CONFERENCE ON DISARMAMENT

CHEMICAL WEAPONS

WORKING PAPERS OF THE

Ad Hoc COMMITTEE ON CHEMICAL WEAPONS 1990

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EXTERNAL AFFAIRS AND INTERNATIONAL TRADE CANADA

OTTAWA, CANADA

FEBRUARY 1991

CONFERENCE ON DISARMAMENT

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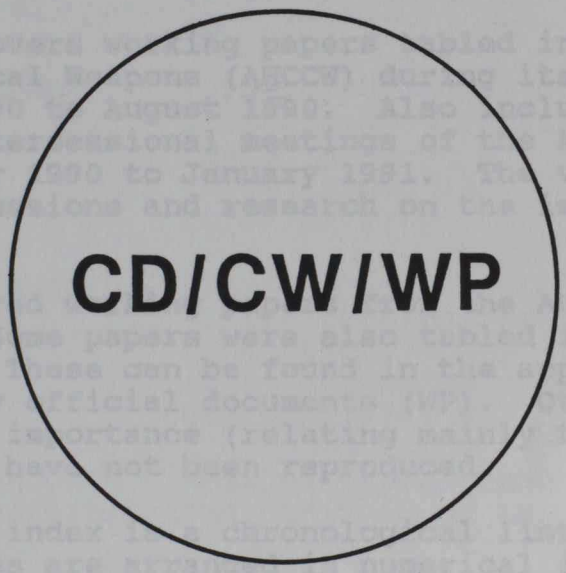

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ARMS CONTROL AND DISARMAMENT DIVISION OF

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OTTAWA, CANADA

FEBRUARY 1991

PREFACE

CD/CW/WP

This volume covers working papers tabled in the Ad Hoc Committee on Chemical Weapons (AHCCW) during its 1990 sessions from 30 January 1990 to August 1990. Also included are working papers from the intersessional meetings of the AHCCW which took place from November 1990 to January 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 1990
Chronological Index

1990

Serial	Reference	Country	Description	Date
460.1	CD/CW/ WP.273	AHCCW	Draft report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament (Not Reproduced)	30.1.90
462	CD/960	France	Second national trial inspection (also issued as CD/CW/WP.274)	1.2.90
464	CD/966	USSR	Experimental challenge inspection at a military installation (also issued as CD/CW/WP.275)	14.2.90
466	CD/969	Hungary	Provision of data relevant to the Chemical Weapons Convention (also issued as CD/CW/WP.277)	19.2.90
466.1	CD/CW/ WP.276	AHCCW Chairman	Working paper presented by the Chairman of the Ad Hoc Committee: 'Organization of work for the 1990 session' (Not Reproduced)	19.2.90
472	CD/975	FRG	Report on a trial challenge inspection (also issued as CD/CW/WP.278)	9.3.90
472.1	CD/CW/ WP.279	Canada	Thiodiglycol	15.3.90
472.2	CD/CW/ WP.280	Sweden	Provision of data relevant to the Chemical Weapons Convention	16.3.90

Serial	Reference	Country	Description	Date
472.3	CD/CW/ WP.281	Japan	Provision of data relevant to the Chemical Weapons Convention	16.3.90
472.4	CD/CW/ WP.282	Western Group	Technical Support for the Chairman of the Ad Hoc Committee	16.3.90
475	CD/983	FRG	Report on the second trial inspection (challenge inspection) in the Federal Republic of Germany (also issued as CD/CW/WP.283)	5.4.90
476	CD/984	FRG	Ad Hoc verification: the establishment of national registers (also issued as CD/CW/WP.284)	10.4.90
476.1	CD/CW/ WP.285	Norway	Report on a national trial inspection of an industrial chemical facility	10.4.90
476.2	CD/CW/ WP.286	Australia	Ad hoc verification: discussion paper	11.4.90
476.3	CD/CW/ WP.287	Italy	Production capacity	11.4.90
476.4	CD/CW/ WP.288	Australia Canada Finland France FRG Nether-lands Norway Switzer-land UK	International Interlaboratory Comparison (Round Robin) Test	11.4.90
477	CD/985	Poland	Provision of data relevant to the Chemical Weapons Convention (also issued as CD/CW/WP.289)	17.4.90

Serial	Reference	Country	Description	Date
478	CD/987	Canada	National trial inspection at a single small-scale facility (also issued as CD/CW/WP.290)	19.4.90
479	CD/988	India	Letter dated 19 April 1990 from the Permanent Mission of India addressed to the Secretary-General of the Conference on Disarmament transmitting a document entitled 'Report of the national trial inspection conducted by India' (also issued as CD/CW/WP.291)	20.4.90
485	CD/996	GDR	Report on a trial challenge inspection in a chemical industry plant (also issued as CD/CW/WP.292)	12.6.90
486	CD/997	GDR	Inspection methodology for challenge inspections in industrial chemical plants (also issued as CD/CW/WP.293)	12.6.90
487	CD/998	GDR	Application of trace analysis to exploit memory effects in challenge inspections (also issued as CD/CW/WP.294)	12.6.90
488	CD/999	Austria	Report on a national trial inspection (also issued as CD/CW/WP.295)	12.6.90
490.1	CD/CW/ WP.296	UK	Addition of chemicals to the schedules	18.6.90
490.2	CD/CW/ WP.297	Finland	Provision of data relevant to the Chemical Weapons Convention	20.6.90

