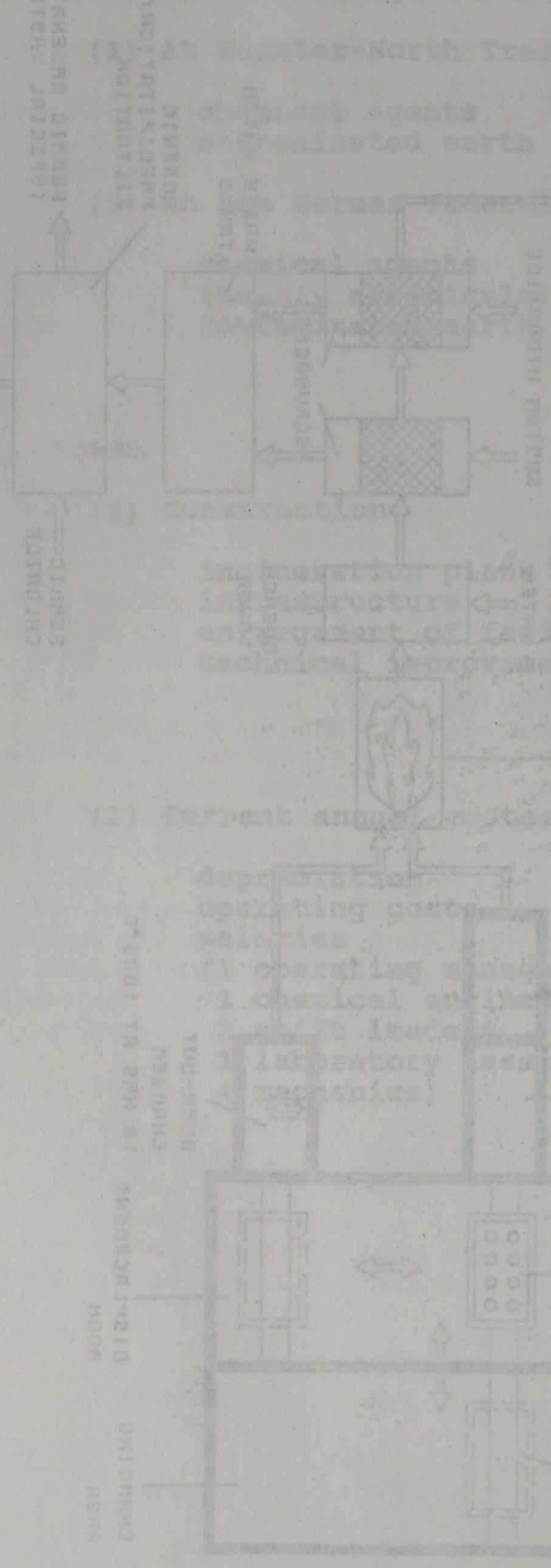
incineration plant & related infrastructure	16	Mill. DM
enlargement of facilities	4	Mill. DM
technical improvements	8	Mill. DM
total:	28	Mill. DM

(2) Current annual costs:

depreciation	1.1	Mill. DM
operating costs	1.0	Mill. DM
salaries	0.7	Mill. DM
(1 operating manager, 1 chemical engineer, 2 shift leaders, 1 laboratory assstant, 4 mechanics)		
total:	2.8	Mill. DM.

Annex 2

Functional diagramme of the Munster incineration plant



CONFERENCE ON DISARMAMENT

CD/CW/WP.375
20 November 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

ITALY

Italian experience of the destruction of old and obsolete chemical weapons

1. Introduction

The Military Establishment of Nuclear, Bacteriological and Chemical (NBC) Defence and the Chemical, Physical and Biological Technical Center of the Italian Army are responsible, in Italy, for the problems relating to the destruction of the old stocks discovered on the national territory; both the Establishment and the Center are located in the Military Area of Santa Lucia - Civitavecchia - Rome, and are under the supervision of the General Direction of the Weapons, Ammunitions and Land Armaments.

Since the end of World War II the NBC Establishment was responsible for retrieving and stocking in safety the old stocks, found on the national territory.

In 1974 studies started for realization of a plant for the old stocks destruction, particularly of the mixture mustard-phenyldichloroarsine (Y-PhDA).

We learned from the analyses carried out by the Technical Center, with the collaboration of the Atmospheric Pollution Institute, that the above-mentioned mixture consisted of mustard (50 per cent), PhDA (45 per cent) and degradation products (mainly arsenic trichloride and hydrochloric acid) (5 per cent).

The Technical Center and NBC Establishment proposed two different chemical methods for the destruction of the mixture by oxidation and hydrolysis, the first method using, as oxidant, hydrogen peroxide, the second nitric acid.

The study for the realization, based also on economic considerations and environment protection, led to a preference for the Technical Center method, namely the one using H_2O_2, in fact the destruction by nitric acid involved the formation, during the process, of a great quantity of vapours and gases (nitrogen oxides), another problem was the difficult control of the reaction temperature.

2. Destruction plant of the mixture Y/PhDA

2.1. Process characteristics

The process is based on a liquid phase oxidation of the mixture by hydrogen peroxide (130 vol.), followed by a neutralization by lime and then cementation of the reaction products and preservation in safety of the concrete thus obtained.

The mixture is emulsified with a surface active compound and then reacts with the concentrated solution of hydrogen peroxide.

The addition of hydrogen peroxide causes a rapid raise in temperature.

Through a cooling system the temperature is then stabilized at 95° C, which seems to be the right compromise value to allow the following:

- a rapid reaction rate, on the one hand;

- operative conditions in safety, on the other.

The control of the reaction temperature is obtained by a feedback mechanism which affects the quantity of hydrogen peroxide added and the water cooling system.

The analytical controls showed that both the mustard and the PhDA were transformed, after the oxidation reaction, into products with a lower toxicity, mainly by the presence of arsenic.

During the reaction a great quantity of hydrochloric acid is generated in the reactor, making a subsequent neutralization treatment necessary, for this treatment a solution of lime is used.

At the end of the neutralization the reaction products do not contain mustard and PhDA but they still contain, of course, arsenical products.

This presence does not allow the treatment of the residual products as normal industrial waste.

For this reason these products are mixed with cement and sand forming a concrete and stored in special containers of vibrated concrete. This conservation method guarantees against the release of the residual products into the environment, in fact a number of factors seem to indicate that chemical bonds are formed between the cement and the molecules of the residual products.

The project was realized by the technical personnel of the NBC Establishment (mainly Maj. Costantino) and the construction of the plant was assigned to the ITALIMPIANTI company.

2.2. Plant description

A side section of the plant is showed in Figure 1, where it is possible to see that the products move by gravity.

The main components of the plant are:

- bunching tank;
- dephlegmators;
- pilot reactor;
- emulsifying solution tank;
- oxidation reactor;
- neutralization reactor;
- cooling towers;
- chiller;
- cooling system;
- control room.

2.3. Detailed plant operation (Fig. 2)

The mixture of Y/PhDA contained in three tanks, is transferred by suction into a vacuum bunching tank.

The addition of an emulsifying solution, equivalent to 25 per cent of mixture volume, and a homogenizing system by turbine agitator were included in the system to minimize the negative factors, for example connected with the presence of pitchy materials in suspension.

The mixture is transferred by gravity from the bunching tank to the oxidation reactor, where it is further emulsified by agitation after the addition of a solution of water and a surface active compound.

The oxidation reaction is started by the addition of hydrogen peroxide into the reactor, this reaction is heavily exothermic and develops a great amount of hydrochloric acid.

The extremely corrosive conditions imposed the use of an enamelled steel reactor.

The need to avoid possible hydrogen peroxide pockets and the subseqent rise in temperature in the reactor, makes it necessary to use an impeller stirrer that allows a complete homogenization to be obtained.

The oxidation reaction of the mixture occurs at atmospheric pressure and at 95° C, utilizing a cooling system for the reactor by a forced water circulation jacket.

At the conclusion of the reaction, which is evidenced by the sudden fall of temperature, the residual products are cooled to 40° C and transferred into the neutralization reactor by gravity.

The neutralization reaction occurs in the enamelled reactor through the addition of water and lime.

After this treatment, the products are transferred into a properly modified concrete mixer by hermetic seal duct.

A measured quantity of cement, coming from a silo, and the sand, additioned by belt conveyor, flow together into the same concrete mixer.

At the end of this treatment a concrete is obtained, it is transferred into vibrated cement containers that are sealed with special adhesive and cement plug.

The sealed containers are stocked in a special depot of the Military Area in accordance with the destruction programme of the Y-PhDA and the laws in force about the stocking of toxic and noxious products.

The whole plant, the different subsystems and the piping are constructed in a manner to ensure that the destruction process can be easily verified.

2.4. Safety features

The plant was projected to ensure the "risk prevention" for the environment, the staff and the limitrophe zone.

The fundamental elements of the project imply the use of oxidation and neturalization reactions at atmospheric pressure, low temperature (less than 95° C), with controlled addition of reagent and absence of great production of vapours and gases.

These conditions allow the elimination of the overpressure in the reactors, excluding the possibility of breaches by explosion.

The plant is provided with a suction system that maintains the inner part in light depression, it is also provided with activated carbon filters to absorb possible vapour leaks from reactors.

A sensor system has the task of checking the presence or absence of toxic atmosphere in the reactor room.

Efficiency and safety of the plant were tested by checking:

- the correspondence to the project's characteristics of the machinery and equipment;
- the neutralization chemical process by employment of non-toxic simulators;
- the "risk prevention" and the "first alarm" by the simulation of possible damages and accidents.

The use of a control computer to run the plant and locate possible breakdowns is included.

The plant passed the test runs, using also a pilot reactor. In this phase 1.5 metric tons of mixture Y-PhDA have been destroyed with batches of 20-25 kg.

The industrial phase will begin within a few days and will allow for the destruction of 250 kg of mixture per batch.

2.5. Costs

The cost of the destruction plant was US$ 2,100,000. The operating costs per year amount approximately to US$ 170,000. The costs of the personnel operating the plant (8-10 people) amount to US$ 200,000. Taking into account all costs factors, the costs for the destruction of 1 kg of mixture amount roughly to US$ 18-20.

3. Other plants

In a later moment, after having discovered a certain quantity of old and obsolete rounds and an important amount of adamsite, it was considered necessary to cope also with these new problems.

Two further plants, the first for the discharging of old and obsolete chemical rounds and the second for the demilitarization of adamsite mixed with inert materials, are planned.

3.1. Automatic plant for discharging old chemical rounds

The plant will be constituted by:

- a structure for the execution of radiographies;
- a structure for the discharging of rounds;
- a command and control room.

3.2. Adamsite treatment plant

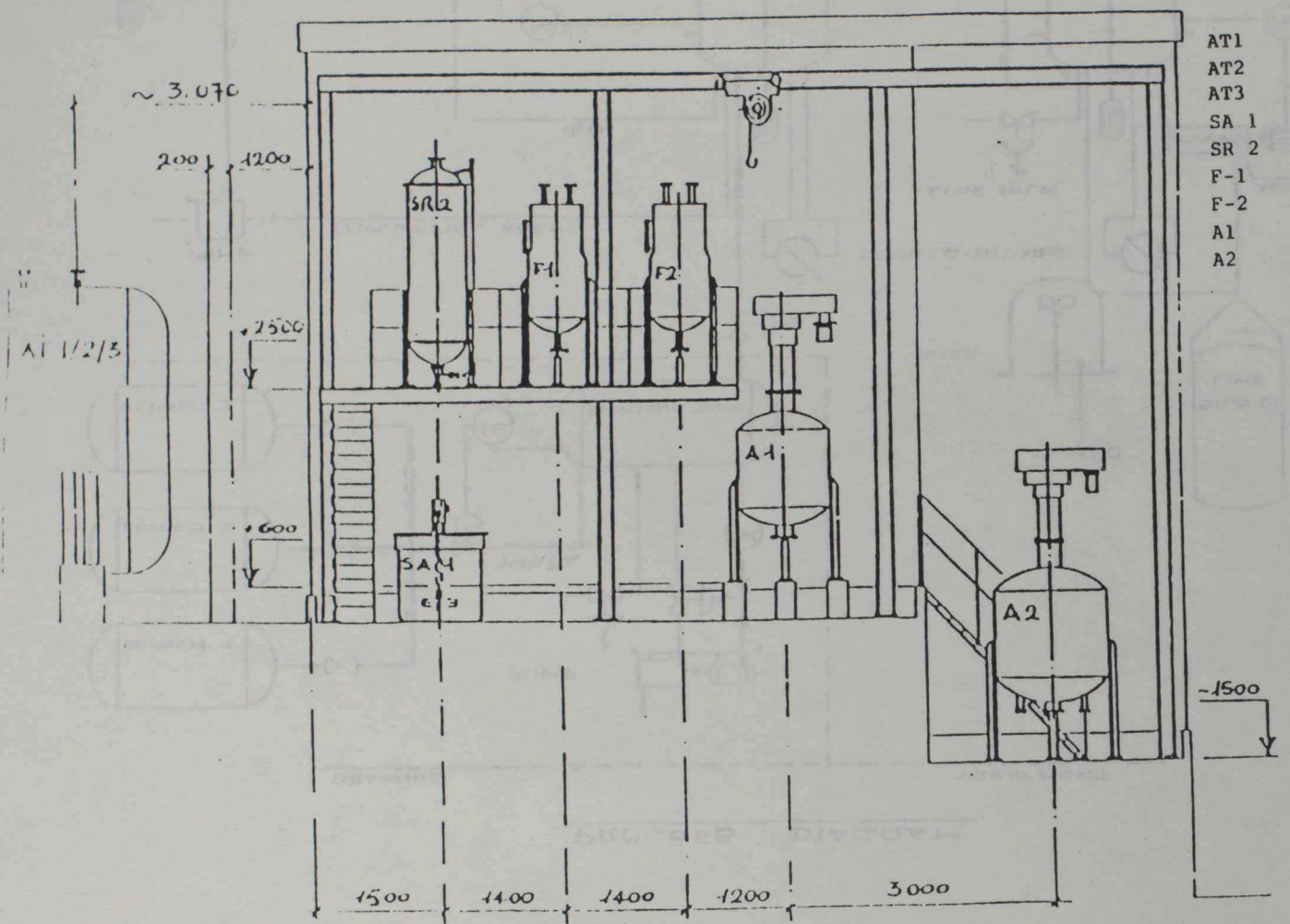
The adamsite retrieved on the national territory is currently stocked in metal containers.

The plant will allow the treatment of adamsite mixed with inert material by transformation into concrete.

The phases of the treatment will be:

- Crushing and milling;
- Mixing with cement and sand to form a concrete;
- Stocking the concrete in cement containers;
- Cleaning of the metal containers.

REACTOR ROOM



AT1	DEPOSIT No.1
AT2	DEPOSIT No.2
AT3	DEPOSIT No.3
SA 1	TANK OF EMULSIFYING AGENT
SR 2	BUNCHING TANK
F-1	DEPHLEGMATOR
F-2	DEPHLEGMATOR
A1	OXIDATION REACTOR
A2	NEUTRALIZATION REACTOR

SCALE 1:60

FIGURE 1

PROCESS DIAGRAM

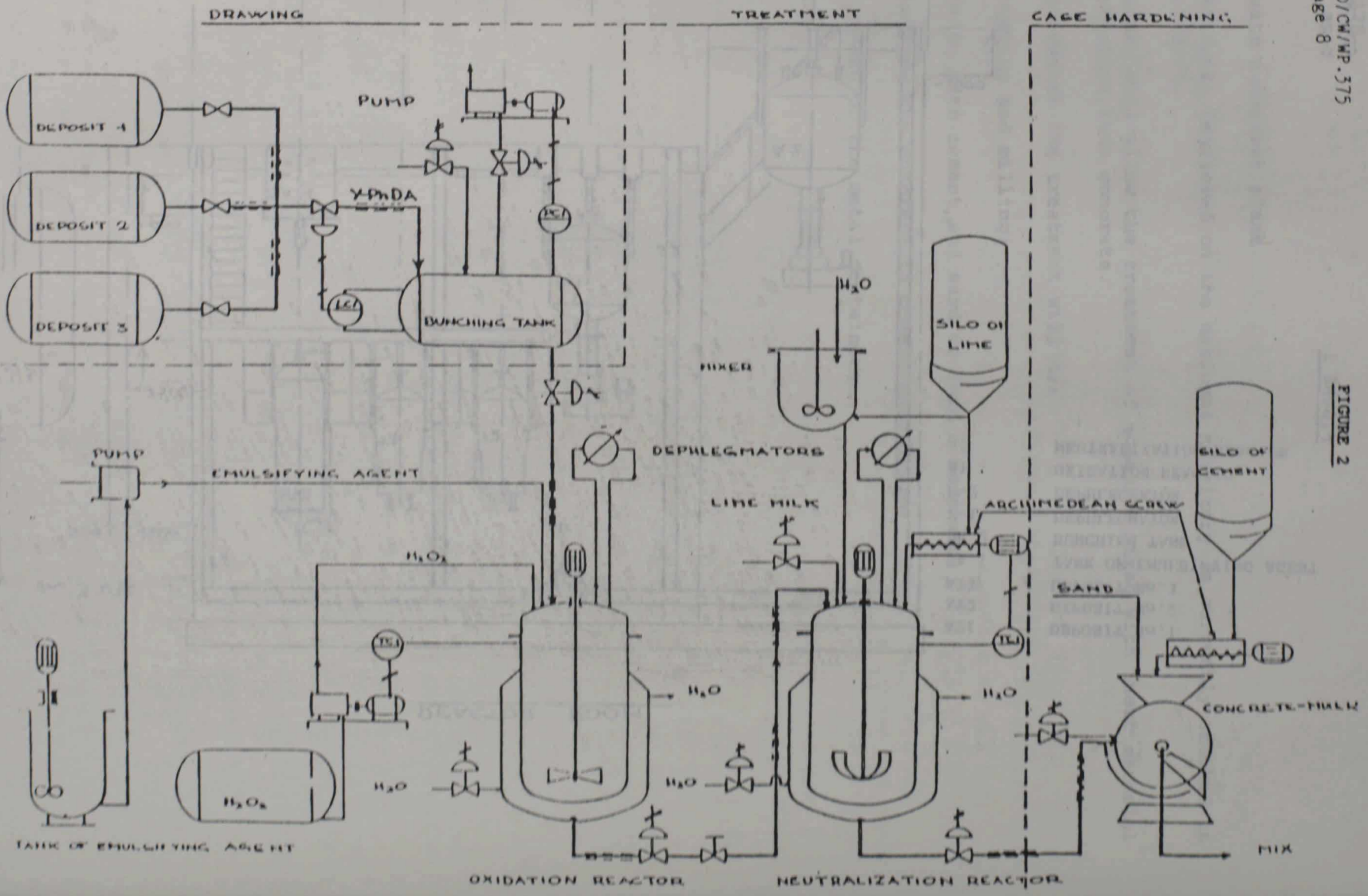


FIGURE 2

CONFERENCE ON DISARMAMENT

CD/CW/WP.376
6 December 1991

ENGLISH ONLY

Ad Hoc Committee on Chemical Weapons

THE NETHERLANDS

Verification of alleged use of chemical warfare agents: retrospective immunochemical detection of exposure to mustard gas

SUMMARY

It is argued that unequivocal methods are needed to verify exposure of alleged victims to chemical warfare agents, in order to sustain the credibility of the forthcoming Chemical Weapons Convention. Since samples for analysis can often be obtained only several days after exposure or later, the methods should be very sensitive and should relate to long lasting, specific effects of the CW agent involved.

In this context immunochemical methods have been developed for retrospective detection of exposure to sulfur mustard. Other agents are under consideration. Since the adducts of CW agents with macromolecules have life times of several days up to several months, the adducts of sulfur mustard can be detected over considerable time periods. In principle, immunochemical detection techniques of reaction products of CW agents with macromolecular constituents in the body of supposed victims, when developed, are simple to perform and can be applied under field conditions. These methods are also highly useful to monitor exposure to CW agents in persons involved in the destruction of stocks of such agents.

The methods are based on reaction products of sulfur mustard with DNA and proteins. The feasibility of the approach has been demonstrated using monoclonal antibodies raised against the major reaction product of sulfur mustard with DNA bases. With a competitive ELISA based on these antibodies we detect exposure of human blood to $\geq$ 2 μM sulfur mustard, whereas direct exposure of human skin to non-blistering Ct-values of sulfur mustard can be detected with immunofluorescent methods based on the same antibodies.