Serial	Reference	Country	Description	Date
492	CD/1008	Norway	Use of sorbent extraction in verification of alleged use of chemical weapons (also issued as CD/CW/WP.298)	26.6.90
	CD/CW/ WP.299		Withdrawn	
492.1	CD/CW/ WP.300	USA	Revisions to Article VI, Permitted Activities	27.6.90
492.2	CD/CW/ WP.301	USA	Report on the second United States trial inspection exercise	27.6.90
492.3	CD/CW/ WP.302	Nether-lands	Analytical chemical results of the second trial inspection on verification of non-production of chemical warfare agents in a civil chemical industry in the Netherlands	28.6.90
492.4	CD/CW/ WP.303	USSR USA	Proposed revisions to the rolling text	28.6.90
494	CD/1012	UK	Verification of the Chemical Weapons Convention: Practice challenge inspections of government facilities: Analysis of results (also issued as CD/CW/WP.304)	11.7.90
495	CD/1014/ Rev.1	Romania	Data relevant to the Chemical Weapons Convention (also issued as CD/CW/WP.305/Rev.1)	16.7.90
495.1	CD/CW/ WP.306	AHCCW	Report of the Technical Group on Instrumentation	17.7.90
497	CD/1018	Nether-lands	Report on a trial challenge inspection (also issued as CD/CW/WP.307)	19.7.90

Serial	Reference	Country	Description	Date
497.1	CD/CW/ WP.308	Nether-lands	Criteria for confirmation of chemical warfare agents identification	19.7.90
498.1	CD/CW/ WP.309	Switzer-land	National trial inspection (documents and annexes to CD/CW/WP.247)	25.7.90
499	CD/1020	GDR	Report on a trial challenge inspection (also issued as CD/CW/WP.310)	26.7.90
500	CD/1021	Czecho-slovakia	Report on a trial challenge inspection at a chemical facility (also issued as (CD/CW/WP.311)	26.7.90
501	CD/1022	Czecho-slovakia	Report on a trial challenge inspection at a military facility (also issued as CD/CW/WP.312)	26.7.90
503	CD/1024	Peru	New article of a convention on chemical weapons relating to the environment (also issued as CD/CW/WP.313)	31.7.90
504	CD/1025	Peru	Proposal for the inclusion in the Chemical Weapons Convention of an Article on 'Duration' (also issued as CD/CW/WP.314)	31.7.90
505	CD/1026	FRG	Chemical Weapons Verification Workshop, Munster, 14-15 June 1990 (also issued as CD/CW/WP.315)	3.8.90
505.1	CD/CW/ WP.316	AHCCW Chairman	Chairman's summary of the 1990 open-ended consultations on Article IX	6.8.90

Serial	Reference	Country	Description	Date
505.2	CD/CW/ WP.317	AHCCW	Draft report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament (Not Reproduced)	6.8.90
506	CD/1029	France	Report on a trial challenge inspection (also issued as CD/CW/WP.318)	8.8.90
507	CD/1030/ Rev.1	Canada	Report on a national trial inspection (also issued as CD/CW/WP.319/Rev.1)	8.8.90
508	CD/1031	PRC	Fundamental position and propositions on challenge inspection (also issued as CD/CW/WP.320)	10.8.90
510	CD/1040	Iran	National trial inspection (also issued as CD/CW/WP.321)	31.8.90
511	CD/1042	Chile	Multilateral exchange of data relevant to the Chemical Weapons Convention (also issued as CD/CW/WP.321)	3.12.90
511.1	CD/CW/ WP.323	AHCCW	Editing of the Draft Convention	10.1.91
511.2	CD/CW/ WP.324	AHCCW	Chairman's Paper: Article X: Assistance and protection against chemical weapons	10.1.91
511.3	CD/CW/ WP.325	AHCCW	Draft report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament on its work during the period 8-18 January 1991 (Not Reproduced)	17.1.91

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
460.1	CD/CW/ WP.273	AHCCW	Draft report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament (Not Reproduced)	30.1.90
466.1	CD/CW/ WP.276	AHCCW Chairman	Working paper presented by the Chairman of the Ad Hoc Committee: 'Organization of work for the 1990 session' (Not Reproduced)	19.2.90
505.2	CD/CW/ WP.317	AHCCW	Draft report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament (Not Reproduced)	6.8.90
511.3	CD/CW/ WP.325	AHCCW	Draft report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament on its work during the period 8-18 January 1991 (Not Reproduced)	17.1.91

AHCCW	CD/CW/WP.273	Draft report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament NOT REPRODUCED	30.1.90
		* * *	
France	CD/CW/WP.274	Second national trial inspection NOT REPRODUCED (see WP volume)	Also issued as CD/960 1 Feb. 90
		* * *	
USSR	CD/CW/WP.275	Experimental challenge inspection at a military installation NOT REPRODUCED (see WP volume)	Also issued as CD/966 14 Feb. 90
		* * *	
	CD/CW/WP.276	Working paper presented by the Chairman of the Ad Hoc Committee: Organization of Work for the 1990 session NOT REPRODUCED	19.2.90
		* * *	
Hungary	CD/CW/WP.277	Provision of data relevant to the Chemical Weapons Convention NOT REPRODUCED (see WP volume)	Also issued as CD/969 19 Feb. 90
		* * *	
FRG	CD/CW/WP.278	Report on a trial challenge inspection NOT REPRODUCED (see WP volume)	Also issued as CD/975 9 Mar. 90