GE.91-62915

1. INTRODUCTION

Methods to verify the alleged use of chemical warfare (CW) agents should be available in order to sustain the credibility of a Chemical Weapons Convention (CWC) banning the production, posession, and use of chemical weapons (1). Presumably, allegations for the use of CW agents will be based primarily on the observation of injuries in supposed CW victims. In view of the far reaching political and military consequences of illegal use of CW agents, unequivocal methods should be available which stand in a court of law to prove, or disprove, the exposure of alleged victims to CW agents. Methods such as survey interviews of supposed victims can at best give circumstantial evidence for alleged use of CW agents (2).

Several incidents in the recent past demonstrated the present lack of reliable methods to verify exposure to CW- agents. The most straightforward case was the use of mustard gas, and possibly also of tabun, in the First Gulf War (3). With severe casualties in hospitals all over Europe, analyses of agents and metabolites had to be improvised. The results were inconclusive (4). The controversies with regard to the use of mycotoxins as an agent ("yellow rain") in Southeast Asia, which arose from the analyses of environmental and biological samples, were widely publicized. These incidents were reviewed (5). Rather recently, rumors were spread on the use of CW agents in Angola. Samples from the casualties were analyzed, with disputable results (6-8). In the more distant past, the alleged use of CW agents in Yemen could not be confirmed, due to lack of adequate methods of analysis (9).

Experience with the above-mentioned incidents learned that urine, blood and other biopsies or autopsies for analysis can often be obtained only several days or even weeks after exposure. Therefore, verification methods for biological samples should be very sensitive and should relate to long lasting, specific effects of the CW agent under investigation. Such methods are not yet available for the common CW agents. For example, intact nerve agents can be analyzed in blood, brain, and muscle tissues at minimum detectable levels in the low picomolar range. However, these levels are exceeded in primates only for a few hours after intoxication at high doses (10). An alternative, the observation of low levels of cholinesterase activity, is not specific for nerve agents. Possibly, development of sensitive methods of analysis for hydrolysis products in urine may provide a more promising approach to retrospective detection of nerve agent exposure (11).

The large scale use of sulfur mustard in the First Gulf War, demonstrated the renewed interest in this agent. Therefore, we selected this agent (12) to develop methods for retrospective detection of exposure. Presently available methods seem unsatisfactory. Recent reports on detection with gas chromatography in combination with mass spectrometry (GC-MS) of intact sulfur mustard in an abdominal fat sample obtained from autopsy of an Iranian soldier who died seven days after exposure to sulfur mustard (13), and in the urine of another soldier seven days after exposure (14,15), need further confirmation. Neither has the older report by Stade (16) been confirmed on the presence of intact agent in skin blisters caused by sulfur mustard. Attempts to verify exposure to sulfur mustard via analysis in blood or urine of its hydrolysis product thiodiglycol (17), and of thiodiglycol derivatives which are (re)converted into sulfur mustard with hydrochloric acid (18,19), were complicated by the presence of these products in samples from non-exposed volunteers. Reports on the identity of further metabolites of sulfur mustard are contradictory and lack spectrometric evidence (20,21). The metabolism of

sulfur mustard is currently being investigated by CBDE (Porton Down, UK). Based on radioactivity measurements, 80-90% of the metabolites are excreted within 48 hours.

We have chosen to develop immunochemical detection methods of "adducts," i.e., reaction products that are generated by alkylation of DNA bases and proteins by in vivo exposure to sulfur mustard.# This choice is based on extensive experimental evidence which shows that analogous methods of analysis for DNA adducts of cytostatic agents and environmental alkylating agents can be highly selective, detecting one modified base in DNA among $\leq 10^8$ unaltered bases (23). The minimum detectable concentration of modified bases lies in the low femtomolar range. If cells producing monoclonal antibodies to the adducts can be isolated, detection methods based on these antibody-adduct interactions can be performed on a large scale, with quantitative results. Although alkylated bases in DNA can undergo secondary reactions and the damage resulting from adduct formation tends to be repaired, the adducts are detectable for days or even weeks after exposure (23).

In general, methods to detect exposure to alkylating agents based on analysis of protein adducts in biological samples (biomonitoring; for reviews see 24-26) are complementary to methods based on analysis of DNA adducts. In contrast to the immunochemical detection methods for the latter adducts, protein adducts are usually quantified by GC-MS analysis after total hydrolysis of the protein and derivatization of the alkylated amino acid(s). Therefore, relatively few results are available for the immunochemical detection of protein adducts (27,28). A priori, it should be assumed for stoechiometric reasons that in vivo exposure to alkylating agents yields much more adducts to proteins than to DNA, unless the agent reacts specifically with DNA. Moreover, it has been shown that the life span of proteins is not shortened by alkylation. Human hemoglobin, with a biological half life of 16-18 weeks, has been proposed as an easily available protein for biomonitoring exposure to various alkylating agents (24-26).

In recent experiments the degree of alkylation by ethylene oxide of the N-terminal valine in human hemoglobin was determined by means of radioimmunoassay as well as by GC-MS analysis. A good correspondence of the results was found. With ethylene oxide and other directly alkylating agents, a reasonably linear relationship between levels of alkylation of DNA and proteins was also observed (29).

When sulfur mustard is used in chemical warfare, the agent affects the skin in liquid or vapor form, whereas inhalation of vapor or aerosol causes extensive damage of the respiratory tract and lungs. Extensive, long-lasting systemic intoxication is also observed due to rapid penetration of the agent into the general circulation both via inhalation and the skin (12). Therefore, DNA and proteins from various biopsies may serve as samples to monitor exposure to the agent. Primarily, skin biopsies and nucleated blood cells are convenient to assess damage to DNA. Hemoglobin, albumin, and skin biopsies are logical targets for immunochemical detection of sulfur mustard adducts to protein.

The use of immunochemical methods to detect compounds listed in Schedules 1 and 2 at facilities has been suggested (22).

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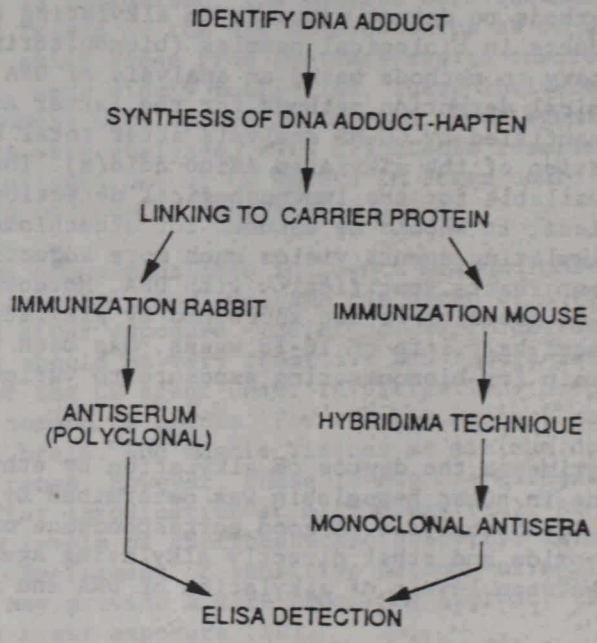
2. APPROACH

Before dealing with the work on sulfur mustard, it may be useful to summarize how an immunochemical detection method against adducts of small molecules like sulfur mustard is developed (see Scheme 1). The reaction of alkylating agents with DNA or proteins generally results in a number of different types of adducts, because of reaction with various components of the macromolecule. Therefore, before antibodies to one of these adducts can be raised, its chemical structure should be established. Next, a relatively small molecule is synthesized that comprises all essential structural elements of the adduct. This "hapten" is usually not suitable for direct use in immunizations, because it is too small to elicit an immune response. Therefore, the hapten is synthesized in such a way that it contains a "handle" which serves to couple it to a carrier protein. The protein, carrying multiple hapten molecules, is used for immunization. It is injected into rabbits in order to raise polyclonal antibodies against the adduct.



Scheme 1 Approach for the development of an immunochemical detection method for adducts to DNA.

This gives an antiserum having various antibodies in it, with a range of affinities for the adduct. It can be used to develop quickly the immunochemical detection techniques before monoclonal antibodies with a homogeneous affinity for the adduct have been raised, which is a rather time consuming effort. In order to obtain monoclonal antibodies, mice are immunized. Then, spleen cells are fused with immortal plasmacytoma cells. The fused cells, i.e., the so-called hybridomas, are selectively cultured in a special medium. Next, they are selected on the basis of the production of adduct-specific antibodies. Subsequently, they are diluted until single clones are obtained. Each of these

excretes one type of monoclonal antibody and can be kept alive and grown in unlimited quantities.
Most often, the antibodies are used to detect adducts with a so-called competitive ELISA (enzyme-linked immunosorbent assay; Figure 1). A fixed amount of

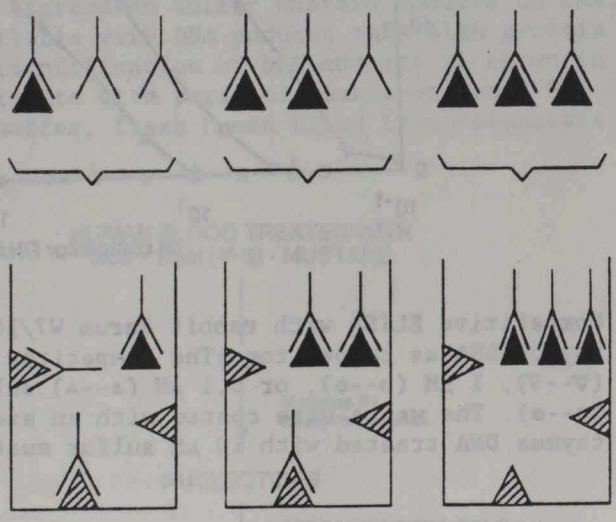


Figure 1 Scheme for a competitive ELISA. The upper part of the Figure symbolizes the preincubation of a fixed amount of antibodies (forkshaped symbols) with three different amounts of the antigen to be detected (black triangles). The lower part symbolizes the situation after the preincubation mixtures have been incubated in the wells of a plastic microtiter plate coated with excess of a different antibody-binding antigen (striped triangles). In the subsequent steps, the antibody molecules bound to the coating antigen are assayed quantitatively.