CONFERENCE ON DISARMAMENT

CD/CW/WP.279 */
16 March 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

CANADA

Thiodiglycol

1. Considerable attention is being given to Article VI and its related schedules of chemicals. Two earlier working papers, CD/CW/WP.231 and CD/CW/WP.259, developed a theme concerning criteria other than toxicity for the determination of whether a chemical should appear on a particular schedule. In CD/CW/WP.259, pinacolyl alcohol was used as an example to illustrate the difficulties faced when attempting to develop and utilize suitable guidelines for placing a chemical that poses a risk to the Convention on an appropriate schedule. In that instance, the "rolling text", CD/961, lists this alcohol as a candidate for both schedules 1 and 2A. It was noted in the working paper that pinacolyl alcohol had little or no commercial use, an important factor which was one of several guidelines listed for schedule 1. However, it was pointed out that use of this guideline could set up a temporal trap in that there is often a rapid changeover in the industrial uses of chemicals. Valuable commercial uses for chemicals may be developed very quickly and it would be useful to have some means of predicting the future commercial value of chemicals before placing them on schedule 1 which could have the effect of discouraging further development. The working paper developed methodology using technical criteria to show that pinacolyl alcohol had sufficient commercial potential to suggest that schedule 2A might be more appropriate for it than schedule 1, although arguments could still be made that risk factors would override this technical assessment. As a result it was suggested that the modalities for revision of lists must contain a practical way of moving potentially useful chemicals from one schedule to another so that placing a substance on schedule 1 would not preclude valuable commercial developments.

2. In this paper the technical methodology is tested further using another controversial example. Thiodiglycol is readily available from industrially important chemicals, and attention has recently been focussed on its use as an immediate precursor to produce sulfur mustard for chemical weapons purposes. That thiodiglycol poses a risk to the convention is not in doubt. In the current rolling text,

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*/ Re-issued for technical reasons.

CD/961, thiodiglycol has been placed on schedule 2A, although there have been suggestions that it should possibly be placed on schedule 3. The argument for placing it on schedule 3 relates to the extent of its use in industry and the possible difficulty in applying intensive verification measures. This situation made thiodiglycol a useful candidate for further analysis.

3. There are two agreed criteria for Schedule 2A, while a third is still under discussion. The first of these relates to the use of a substance as a key precursor in the synthesis of a schedule 1 chemical, and the second to the potential risk that it poses to the Convention. The third criterion involves the commercial production quantities of the chemical, although no upper limits have yet been established for schedule 2. Thiodiglycol clearly meets the first two criteria.

4. Schedule 3 is concerned with two groups of chemicals: dual purpose and precursor chemicals. Dual purpose chemicals are toxic materials which have a previous history of use as chemical weapons and so pose some risk to the objectives of the convention, but which are produced in such large commercial quantities that routine inspection techniques are difficult to apply. The agreed characteristics of precursor chemicals on schedule 3 are that they are important in the production of one or more chemicals listed in schedule 1 or schedule 2, although they are usually more than one step away from the final product. They are produced in large commercial quantities for a variety of industrial uses. However schedules 2 and 3 may overlap in terms of the commercial production quantities involved.

5. Commercial production data for thiodiglycol is difficult to obtain. While some studies are underway, definitive information has not yet reached the Conference on Disarmament. However, thiodiglycol is known to have a variety of specialty uses. For instance, an industrial applications review prepared by Morton Thiokol, Inc, indicates that it is used in elastomers, lubricants, stabilizers, antioxidants, inks, dyes, photographic/copying processes, antistatic agents, epoxides, coatings, metal plating and textiles.

6. In order to explore further the notion of commercial utility, and in particular the potential uses of thiodiglycol, a literature survey was carried out using Chemical Abstracts from 1975 to 1988. This survey produced 346 references from 26 countries. An unusually large number of these references, some 56 %, came from industry; even more unusual was that some 52 % of the total resulted from the patent literature, indicating further that thiodiglycol is considered to be a useful chemical. The appended tables

show the classification and distribution of the research retrieved in this search, and also that the number of literature citations has generally been increasing. The complete bibliography is available from the Canadian Mission.

7. Of course, a survey of this nature is more illustrative than conclusive but, in the absence of information from producers and suppliers, it indicates that this chemical is of importance to many industries and that there is visible potential for its use to expand. While its current production may be at a level that can be monitored by the verification procedures of schedule 2A involving data reporting and on-site inspection; from another point of view, its end-uses are varied and involve a large number of customers using varying amounts of the chemical. In other words, there could be considerable difficulty in monitoring thiodiglycol until it has been transformed such that it no longer poses a risk to the Convention. This is not a problem to be ignored, as it has real resource implications for the International Authority as well as for industry. It could even be a factor in considering the placement of thiodiglycol on schedule 3. However, if chemicals on schedule 3 were only to be monitored by data reporting without any other supporting measures, caution due to the risk thiodiglycol poses to the Convention would seem to argue in favour of its placement on schedule 2A. In the end, the criteria for placement of chemicals are not likely to be so well defined as to obviate any role for judgement. Decisions will need to be made that give particular importance to the risk that a chemical poses to the objectives of the convention while giving due consideration to the potential monitoring dificulties and costs to the international authority as well as the concerns of industry.

CONCLUSIONS

8. In the absence of detailed production and consumption data for thiodiglycol, a literature search was conducted using chemical abstracts, which indicated that thiodiglycol has considerable potential commercial utility. In keeping with current discussions of this issue, this could have a bearing on the placement of this chemical on schedule 2A or 3. Further data from industry on the production and consumption of this chemical would be useful in order to complete the discussion on its placement and, as other CD members have indicated, it would be useful if states or organizations having such information would make it available to the Ad Hoc Committee on Chemical Weapons. This example once again focusses attention on the ongoing discussion of guidelines for the placement of chemicals on schedules and for their movement, and suggests that judgements in relation to risks posed to the objectives of the convention will continue to have an important role to play in such decisions.

THIODIGLYCOL

TABLE 1

COUNTRY	NO. OF PUBLICATIONS	(%)
USA	81	23.41
JAPAN	71	20.52
FEDERAL REPUBLIC OF GERMANY	48	13.87
USSR	37	10.69
UNITED KINGDOM	21	6.07
FRANCE	15	4.34
INDIA	12	3.47
SWITZERLAND	11	3.18
POLAND	7	2.02
AUSTRIA	5	1.45
CZECHOSLOVAKIA	4	1.16
HUNGARY	4	1.16
NETHERLANDS	4	1.16
AUSTRALIA	3	0.87
CANADA	3	0.87
CHINA	3	0.87
GDR	3	0.87
ITALY	3	0.87
BELGIUM	2	0.58
BULGARIA	2	0.58
SPAIN	2	0.58
SWEDEN	2	0.58
DENMARK	1	0.29
FINLAND	1	0.29
ROMANIA	1	0.29
TOTAL	346	

THIODIGLYCOL

TABLE 2

DISTRIBUTION OF PUBLICATIONS

COUNTRY	UNIVERSITY	INDUSTRY	GOVERNMENT
USA	19	56	6
JAPAN	14	56	1
FRG	6	42	-
USSR	31	4	2
UNITED KINGDOM	7	9	5
FRANCE	4	9	2
INDIA	10	-	2
SWITZERLAND	3	8	-
POLAND	6	1	-
AUSTRIA	5	-	-
CZECHOSLOVAKIA	3	1	-
HUNGARY	4	-	-
NETHERLANDS	-	-	4
AUSTRALIA	3	-	-
CANADA	1	-	2
CHINA	2	1	-
GDR	2	1	-
ITALY	1	2	-
BELGIUM	-	2	-
BULGARIA	2	-	-
SPAIN	2	-	-
SWEDEN	1	1	-
DENMARK	1	-	-
FINLAND	-	1	-
ROMANIA	-	1	-
TOTAL	127 (36.7 %)	195 (56.4 %)	24 (6.9 %)

THIODIGLYCOL

TABLE 3

SOURCE OF PUBLICATIONS

COUNTRY	MANUSCRIPT	PATENT	REPORT
USA	36	43	2
JAPAN	17	54	-
FR GERMANY	9	39	-
USSR	29	8	-
UNITED KINGDOM	13	8	-
FRANCE	6	9	-
INDIA	12	-	-
SWITZERLAND	3	8	-
POLAND	6	1	-
AUSTRIA	5	-	-
CZECHOSLOVAKIA	2	2	-
HUNGARY	3	1	-
NETHERLANDS	3	-	1
AUSTRALIA	3	-	-
CANADA	3	-	-
CHINA	3	-	-
GDR	1	2	-
ITALY	2	1	-
BELGIUM	-	2	-
BULGARIA	2	-	-
SPAIN	2	-	-
SWEDEN	1	1	-
DENMARK	1	-	-
FINLAND	-	1	-
ROMANIA	-	1	-
TOTAL	162 (46.8 %)	189 (52.3 %)	3 (0.9 %)

THIODIGLYCOL

TABLE 4

SUBJECT OF PUBLICATIONS

COUNTRY	SCIENCE	TECHNOLOGY	BIOLOGY	TOXICOLOGY
U.S.A.	42	27	10	2
JAPAN	26	36	9	-
FR GERMANY	24	22	2	-
U.S.S.R.	24	11	2	-
UNITED KINGDOM	10	8	1	2
FRANCE	7	6	2	-
INDIA	9	2	1	-
SWITZERLAND	3	6	2	-
POLAND	4	2	1	-
AUSTRIA	4	1	-	-
CZECHOSLOVAKIA	3	1	-	-
HUNGARY	3	1	-	-
NETHERLANDS	1	-	-	3
AUSTRALIA	1	-	2	-
CANADA	3	-	-	-
CHINA	1	1	1	-
G.D.R.	2	1	-	-
ITALY	2	-	1	-
BELGIUM	2	-	-	-
BULGARIA	2	-	-	-
SPAIN	2	-	-	-
SWEDEN	1	1	-	-
DENMARK	1	-	-	-
FINLAND	1	-	-	-
ROMANIA	-	1	-	-
TOTAL	178 (51.45 %)	127 (36.71 %)	34 (9.83 %)	7 (0.02 %)

Thiodiglycol Publications (1)

1956-60	13
1961-65	10
1960-70	13
1971-75	27
1976-80	35
1981-85	35

Thiodiglycol Publications (2)

1962-65	12
1966-70	59
1971-75	64
1976-80	67
1981-85	106
1986-88	38

(1) Based on a review of mainly patent literature carried out by Morton Thiokol Inc. of industrially important processes.