antibody is mixed with various amounts of the antigen that should be analyzed. Each of these mixtures is added to a small plastic well, which is coated with a fixed amount of another antigen which also can bind the antibody. The surplus antibody in the mixture is allowed to bind to the antigen attached to the wall. Next, the wells are washed, leaving the coating antigen-antibody complexes behind. Then, another antibody bearing an enzyme is added, which has affinity for the first antibody and is bound to the antigen-antibody complex. The enzyme splits a substrate into a product which is measured, e.g., fluorometrically.
The more antibody is bound to the first antigen, the less of it binds to the coating antigen. In this way, a dilution series of the analyte produces sigmoid curves as shown in Figure 2. With increasing concentration of adduct-containing

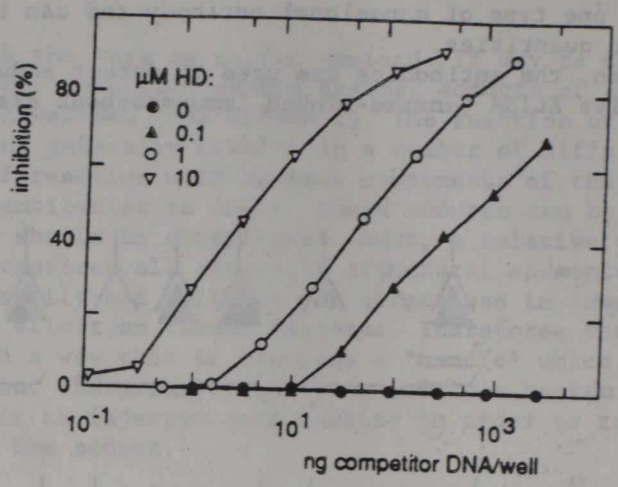


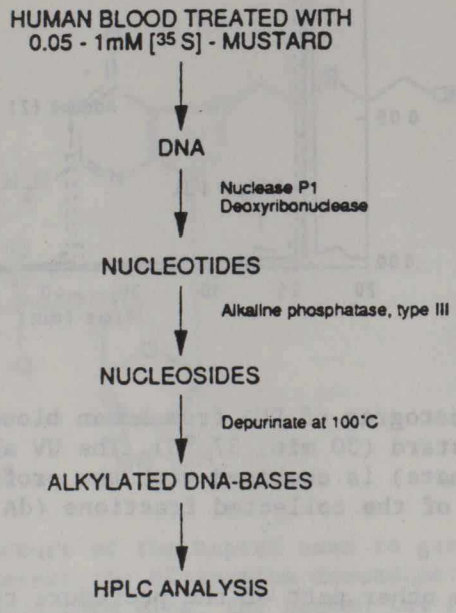
Figure 2 Competitive ELISA with rabbit serum W7/10 and single-stranded calf thymus DNA as competitor. The competitor DNA was treated with 10 μM (▽--▽), 1 μM (o--o), or 0.1 μM (▲--▲) sulfur mustard or was untreated (●--●). The wells were coated with an excess of single-stranded calf thymus DNA treated with 10 μM sulfur mustard.

DNA, there is more inhibition of substrate splitting. A similar effect is observed when DNA's are used with increasing adduct content.

3. RESULTS AND DISCUSSION

a. Identification of adducts with DNA

As mentioned above, the chain of events leading to a monoclonal antibody has to be initiated by identification of the adduct against which to generate an antibody. Work was started on sulfur mustard adducts to DNA, because more experience was available with DNA adducts than with protein adducts. Part of the procedure for identification of DNA adducts is shown in Scheme 2. After introductory experiments with purified double-stranded DNA from calf thymus in aqueous phosphate buffer, fresh human blood from volunteers was used.



Scheme 2 Work-up and analysis of sulfur mustard adducts with DNA in white blood cells of human blood.

It was incubated with ≤ 1 mM radioactive sulfur mustard. DNA was isolated from the blood cells and broken down enzymatically to nucleotides and nucleosides. The alkylated purine bases were released from the latter and analyzed with

liquid chromatography (HPLC). The chromatogram (Figure 3) shows one major and two minor radioactive peaks, corresponding with different

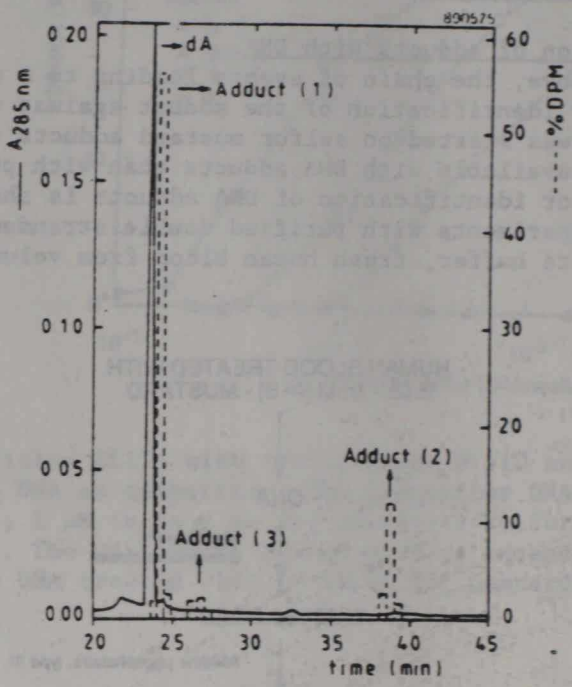


Figure 3 HPLC chromatogram of DNA from human blood, treated with 1 mM [^{35}]S-sulfur mustard (30 min, 37 °C). The UV absorbance profile (285 nm; left ordinate) is combined with the profile of radioactivity (right ordinate) of the collected fractions (dA = 2'-deoxyadenosine).

alkylated bases. The other part of the procedure to identify the DNA adducts was based on the synthesis of four adducts which are possibly formed with sulfur mustard (Figure 4).

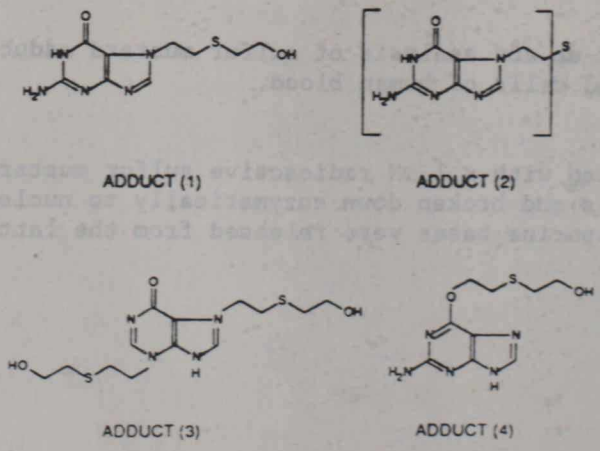


Figure 4 Chemical structures of synthesized sulfur mustard adducts with DNA bases.

Three of the adducts were supposed to be formed according to early investigations (30): the N7-guanine monoadduct with sulfur mustard (adduct 1), the corresponding di-adduct (adduct 2), and the N3-adenine monoadduct (adduct 3). Co-chromatography with the radioactive peaks (Figure 5) and other evidence, e.g., HPLC combined with thermospray-MS detection or UV spectroscopy, learned that the N7-guanine monoadduct was the major product, whereas the di-adduct and N3-adenine monoadduct were the minor products. Evidence for formation of the O6-guanine monoadduct (adduct 4) was not found (31).

b. Detection of DNA adducts in nucleated blood cells

Since the N7-guanine monoadduct is obviously the major adduct, a hapten based on this adduct (Figure 5) was synthesized. With reference to the structure of DNA, it was intended to synthesize this adduct containing a deoxyribose-5'-phosphate moiety. However, it

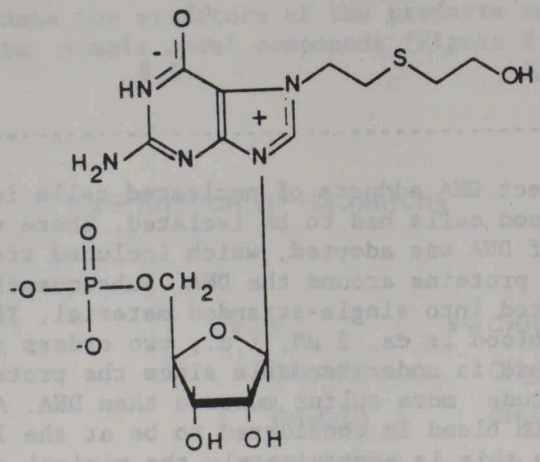


Figure 5 Chemical structure of the hapten used to generate monoclonal antibodies against the N7-guanine monoadduct of sulfur mustard. Before injection into mice, the hapten was bound to a protein via an activated ester of the phosphate moiety.

appeared that the glycosidic bond in such a hapten is too unstable during synthesis. Therefore, the deoxyribose was replaced by a ribose unit. Alkylation at N7 of guanine of this guanosine-5'-phosphate with sulfur mustard gave the hapten in sufficient quantity, in which the phosphate group is the handle to bind the hapten to a carrier protein. This product was linked to the carrier protein by means of an activated ester of the phosphate. Fortunately, the first attempts after injection in mice gave not less than ten hybridoma cell lines that produced monoclonal antibodies of the preferred class, i.e., IgG. Four of these were very specific for the major adduct in DNA. In fact, the sensitivity reached with these four monoclonals was approximately the same as that of the polyclonal antiserum that had been raised against calf-thymus DNA alkylated with sulfur mustard.

Optimization of the ELISA was labor-intensive. This was done with adducts induced in purified DNA. The major problem appeared to be the unwinding of the double-stranded DNA, which is also crosslinked by the sulfur mustard, into single-stranded DNA, without destruction of the N7-guanine adducts. Unwinding appeared to be essential for a good recognition of the adduct. Finally, a

procedure was adopted in which the DNA was made single-stranded by means of treatment with a low concentration of formamide, at low ionic strength. In this way (Table), exposure of single-stranded DNA to ≥10 nM sulfur mustard could be detected in the ELISA, whereas the same detection limit now holds for double-stranded DNA, when unwound in the proper way.

Table - Detection limits of competitive ELISA for calf-thymus DNA alkylated with 1 μM sulfur mustard

DNA sample	Detection limit N7-guanine monoadduct (adduct 1) at 50% inhibition point (fmol/well)
Single stranded	3.8
Double stranded	45
Double stranded/ unwound	2.9

In order to detect DNA adducts of nucleated cells in sulfur mustard-treated blood, white blood cells had to be isolated. These were lysed and a procedure for isolation of DNA was adopted, which included treatment with a proteinase in order to remove proteins around the DNA. Subsequently, the double-stranded DNA could be converted into single-stranded material. The detection limit in white blood cells in blood is ca. 2 μM, i.e., two orders of magnitude higher than for purified DNA. This is understandable since the proteins in blood bind several orders of magnitude more sulfur mustard than DNA. A detection limit of 2 μM sulfur mustard in blood is considered to be at the lower limit of toxicological relevance, since this is approximately the minimal concentration of the agent that inhibits proliferation of cells.

c. Detection of DNA adducts in human skin

Recently, experiments were performed to detect local DNA damage in skin samples. Pieces of human skin obtained from cosmetic surgery were exposed to air saturated with mustard vapor (at 30 °C, i.e., at a vapor concentration of 1260 $mg.m^{-3}$) for periods ranging from 0.25 to 10 min. The pieces of skin were then frozen to cut 5 μM slices, which were fixed on glass slides. Proteins and RNA were degraded enzymatically on the slide, and DNA was unwound. Subsequently, the preparation was treated with the monoclonal antibody against the N7-guanine adduct. Next, the antibody residing on the DNA adducts was allowed to bind to goat-anti-mouse antibodies. The latter antibodies contained a covalently attached fluorescent group emitting green light. The preparation was also treated with propidium iodide which intercalates with DNA and emits red light when properly irradiated. The coupes were analyzed under a laserscan fluorescence microscope. The red fluorescence from the propidium group was recorded which serves to locate exactly the nuclei of the epidermal cells in general. Scanning for the green light emitted by the fluorescent antibody locates the DNA that has reacted with sulfur mustard. In a slice of skin exposed for ≥ 1 min to sulfur mustard vapor it was observed that many of the nuclei of the epidermal cells showed this fluorescence. At this stage of the investigations the detection limit is at an exposure time of 0.5 min exposure, which amounts to a Ct-value of sulfur mustard (630 $mg.min.m^{-3}$) that would cause erythema, but not blisters, on human skin (12).

This preliminary result is considered as a valuable new development in the range of immunochemical detection techniques. Probably, the detection limit can be significantly improved.

d. Attempts to detect protein adducts

As described earlier, there are clearcut advantages in the use of protein adducts for retrospective detection of exposure to sulfur mustard. The adducts are longer-lived than those with DNA, which are usually removed by cellular repair systems. Moreover, ca. 10^3 x larger amounts of sulfur mustard are bound to protein than to DNA in human blood. Presumably, this is probably also the case in skin biopsies. On the other hand, the adducts with proteins are less defined and concentrated than those with DNA. It is difficult to find out which amino acid in proteins would form adducts preferentially with sulfur mustard (32). Therefore, it is also difficult to define which hapten should be synthesized for generation of antibodies.

In order to elucidate the structure of the products resulting from alkylation with sulfur mustard, simple model compounds (Figure 6) were synthesized of those amino acids

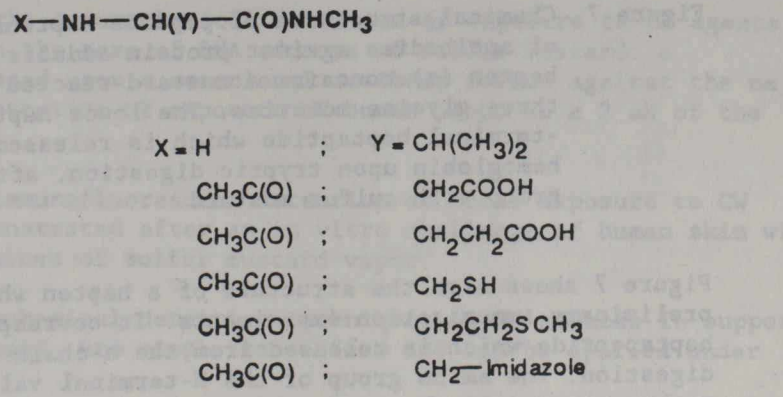
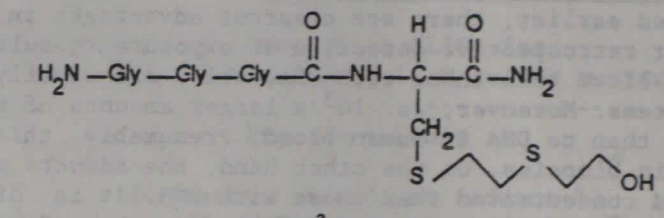


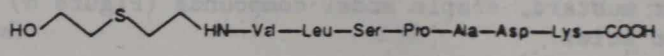
Figure 6 Chemical structures of model compounds used to elucidate the structures of sulfur mustard adducts with amino acids in proteins.

which are potential substrates for alkylation by sulfur mustard. Except for the α-amino group of valine (vide infra), the α-amino and α-carboxylic groups of the amino acids were acylated and amidated, respectively, as they are in proteins. The primary reaction products with sulfur mustard were identified by means of thermospray LC-MS. It was shown that the free carboxylic acid functions of glutamic and aspartic acid are alkylated, whereas cysteine and methionine react with mustard at the sulfur atom. Both ring nitrogens of histidine and the α-amino group of valine are alkylated.
A start was also made with the measurement of the relative rates of alkylation of the model peptides. One result of these measurements stands out quite clearly: the cysteine model compound reacts several orders of magnitude faster than the other model compounds. Since cysteine is reactive in various blood proteins and is also present in skin proteins, work is in progress on the

synthesis of a hapten (Figure 7), in which the cysteine adduct is connected to three glycines serving as a spacer for binding to the carrier protein.



a



b

Figure 7 Chemical structures of peptide haptens to be used for the generation of antibodies against protein adducts of sulfur mustard. The upper hapten (a) contains a mustard-reacted cystein coupled to a spacer of three glycine moieties. The lower hapten (b) represents the N -terminal heptaptide which is released from the α-chain of human hemoglobin upon tryptic digestion, after alkylation of the amino group with sulfur mustard.

Figure 7 shows also the structure of a hapten which was used already for preliminary immunization experiments. It corresponds with the amino-terminal heptapeptide which is released from the α-chain of hemoglobin upon tryptic digestion. The amino group of the N-terminal valine in this heptapeptide is rather exposed in the native structure of hemoglobin and, therefore, is alkylated in vivo by various agents, such as ethylene oxide (27). Investigation of the alkylation of the valine model compound (cf. Figure 6) in which the α-amino group was unprotected showed that an α-N-(2-hydroxyethylthioethyl) adduct was readily formed. This structure is present in the hapten derived from the heptapeptide, which was obtained by synthesis in a peptide synthesizer and subsequent N-alkylation. Recent investigations showed that the α-N-adduct of terminal valine is also formed upon in vitro incubation of hemoglobin with sulfur mustard, representing ca. 6% of the total alkylation of the protein. Since blood is a convenient ingredient to sample and since hemoglobin has a biological half life of several months, hemoglobin appeared to be a very suitable substance for biomonitoring. Therefore, it was attempted to obtain antibodies against this protein adduct. The alkylated heptapeptide was bound to a carrier protein and injected into mice. Several monoclonal antibodies were obtained having affinity for mustard adducts with amino acids. However, sofar they all belong to the IgM class, which is rather unsuitable for detection purposes. Efforts to obtain IgG antibodies are being continued.

4. CONCLUSIONS

- Since allegations for the illegal use of CW agents will be based primarily on the observation of injuries in supposed victims, unequivocal methods to verify such exposure in victims should be available in order to sustain the credibility of a Chemical Weapons Convention.

- Presently available methods for retrospective detection of exposure to CW agents are unsatisfactory since these lack specificity as well as sensitivity and are unsuitable when samples are taken at long time periods after the insult.

- Methods based on immunochemical or equivalent analyses of characteristic adducts of CW agents with DNA, proteins or other macromolecular constituents in the body should provide for the necessary specificity and sensitivity.

- Since the adducts of CW agents with macromolecules have life times of several days up to several months, the adducts can be detected over time periods which, based upon recent experience with CW incidents, are needed for retrospective detection of exposure.

- The feasibility of the immunochemical detection of exposure to CW agents has been demonstrated in the case of DNA adducts of sulfur mustard: a competitive ELISA based upon a monoclonal antibody raised against the major adduct allows the detection of exposure of human blood to ≥ 2 μM of the agent.

- The feasibility of immunofluorescent detection of local exposure to CW agents has been demonstrated after an in vitro challenge of human skin with non-blistering Ct-values of sulfur mustard vapor.

- In principle, immunochemical detection techniques of CW adducts in supposed victims, when developed, are simple to perform and can be applied under field conditions.

- Since the immunochemical detection methods can be quantified and used for dosimetry of exposure, these methods are also highly useful to monitor the internal dose of CW agents in persons involved in the destruction of stocks of these agents.

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CONFERENCE ON DISARMAMENT

CD/CW/WP.377
9 December 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

REPORT OF EXPERTS' MEETING ON THE DESTRUCTION OF CHEMICAL WEAPONS

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Presented by the Friend of the Chair
on Technical Matters (P. CANONNE)

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During the 1991 Session, the Ad Hoc Committee on Chemical Weapons decided to organise a meeting devoted to technical aspects of the destruction of chemical weapons (cf. CW/FCTM/PC/7/Rev.2 - 7 August 1991) and appointed Dr Jacobus OOMS, of The Netherlands, as the Chairman of this meeting. The meeting was held on 8 - 10 October 1991.

Background papers on different aspects of destruction were submitted and discussed. A list of the participants, a list of these papers and the agenda are included as an annex to this report.

A preliminary report was made orally by the Chairman to the Ad Hoc Committtee on 11 October 1991.

The results of the discussions are presented in this report, as viewed by the three rapporteurs (R. TRAPP, D. FROMENT, B. ODERNHEIMER) and by the Friend of the Chair.

I - GENERAL BACKGROUND - EXPERIENCES FROM PAST DESTRUCTION ACTIVITIES

An overview was presented on destruction technologies for chemical weapons (in bulk storage as well as in ammunition storage), and for former CW production facilities (1). Information was also provided on several specific CW destruction operations conducted in the past, and in particular on the destruction of mustard gas weapons in Britain, on the destruction of several types of chemical weapons at Rocky Mountain Arsenal in the USA (2) and on the destruction of BZ at Pine Bluff Arsenal, USA (3). During the discussions, further information was provided on destruction experiences from countries.