(2) Based on a literature review carried out in 1988 and using CA database. The period from 1975 was covered but the time lag in abstracting some journals and (especially) patents means that many citations appear from earlier years.

CONFERENCE ON DISARMAMENT

CD/CW/WP.280
16 March 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

SWEDEN

Provision of data relevant to the Chemical Weapons Convention

Sweden recognizes the importance of exchange of data on the chemical industry which will contribute to the negotiation of a Chemical Weapons Convention. In the attached document Sweden therefore submits a declaration on its production and import of chemicals listed in the draft Convention according to the outline in document CD/828 of 12 April 1988.

The data provided is based on information communicated to the National Chemicals Inspectorate pursuant to the Act on Chemical Products, effective since 1 January 1986. Under this Act, any import or manufacture of chemicals in quantities above 100 kg/year shall be reported to the Swedish Chemical Products Register.

Supplementary data regarding Schedule 1 chemicals has been provided by the National Defence Research Establishment.

The survey includes the chemicals listed in Schedules 1, 2 and 3 in document CD/952 of 18 August 1989 and reflects the situation in Sweden at the beginning of 1989.

The threshold for schedule 1 chemicals, used in the survey, is the one established by the register at 100 kg/year. For the purpose of this declaration the threshold for schedule 2 and schedule 3 chemicals has been established at 1 ton/year. The present reporting system in Sweden does not cover production of chemicals for captive use.

No schedule 1 chemical has been reported to the register. Data on schedule 2 and schedule 3 chemicals appears in Tables 1 and 2.

Schedule 1 chemicals were produced for protective purposes in quantities above 100 grams/year at one single facility.

TABLE 1

SCHEDULE 2 CHEMICALS	No. of Companies	
	Import	Production
N,N-Diethyl aminoethyl 2-chloride.HCl (869-24-9)	1	-
N,N-Dimethyl aminoethane 2-ol (108-01-0)	3	1
N,N-Diethyl aminoethane 2-ol (100-37-8)	2	-

TABLE 2

SCHEDULE 3 CHEMICALS	No of Companies	
	Import	Production
Phosphorus oxychloride (10025-87-3)	3	-
Thionyl chloride (7719-09-7)	1	-
Phosphorus pentachloride (10026-13-8)	1	-

CONFERENCE ON DISARMAMENT

CD/CW/WP.281
16 March 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

JAPAN

Provision of Data relevant to the Chemical Weapons Convention

With a view to contributing to the progress of the negotiations on the Chemical Weapons Convention, Japan presents its industrial data in accordance with the basic ideas of document CD/828, the first important step initiated by the Federal Republic of Germany. The data were collected with the cooperation of the Japanese Chemical Industries Association. In preparing the provision of data, thresholds proposed by the United States of America in document CD/802 were taken into consideration. Although some additional information might be submitted at a later stage, the data below, concerning relevant chemical substances and facilities producing and consuming these substances, reflect by and large the situation in 1989 of the Japanese Chemical Industry.

Verification to be applied to the industrial activities relating to Article VI of the Draft Convention needs to be not only effective, but also efficient and reasonable with the available resources of the Technical Secretariat in mind. It is, therefore, very important to establish a practical verification system especially in the area of the "non-production of chemical weapons" since the verification of "non-production", which is applied to the economic activities of the rapidly-developing chemical industry, is supposed to last for long after even the ten-year destruction period.

In the course of the negotiations, we have been stressing the need for striking a proper balance between risks posed to the objectives of the Convention and costs for maintaining a verification system. We hope that our data, together with the data which were already provided, and which will be provided more in detail in future by other countries, will contribute to searching for such a balance and to establishing an appropriate verification system. In this context, further consideration may be required to improve the relevant parts of the present "Rolling Text" (CD/961), including the placement of some chemicals on schedules, thresholds, the size of the International Inspectorate and so forth.

	Chemicals Produced	Number of Relevant Facilities of Each Chemical					
		Production Facilities			Processing and Consuming Companies (1)		
Schedule 2 Part A		1 to 10 tons/year	above 10 tons/year	total	1 to 10 tons/year	above 10 tons/year	total
	Arsenic trichloride		3	3		3	3
	2,2-Diphenyl-2-hydroxyacetic acid	1		1			
	N,N-Dialkyl(Me,Et,n-Pr or i-Pr) aminoethyl-2-chloride (2)		5	5	8	14	22
	N,N-Dialkyl(Me,Et,n-Pr or i-Pr) aminoethane-2-ol (2)		5	5	78	39	117
	Bis(2-hydroxyethyl) sulphide (thiodiglycol)		3	3	18	9	27
	total	1	16	17	104	65	169
Schedule 3		above 30 tons/year			above 30 tons/year		
	Phosgene	11			12		
	Cyanogen chloride	3			3		
	Hydrogen cyanide	10			11		
	Phosphorus oxychloride	7			38		
	Phosphorus trichloride	7			43		
	Di- and Trimethyl/Ethyl Esters of Phosphorus [P Ⅲ] Acid	2			3		
	Sulphur monochloride	2			8		
	Sulphur dichloride	1			1		
	Thionyl chloride	1			10		
	Phosphorus pentachloride	1			1		
	total	45			130		

(1) The number of companies totaling self-consumers and direct purchasers including trading houses.