It soon became obvious in the presentations and the subsequent discussions that destroying chemical weapons stocks, and former production facilities as well, are tasks considerably more complex and costly than the original production activities were. This is due to a number of factors including, inter alia, occupational and environmental safety requirements. Among these factors, occupational safety considerations are paramount.

It is noted in this respect that although national standards are today available as guidance for risk assessments and the establishment of design criteria for destruction plants and operations, international harmonization of such standards would be should in the future be considered. It was argued that in setting standards, requirements should be kept within limits justified by scientific evidence rather than unrealistic "zero" settings. In this respect, chemical warfare agents do not differ from other hazardous material. Also, the "Best available technics not entailing excessive cost" and the "Best practical environmental option" principles now in use within the EEC, were mentioned as a good starting point for further consideration.

A number of technical options are, in principle, available for destruction programs. Yet, current legislation in a number of countries as well as public perception reduce the number of truly available options for destroying chemical weapons to basically two : chemical degradation and incineration.

For a number of reasons, more recent destruction activities on larger scales have favoured incineration. The advantages here are that smaller amounts of waste material are generated (about a third as compared to the destruction of a similar amount of chemical warfare agents by hydrolysis), that process control and stability are easier to achieve (better predictability of the reaction independent of the composition of tactical mixtures and the like), and that a higher throughput can be achieved. That does not exclude the use of chemical degradation techniques under other circumstances, and in fact decisions about the best approach to destroy a chemical weapons stockpile should perhaps be taken on a case by case basis taking into account the properties of the agent(s) and the ammunition(s), plant characteristics, and site characteristics.

Due account has to be given to proper safety régimes at a destruction site : air contamination monitoring, monitoring of exhaust gases (real time or near real time stack monitoring), waste analysis to rule out contamination with undestroyed agent, proper waste treatment and disposal are all important.

An issue hinted at yet often not considered in this context is the destruction of old CWA identification kits as these may contain active agents.

Time requirements for the destruction of chemical stocks are typically considerable. So are costs. One example more

thoroughly discussed was the destruction of BZ in the USA, an agent of lesser risk than nerve agents or mustard gas. Still, the overall operation from planning to completion including decontamination of the destruction plant itself took 14 years, for just about 700 agent tons. While, based on experience available today, several parts of chemical weapons destruction programs could in future be compressed, some cannot : time requirements for systemizing the plant and its equipment after construction will need, in a similar plant set-up, about 2 years. Training of personnel would also require about 6 months (but could be combined with the systemizing phase). While the operational phase could be shortened slightly by increasing plant availability, it has to be stressed that in the BZ destruction program a utilization coefficient of 68 per cent had already been achieved. Similar experiences are available from the JACADS operational tests, and other such operations.

As far as costs are concerned, the BZ destruction program was again used as an example. Its overall cost was said to have been around US-$ 163 million out of which the plant construction accounted for only 9 million, the equipment and its installation for 31 million, and 52 million were spent on operational costs.

The destruction of former CW production plants is an even more complex task, as for example the dismantling of the pilot nerve agent plant in Nancekuke, UK, has shown. Long-term contamination of parts of such plants will have to be anticipated even after thorough and multiple decontamination. It was also pointed out that under current legislation for example in the USA, conversion of parts of such a plant (as well as plants used for destroying chemical weapons) is not a viable option given the requirement to thoroughly decontaminate all such equipment at the end of the operation. This is most effectively done by thermal treatment which in turn renders the treated equipment unusable.

II - EXPERIENCES FROM ONGOING DESTRUCTION PROGRAMS

In Canada, a transportable incineration system is being used to destroy test nerve agents on the order of several hundreds of kilograms (4). Additionally, ten of tons of mustard gas and hydrolysate solution from previous decontamination operations, and considerably more left-over barrels, shells and the like had to be treated. The plant's design capacity though not fully achieved in reality was around 1.8 agent tons per hour. Given its transportability, the plant has been offered for future use outside Canada. The plant is operated by a total staff of 12 including lab support, air monitoring staff and administration.

Beside the technical lessons learned, a major conclusion of that operation was that public involvement is paramount. Not only are information and education of the public necessary to ascertain public acceptance, but the involvement of a voluntary citizen committee contributed to the high standard of the final destruction operation significantly.

Such public anxieties go well beyond the operation to destroy chemical weapons. It has to be very clear from the outset that the plant will not after completion of its task be converted to other tasks such as hazardous waste disposal unrelated to chemical weapons. While, in the Canadian case, technical design features were helpful to convince the public, in other countries such as the USA legal requirements have served the same end.

Under construction since 1985, the first full-scale CW destruction plant in operation, the USA's JACADS on Johnston Atoll, started test runs with live CW ammunition in June 1990 using the M55 GB rocket as the first sample (5). Further tests with M55 VX rockets and mustard gas projectiles are imminent. The technology used is ammunition-specific as far as disassembling is concerned, but incineration is the basic technical principle for decontaminating residual ammunition parts, and for destroying the chemical warfare agents themselves. Further tests for different ammunition types are under way and were described. In real life, the test runs demonstrated an ability to destroy about 13 rockets per hour (on average) - less than the original goal of 24 rockets per hour. Further tests and plant modifications are expected to increase that figure further. As for the liquid incinerator used to destroy the actual agent, the bottleneck was not the waste treatment system but the incinerator unit itself, due to the nature of the items to be treated.

The JACADS concept was described to be the technological basis for other, still to be built US destruction plants. Its overall cost was about US-$ 811 million.

At present, it is difficult to give precise figures on costs of destruction per CW agent tonnes. Canadian experience, although gathered in a slightly different context, amounts to about US-$ 4000 per tonne.

As a concept emerging from use in the civilian field (destruction of hazardous wastes), controlled explosion of CW ammunition in an environmentally sealed firing pool (aqueous solution with hydrolysing properties) was suggested as an alternative destruction techniques (6). In principle, several environmental limitations relevant for incineration process such as exhaust gas treatment could thus be overcome in a cost-effective manner. So far, that technology has been tested and in fact used with non-CW hazardous waste materials in France. Feasibility studies for destroying chemical weapons are under way. Assuming that these would turn out positively, this technology might under certain circumstances provide a cost-effective alternative to incineration.

The main technological aspects of the destruction of chemical weapons were also clearly summarized (7).

In USSR, the KUASI complex is designed for the destruction in the field conditions of faulty chemical munitions

containing no explosives (8). It provides safe destruction with minimal pollution of the environment, in accordance with the standards effective in the USSR. KUASI technology is based on the method of chemical agent detoxication by suitable reagents followed by incineration of produced reaction masses. This system was used for the destruction of more than 4000 munitions of various calibres, filled with sarin, soman and VX, in the period of 1980 - 1990.

III - DESTRUCTION OF OLD/OBSOLETE STOCKS

If the destruction of military stockpiles of CW is a difficult one, from contributions by Germany, Italy and Belgium, it became clear that the destruction of old/obsolete stocks causes particularly difficult problems. This is mainly caused by a number of factors particular to these "weapons" :

- it is sometimes very difficult to differentiate between chemical munitions and conventional ones, due to their very bad state, in particular external corrosion

- for the same reason, it is sometimes difficult to identify the precise origin of CW

- the explosive charges are sometimes extremely difficult, and in some cases impossible to remove, the priming cap being sometimes active

- the chemical agents come in an enormous variety, sometimes in very complex mixtures (in particular agents and explosives) ; in Belgium alone, at least 50 different agents were identified (HD, HN1, HN2, L1, L2, L3, phosgene, phenyl dichloroarsine, etc.)

- the variety and the presence of arsenicals in different forms complicate the destruction process, and large amounts of toxics end products are formed

- findings of munitions are completely unpredictable, so planning destruction processes is hardly possible

- each munition has to be handled individually and extremely carefully which does make the destruction program necessarily a lengthy one.

A great deal of study still has to be carried out, but, despite these difficulties, the solutions presented at the Seminar are certainly impressive.

In Germany, a large variety of CW munitions, accidently or systematically recovered in the past four decades, require individual assessment by explosives ordnance disposal personnel of their condition prior to destruction. After removal, transportation and intermediate storage, in which a number of stringent standards are met, the munitions have to be cleaned and details are

determined by X-raying each item. Then the demilitarization process is achieved, with very high level protective methodologies. The final step of the destruction includes incineration at high temperatures. The combustion flue gases are scrubbed, while the aqueous solution of mineralized arsenic (III) compounds is subjected to oxidation and subsequent arsenic precipitation. The resulting cakes containing arsenic are finally deposited in an old underground salt mine. The incineration plant is designed for a destruction capacity of approximately 70 agent tons per year (cf. CD/1026 - 3 August 1990). An additional plant is currently being planned for the disposal of both soil contaminated with arsenicals and explosives mixed with toxic chemicals.

In Italy (10), the old CW, particularly those composed of a mixture of mustard and phenyldichloro-arsine, are processed in a liquid phase by oxidation by hydrogen peroxide, followed by a neutralisation by lime and then cementation of the reaction products and preservation in safety of the concrete thus obtained. The cost of the destruction plant was US-$ 2,1 million. The operating costs per year, including personnel, amount approximately US-$ 370 000. Two further plants are in phase of planning, in order to cope with new problems related to the discovery of old and obsolete rounds and an significant amount of adamsite.

In Belgium (11), an installation to dismantle problem munitions from the First World War is in its final phase and a dismantling scheme has been settled. For the time being, the following steps are used : transportation in wooden cases, initial cleaning with high-pressure water, sorting of the munitions in two groups (explosive non toxic or munitions non positively identified), storage by type and caliber. The neutralized chemical products are stored as necessary and will be destroyed later, with contaminated materials. In the near future, dismantling is planned to separate shells into three parts : the contents, the detonator and/or explosive charge, and the metal casing.

IV - SAFETY AND ENVIRONMENTAL ISSUES

All countries involved in destroying CW have taken great care to ensure the safety of both the personnel operating the facilities and of the public living or working in the vicinity of the installations. Health monitoring of destruction personnel on a regular basis is a vital task. Psychological stress factors are also taken into account. Currently long-term health monitoring of personnel involved in such operations, after conlusion of the tasks, is usually not required. Yet, medical records are typically kept for several decades.