(2) Only dimethyl and diethyl derivatives are considered to be produced, processed or consumed among dimethyl, diethyl, dinormal propyl and diisopropyle derivatives.

CONFERENCE ON DISARMAMENT

CD/CW/WP.282
16 March 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

TECHNICAL SUPPORT FOR THE CHAIRMAN OF THE AD HOC COMMITTEE

Western Group Working Paper

1. Introduction

As shown by Dr Rautio's Technical Instrumentation Group, the Chairman of the Ad Hoc Committee has need from time to time for technical support not normally available in Geneva. This Paper and attached Flow Chart describe a mechanism for establishing expert Study Groups under a Technical Co-ordinator to meet this need.

2. Choice of Topics

The number of topics to be taken up and their timing should be determined by need, available resources and expertise. The procedure for proposing new topics should not be over-formalised. Any delegation or Working Group Chairman would put a suggestion firstly to the Technical Co-ordinator (see Section 4). This new topic, plus any additional information or suggestion from the Technical Co-ordinator would be presented to the Extended Bureau and then passed, with its recommendations, to the Ad Hoc Committee for approval and establishment of a Study Group. The Ad Hoc Committee would allocate resources to the Study Group and establish time frames for the study. It would also confirm the Study Group's Chairman and Rapporteur and call for the nomination of experts.

3. Method of Working

i) No more than three or four topics should be under active consideration at any time. Topics could be taken up by study groups similar to last year's Technical Instrumentation Group with additional experts in the particular field of study as required provided by Member States to focus on specific technical aspects.

ii) The process would not necessarily be limited to CD sessions and could involve several stages: an initial meeting of perhaps a week, time for further research and reflection, and a second meeting during which a report would be prepared for submission to the Ad Hoc Committee within a specified time-frame. Further meetings and reports could be arranged if necessary.

4. Technical Co-ordinator

i) Technical co-ordination should not require an independent organ of the negotiations, but rather a co-ordinating mechanism assisting and directed by the Chairman of the Ad Hoc Committee.

ii) The Technical Co-ordinator would normally come from the Ad Hoc Committee Chairman's delegation, and would assist individual Study Groups as appropriate.

iii) The Technical Co-ordinator would have two primary functions:

a) responsibility for consultations within the Ad Hoc Committee on additional specific topics which should become the subject of in-depth study;

b) responsibility for administrative arrangements of these study groups and liaison between the groups and the Ad Hoc Committee.

To these ends the Technical Co-ordinator would consult informally as necessary with other delegations and experts.

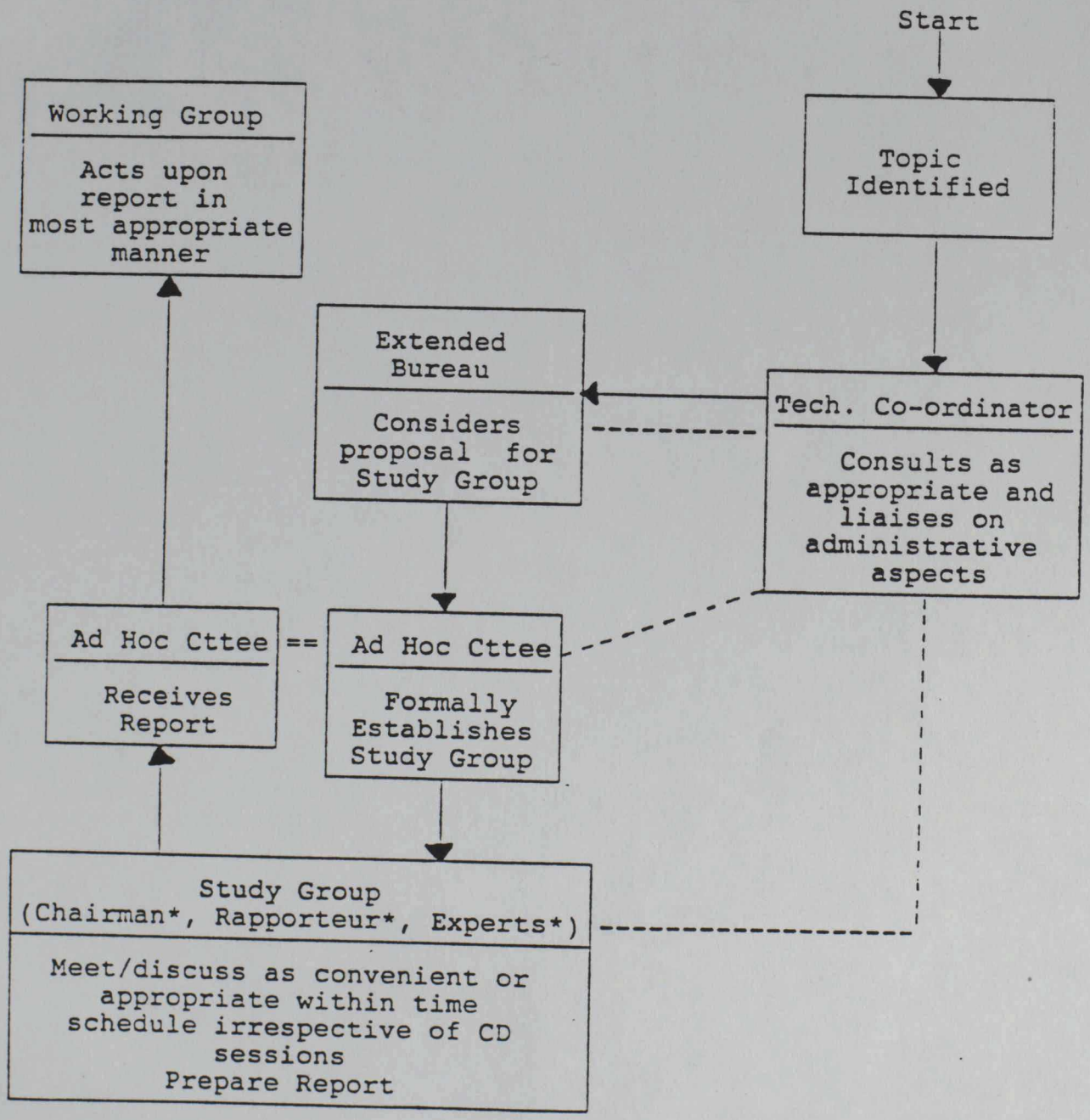
iv) The Technical Co-ordinator would ask delegations to volunteer an expert to chair a study group and to volunteer an official rapporteur for the group.

5. Costing

If the group met while the CD was in session, the services of the secretariat should be used fully. If the group met elsewhere, or at a time when the CD was not in session, then the countries volunteering the study group chairman and/or rapporteur could perhaps volunteer to bear the extra administrative costs.

TECHNICAL SUPPORT

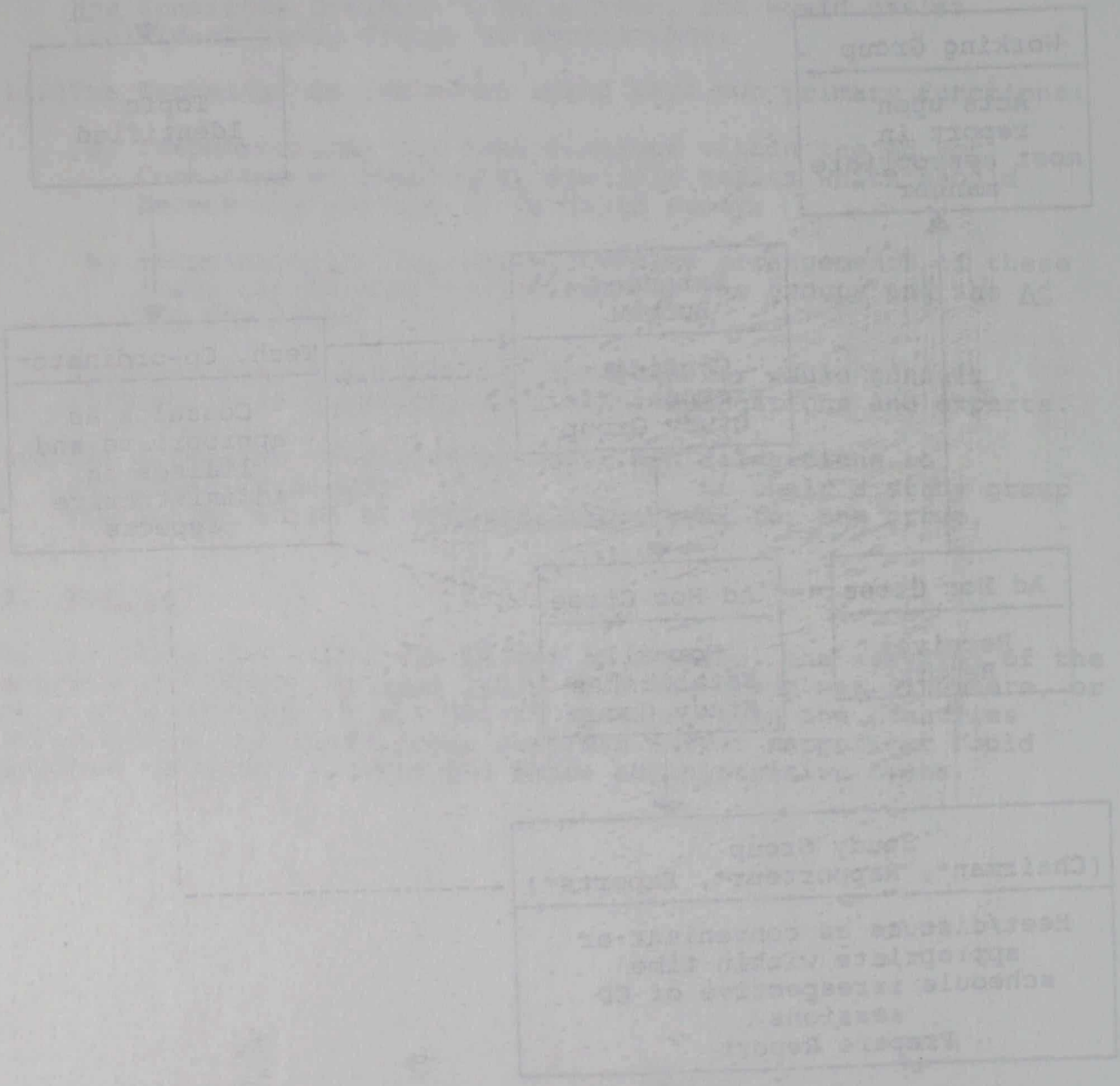
Indicative Flow Chart



———— Sequence of events

- - - - - - Liaison and consultation

* It is understood that delegations providing these officers will bear their costs.