Some medical aspects of CW destruction were presented (12) : necessity of medical service within the destruction facility area, prophylaxis based on administration of reversible

cholinesterase inhibitors, therapy with parasympatholytics ; there are developing perspectives of the treatment or prophylaxis of intoxications with cholinesterase inhibitors (hemodialysis, hemoperfusion, administration of antibodies against organophosphates, new types of antidotes containing both parasympatholytic and reactivator structures, new approaches to pharmaceutical procedures of administration, etc.).

In the United States, the safety and environmental requirements for CW destruction facilities (13) must comply with all existing national environmental and safety regulations/ standards.

The general design criteria which US chemical demilitarization facilities must satisfy are the following : no uncontrolled emissions, no process liquid discharges, continuous agent monitoring, personnel interfaces minimized, performance of hazard risk analysis and mitigation. The destruction of CW is regulated by several environmental regulations : National Environmental Policy Act, Resource Conservation and Recovery Act, Toxic Substances Control Act, Clean Air Act and State Act Quality Regulations.

Approaches were also proposed by USSR (14) concerning the safety of people and the environment, with emphasis of the need for the fullest possible exchange of information with specialists on key matters in CW destruction.

From most of the past and existing destruction programs, there is one aspect however that is of paramount importance : the involvement of the public from the very start of the process of destruction, in order to allay its worries as regards the setting-up of the destruction facility and the subsequent environmental impact. Dialogues and information exchange are necessary to the full acceptance.

V - NEW AND EMERGING TECHNOLOGIES

Several new and emerging technologies were mentioned, which are being studied and evaluated as candidate approaches to CW destruction in various countries and which include enzymatic degradation, adiabatic compression and plasma technologies. Experiences gained thus far with respect to specific chemicals were reported.

It was concluded that some of the new approaches, most of which are primarily aimed at decontamination and environmental purposes rather than large-scale CW destruction, could become very important in the future. Therefore, research in these areas should be encouraged. Typical applications would include toxic waste treatment and decontamination of soil, harbour sludges and water.

In the specific CW context, however, new and emerging technologies were generally assessed to be in the early stages and require further extensive studying and testing. It became evident

that, given the timeframe under present consideration for CW destruction, the number of technologies readily available would be very limited. The destruction of CW stockpiles will mainly be based on the well-established and proven technologies of chemical degradation and incineration.

It was noted that for the destruction of the various types of CW different technologies could be appropriate. Detailed knowledge of the specific problems involved would be crucial in finding the technological response appropriate in any particular case.

Irrespective of the destruction technology adopted, the planning, the construction of facilities and the destruction itself were perceived as highly complex and costly tasks which require long leadtimes.

VI - VERIFICATION OF CHEMICAL WEAPONS DESTRUCTION

The determination of when the destruction of a CW can be considered as complete (or irreversible in practical terms) was identified as an important subject of further discussion in the CD. In this context, encasement in concrete blocks or other matrices was mentioned.

As far as technical verification requirements under the future chemical weapons Convention are concerned, little if any problems seem to exist. Even for destruction operations designed in the past, without regard to such requirements, an overlay of verification would not have caused major alterations in the design. However, it was considered very useful if, in the planning of destruction facilities, aspects of future verification could already be taken into account.

It was also concluded that the respective provisions of the rolling text have been sufficiently elaborated. To ensure the integrity of declared stockpiles, seals and measuring instruments could be installed and extensive use of records made during inspections.

One problem connected with sample-taking at CW storage sites on the request of inspectors, as provided for in the rolling text, was identified by the United States. It was suggested that the respective provisions be harmonized with national law. While U.S. environmental regulations preclude any release of toxic chemicals from sampling, individual States have juridiction over stringent safety standards. Sampling would only be feasible at certified destruction facilities.

As a consequence, containment, sealing and tagging of munitions selected by inspectors are required prior to transport to a destruction facility. Ongoing studies on non-destructive identification techniques necessary for the assessment of recovered old CW, were thought to be of great importance in this context.

In the case of the verification system for closed CW production facilities, it was concluded that the provision in the rolling text for the continuous presence of inspectors was too extreme and probably unnecessary if critical equipment items have been removed and seals applied. A number of annual visits on short notice was suggested to be more cost-effective. In addition, remote monitoring via telephone lines could be applied.

Concerning the safety of inspectors, it was argued that the individual requirements would vary considerably due to the specifics of facilities. As experience has shown, certain parts of infrastructure, such as ventilation systems, must be maintained in operable condition. It was stressed that some general guidelines on safety requirements were needed.

VII - INTERNATIONAL COOPERATION

International cooperation was considered by all participants to be of particular importance. A number of participants expressed the willingness of their countries to share with other countries the expertise and experience they have gained.

It was noted that some countries have already cooperated closely and successfully in the past. This cooperation was offered to be extended to other countries and could include the exchange of technological know-how and experience, the provision of blueprints and special technical advice, invitations to visit existing installations and assistance in the overall planning of CW destruction activities. In addition, some countries offered to provide equipment ranging from complete facilities to system components, such as special safety equipment, machinery, robots and the like. Furthermore, training of personnel was thought to be an important area of useful cooperation.

It is also interesting to notice that there was in the past a bilateral cooperation between Indonesia and The Netherlands in the destruction of chemical weapons stocks (see CD/270 -21 March 1982).

VIII - FOLLOW-UP OF THE SEMINAR

The possibilities of a follow-up of the seminar are twofold :

1) There was at the end of the meeting a general feeling that it might be useful if another meeting could be organized. Several topics were mentionned, for instance :

- the need for another look at the technical problems of old and obsolete stocks
- assessment of new and emerging technologies of destruction

- safety of international inspectorate, emergency procedures
- information and involvement of the public
- verification aspects
- means for international cooperation.

2) From the overall meeting, it appears that further discussions are needed as regard some provisions in the rolling text (articles IV, V and annexes) :

- sample taking at CW storage sites
- verification system for closed CW production facilities
- determination of the completion of the destruction of CW (irreversibility, inoperability)
- international cooperation and assistance.

IX - CONCLUDING REMARKS

A major conclusion of the meeting is that destruction of chemical weapons stocks is a complex issue with regard its technological, financial, legal, environmental and safety aspects, and the long lead times involved. Given that the negotiations of the Convention are in the final stage, it is imperative that all countries who have to carry out destruction activities now consider and plan how to meet time requirements of destroying their CW stocks and their former CW production facilities.

There was strong support for an appeal to countries to declare, as early as possible, the types and quantities of CW stockpiles they possess or to specify other related problems they are faced with, including old obsolete CW.

As far as bilateral or multilateral cooperation is concerned, it appeared clearly that countries involved in destruction activities are ready to share their experience with other countries also faced with problems of destruction.

A N N E X

LIST OF CONTRIBUTIONS

-:-:-:-:-

(1) Dr Ron MANLEY (UK) : "Destruction of CW stocks, weapons and associated plant" (see CD/CW/WP.373 - 21 October 1991)

(2) Dr Kevin FLAMM (USA) : "US chemical weapons destruction experience at Rocky Mountain Arsenal"

(3) Dr F. JENNINGS (USA) : "US disposal experience at Pine Bluff Arsenal"

(4) Dr J. McANDLESS (CANADA) : "Destruction of chemical agent waste at Defence Research Establishment Suffield"

(5) Dr R. MISIEWCZ (USA) : "Johnston Atoll chemical agent disposal system (JACADS)"

(6) Mr M. BISEAU (FRANCE) : "Preliminary technical study for a new destruction process"

(7) M. G. LEONOV (USSR) : "Main technological aspects of the destruction of chemical weapons" (see CD/CW/WP.367 - 7 October 1991)

(8) Mr V. SHELUCHENKO (USSR) : "Complex for the destruction of faulty chemical munitions" (see CD/CW/MP.369 - 8 October 1991)

(9) Dr M. MARTENS (GERMANY) : "Old chemical weapons disposal (see CD/CW/WP.374 - 31 October 1991)

(10) Lt Cl DI CARLO (ITALY) : "Italian experience about the destruction of old and obsolete chemical weapons" (see CD/CW/WP.375 - 20 November 1991)

(11) Captain M. WOUTERS (BELGIUM) : "Old and obsolete chemical weapons : Technical problems of dismantling"

(12) Dr J. BAJGAR (CZECH AND SLOVAK FEDERAL REPUBLIC) : "Some medical aspects of chemical weapons destruction"

(13) Dr K. FLAMM (USA) : "US chemical weapons and destruction : safety and environmental requirements"

(14) Mr G. LEONOV (USSR) : "Environmental aspects of the destruction of chemical weapons" (see CD/CW/WP.368 - 7 October 1991)

8 October 1991

EXPERTS MEETING ON TECHNICAL ASPECTS OF CW AGENT DESTRUCTION

Agenda

TUESDAY 8 October 1991

10.00 - 13.00 hours	General Background Past Experience
15.00 - 18.00 hours	Ongoing Activities (including costs, time requirements, technical aspects of verification)

WEDNESDAY 9 October 1991

10.00 - 13.00 hours	Old/Abandoned Stocks (including emergency operations) Arsenicals
15.00 - 18.00 hours	Safety and Environmental Issues

THURSDAY 10 October 1991

10.00 - 13.00 hours	New and Emerging Technologies (including availability, costs and time requirements)
15.00 - 18.00 hours	Verification International Cooperation

FRIDAY 11 October 1991

10.00 hours (Room V)	Chairman's report to the Ad-Hoc Committee on Chemical Weapons

EXPERTS' MEETING ON THE DESTRUCTION OF CHEMICAL WEAPONS

Provisional List of Participants

AUSTRIA (Observer Delegation)

Prof. Dr. Helmut HONIG	Professor of Organic Chemistry Graz University of Technology
Dipl.Ing. Josef STOLZ	Chemical Laboratory Austrian Army

AUSTRALIA

Mr. Gordon ECKERSLEY	CD Delegation.

BELGIUM

Mr. Marc WOUTERS	Technical Department of the Army (Vilvoorde).

CANADA

Dr. John McANDLESS	Defence Research Establishment Suffield
Dr. M.C. HAMBLIN	CD Delegation.

CHINA

Mr. Zhongzhou YU	Ministry of National Defence.
Mr. Zang YAN	CD Delegation.

CZECH AND SLOVAK FED.REP.

Dr. Jirí BAJGAR	CD Delegation.

FINLAND (Observer Delegation)

Mr. Aapo Pölhö	Observer delegation to the CD.

FRANCE

Mlle. Corinne BANON	SNPE Ingenierie
M. Hervé BIZEAU	SNPE Ingenierie
M. Pierre CANONNE	CD Delegation.
M. Daniel FROMENT	Ministry of Defence.
M. Bernard TRAVAILLOT	Ministry of Defence.

GERMANY

Col. Klaus ARNHOLD	CD Delegation.
Dr. Stephan BECKER	Lurgi (Frankfurt)
Dr. Hermann MARTENS	WWD (Munster).
Dr. Harald RAMDOHR	Uhde (Dortmund)
Dr. Bernhard ODERNHEIMER	CD Delegation.
Mr. Hermann STEINKAMP	Lurgi (Frankfurt)
Dr. Ralf TRAPP	Pugwash (Observer).
Dr. Hanss-Nicol WERNER	MAN
Mr. Rolf Mafael	CD Delegation.

INDONESIA

Mr. Baginda SIMANDJUNTAK	Ministry of Defence.
Mr. Iskandar HADRIANTO	Permanent Mission of Indonesia, Geneva.

IRAQ (Observer Delegation)

Dr. Zuhair S. SALIH	Professor of Chemistry College of Science Baghdad University, Iraq.
Dr. Adil AL TAI	Professor of Organic Chemistry College of Science Baghdad University, Iraq.

ITALY

Major Giacinto COSTANTINO	NBC Establishment.
Lt.Col. Roberto DI CARLO	CD Delegation (CW Expert).

JAPAN

Mr. Mikio ISHIWATARI	CD Delegation.
LTC. Hitoshi KAWAGUCHI	Joint Staff Office, Defence Agency of Japan.
Mr. Takuji HANATANI	CD Delegation.

NETHERLANDS

Mr. Arend MEERBURG	CD Delegation.
Dr. Jack OOMS	Adviser to CD Delegation. (Chairman of the Meeting)

POLAND

Mr.Sylwin Gizowski	CD Delegation.

SPAIN (Observer Delegation)

Mr. Felix GONZALEZ	Defence Adviser Observer delegation to the CD.

SWEDEN

Mr. Richard EKWALL	CD Delegation.
Dr. P. RUNN	National Defence Research Establishment
Dr. Johan SANTESSON	National Defence Research Establishment

SWITZERLAND (Observer Delegation)

Mr. A. FREY	Observer Delegation to the CD.
Mr. René HAUG	Observer Delegation to the CD.

UNION OF SOVIET SOCIALIST REPUBLICS

Mr. Rifkat CHEBOTAREVSKY	Supreme Soviet of Russia.
Mr. Georgy LEONOV	Chemical Industry.
Mr. Gennady LUTAY	CD Delegation.
Mr. Vladimir ROMANCHUK	State Military-Industry Commission.
Mr. Vladislav SHELUCHENKO	Chemical Industry.
Mr. Oleg UKHAROV	CD Delegation.

UNITED KINGDOM OF GREAT BRITAIN AND NORTHERN IRELAND

Mr. Ron MANLEY	CBDE Porton.
Dr. Graham COOPER	CD Delegation.

UNITED STATES OF AMERICA

Dr. Richard D'ANDREA	Arms Control and Disarmament Agency.
Dr. Kevin FLAMM	P.M. for Chemical Demilitarization.
Dr. Floyd W. JENNINGS	P.M. for Chemical Demilitarization.
Lt.Col. William J. MILLER	Department of Defence.
Dr. Robert MIKULAK	Arms Control and Disarmament Agency.
Dr. Richard MISIEWICZ	P.M. for Chemical Demilitarization.
Col. Guy LOVELACE	Department of Defence.
Col. David LAMBERT	Office of the Joint Chiefs of Staff.
Cpt. Bruce C. FOREMAN	Office of the Joint Chiefs of Staff.

CONFERENCE ON DISARMAMENT

CD/CW/WP.378
16 December 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

FINLAND AND THE NETHERLANDS

Working Paper

Accreditation of Verification Laboratories

1. Introduction

The Chemical Weapons Convention (CWC) will require accredited laboratories to carry out verification analyses for the Technical Secretariat (TS).

In addition to verification analyses, the following regular tasks have to be performed by such laboratories (see also Working Paper CD/CW/WP.342 by Finland and the Netherlands):

- analytical chemical method development,
- technical support to the Organization,
- training of inspectors in analytical chemical tasks,
- synthesis of reference compounds, and
- recording analytical data for the verification database.

The Preparatory Commission has to begin the creation of a laboratory network so that it will be operational at the entry into force of the CWC. (Pending the establishment of a fully fledged Technical Secretariat, it may consider to use existing inspection bodies to start the accreditation process.) The objective of this working paper is to describe in general terms the requirements a laboratory must fulfil in order to receive accreditation.

2. Application for accreditation

When laboratories apply for accreditation, the following aspects of laboratory operation would be considered by the Technical Secretariat:

- adherence to the international principles of Good Laboratory Practice (GLP) applicable to laboratories with testing and research facilities, including an established quality assurance program;
- adherence to norms established under the CW Convention for the specific analytical chemical and physical verification processes, such as:
  a. availability of qualified personnel trained to work with highly toxic materials and following regulations for their safe handling;
  b. laboratory facilities allowing enough space for all operations and designed for work with highly toxic chemicals; and
  c. instrumentation including major chromatographic and spectrometric analysis techniques (CD/CW/WP.272 and CD/CW/WP.306) and qualified personnel to operate them.

3. Principles of accreditation

The Technical Secretariat (TS) will have to ensure that the requirements mentioned above are fulfilled before accreditation. The TS could use a three-step procedure for accreditation by dispatching to the laboratory:

1) a detailed questionnaire,
2) a group of assessors to inspect the laboratory, and
3) proficiency samples for analysis.

The questionnaire (see e.g. ISO-IEC Guide 39) will aid assessment of the laboratory and provide detailed documentation of its capabilities. After evaluation of the questionnaire, the TS will send a team of "inspectors" (assessors) to visit the laboratory to evaluate it and to

uncover possible deficiences. The most important part of testing is, however, periodic analyses of proficiency samples. These tests could be organized in the form of international interlaboratory comparison tests. In these tests the laboratories seeking accreditation must be able to identify compounds of interest to the CWC unambiguously (CD/CW/WP.308).

After accreditation, continuous testing of laboratories is required. This will be done by periodic audits (e.g. every two years) of the laboratories, and either by organizing new interlaboratory comparison tests or by adding proficiency samples among the genuine samples each time samples are sent for analysis to a particular laboratory. The latter procedure would allow checking of the capability of the laboratory each time its services are used.

4. Quality Assurance System

The vital element for the accreditation of the laboratories is a Quality Assurance System. Additional elements include the possession of authentic, fully-validated reference samples, and a validated analytical database. The reference samples and the database could be provided to the laboratories by the Technical Secretariat.
The key element - i.e. the Quality Assurance System appropriate to the types of functions performed for the Technical Secretariat (analytical, synthesis, research, training, etc.) - has to be created by the laboratories themselves. The Quality Assurance System should be laid down in a Quality Manual.

This Manual should cover the following aspects for each laboratory:

- declaration of intent for quality assurance management;
- data about the organization and personnel;
- description of the pretensions of the laboratory;
- description of the premises;
- functioning of the internal auditing system;
- description of the functioning to manage externally

supplied materials and services;
- management and use of technical equipment as well as reference materials;
- compilation of operating (analytical and management) procedures;
- description of the performance of the test experiments and the research study;
- management of the reporting procedures and experimental results;
- description of archives to keep records and documents, including confidentiality and security aspects;
- managing up-dating of the Quality Manual

Typical contents of a Quality Manual for testing laboratories are described in e.g. "Handbook of Quality Assurance for the Analytical Chemistry Laboratory" by James P. Dux and in "The Laboratory Quality Assurance System" by T.A. Ratcliff. General criteria for the technical competence of testing laboratories have been described e.g. by the joint European Standards Institution in EN 45001. In case of the implementation of research and specialized verification activities, the modifications might concern the technical competence of the management and organization. The new version of ISO-IEC Guide 25 covering both test and research laboratories might be a good reference.

The main aspects applicable especially to non-repeating study-protocols are the following:
- the nomination of a study director and co-workers;
- description of the jobs, responsibilities, and authorization personnel;
- advance description of the protocol for the fulfilment of the study;
- essential information in the protocol on the scope of the study, the methods and techniques to be possibly used, and the character and identification of the samples to be analyzed.

5. Assessment of laboratories

The Technical Secretariat will have to establish its own rules of procedure for accrediting the laboratories and for the continuous assessment of their operations. It may use as guidelines those kinds of general criteria as published e.g. by the joint European Standards Institution (EN 45002 and EN 45012). The language of these standards are geared to accreditation of test laboratories, but the content can be considered equally applicable to the assessment of research laboratories.

6. Advantages of accreditation

The accreditation program will assure the Technical Secretariat of some essential facts:

- the laboratory functions in conformity with a total Quality Assurance System laid down in the Quality Manual;
- the laboratory has demonstrated the existence and the consistence of such a Quality Assurance System;
- the laboratory performs the analytical chemical and physical procedures using validated and approved methods;
- the laboratory reports exactly the results and methods used; and
- the laboratory has qualified personnel.

If the Technical Secretariat will validate and accept Recommended Operating Procedures (ROP's) for sampling and analysis as the first candidate methods for sampling and analysis of verification samples, consistent results should be obtained after collaborative testing of the ROP's by all accredited laboratories. Continuous method development could be encouraged by accepting methods developed by the laboratories as recommended procedures promptly after their international testing.

7. References

J.A. Dux, Handbook of Quality Assurance for the Analytical Chemistry Laboratory, Van Nostrand Reinhold, New York, 2nd Ed., 1991.

T.A. Ratcliff Jr, The Laboratory Quality Assurance System, Van Nostrand Reinhold, New York, 1990.

EN 45001 General criteria for the operation of testing laboratories

EN 45002 General criteria for the assessment of testing laboratories

EN 45012 General criteria for certification bodies operating Quality System certification

The joint European Standards Institution, Brussels.

ISO-EIC Guide 25 General requirements for the technical competence of testing laboratories

ISO-IEC Guide 39 General requirements for the acceptance of inspection bodies

International Organisation for Standardization, Geneva.

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