FRG	CD/CW/WP.283	Report on the second trial inspection (challenge inspection) in the Federal Republic of Germany NOT REPRODUCED (see WP volume)	Also issued as CD/983 5 Apr. 90

* * *

FRG	CD/CW/WP.284	Ad Hoc verification: the establishment of national registers NOT REPRODUCED (see WP volume)	Also issued as CD/984 10 Apr. 90

CONFERENCE ON DISARMAMENT

CD/CW/WP.285
10 April 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

NORWAY

Report on a National Trial Inspection of an Industrial Chemical Facility

Introduction

At the 1988 summer session of the Ad Hoc Committee on Chemical Weapons it was suggested that States should conduct national trial inspections of their chemical industry. The purpose of these inspections was to establish an effective verification of non-production of illegitimate products and at the same time protect legitimately sensitive and confidential business information. During the first national trial inspection in Norway it was regarded as important to identify the problems and obstacles encountered. On the basis of experience gained from several different trials, detailed procedures could be elaborated which would later help to clear the ground for multilateral trial inspections.

Norway conducted a national trial inspection in February 1990. The facility inspected was an organic chemical production plant in the private industrial sector. The facility produces a legitimate commercial product using a Schedule 3 chemical, thionyl chloride, which can also be used in the prohibited production of mustard gas. Thionyl chloride is not manufactured at the site, but is imported from another country under the special regulations covering the import of certain chemical compounds.

The objectives of the inspection were:

i) to verify the permitted use of a Schedule 3 compound

ii) to verify the non-production of a Schedule 1 compound

iii) to control the mass-flow of the Schedule 3 compound to prevent diversion for prohibited purposes

iv) to evaluate whether a modern, well equipped chemical plant could produce a Schedule 1 chemical such as mustard gas.

Our inspection was carried out as a routine inspection in accordance with the guidelines for such inspections as laid down in the Protocol on Inspection Procedures, Addendum to Appendix I of CD/961 of 1 February 1990.

Description of the facility and the preparations for the inspection

The facility

The facility inspected is modern and medium-sized by Norwegian standards. It is geographically isolated, situated far from any inhabited area. The facility manufactures a limited number of products. The number of chemical steps involved in production is large, and production time to a final product is several weeks. The plant consists of one operating facility and this was inspected in its entirety. The facility belongs to a pharmaceutical company, which is well acquainted with

international inspections and reporting. The focus of our inspection was thionyl chloride, a Schedule 3 chemical, which can be misused for the production of mustard gas. The chemical is used in a batch process for the production of a pharmaceutical intermediate. The production takes place in a multipurpose reaction vessel to which reactants can be supplied by pipelines from a central storage depot.

Preparations

The inspection team did not make any preliminary visits to the facility prior to the inspection. Detailed information on the process and the plant was obtained by two visits to a pilot plant which uses the same chemical processes. The pilot plant belongs to the same company, but is located in a different part of the country. The pilot plant is similar, though not identical to the industrial plant.

This approach was chosen because an inspection team cannot be expected to know all the details of an unknown facility beforehand. By visiting a different plant first, we avoided the possibility of the members of the inspection team becoming too familiar with members of the staff of the facility. The information obtained from the visit to the pilot plant was used to plan the inspection.

The staff of the facility were given written information on the synthesis of mustard gas a few days before the inspection. Two members of the staff were familiar with the analysis and properties of chemical warfare agents. A representative from the Ministry of Foreign Affairs was present to advise and assist members of the staff. This was a precaution to ensure that the staff were not exposed to undue pressure from the inspection team.

Declaration

The chemical facility had declared that it used a Schedule 3 compound - thionyl chloride - in the permitted production of a pharmaceutical product. The Schedule 3 compound had been obtained by lawful import from another country.

Advance notice of inspection

Notice of the inspection was given to the management of the facility four days beforehand.

Protection of confidential information

At the opening conference, the inspectors signed a document stating that all information acquired was to be treated as confidential business information. The document was of the type used by the pharmaceutical company for outside consultants.

The inspection team

The inspection team consisted of four scientists from the Norwegian Defense Research Establishment, one chemical engineer from industry and one official from the Ministry of Foreign Affairs. All the scientists had experience from work with verification of chemical weapons. The team worked in pairs. Separate pairs were responsible for: a) auditing the records, contacts with trade union and environmental health and protection representatives of the facility; b) inspection of the storage depot and reaction vessels; c) chemical analysis.

Inspection equipment

The inspection team brought with them air-sampling equipment, a chemical agent monitor (CAM), a detection kit

for chemical warfare agents, simple laboratory equipment and analytical standards. In addition the group brought rodents for toxicity testing. All testing equipment had to be approved by the facility from the point of view of fire hazard. Instruments with BASFE or TÜV certificate were accepted.

The inspection team brought their own protective masks and gloves. Other safety-related equipment such as hard hats, safety glasses, or overalls were supplied by the facility.

Preparation on site

No physical preparations were made on-site. The facility adjusted its production so that thionyl choride would be used during the inspection.

The staff and workers had been informed of the inspection by the management.

The trial inspection

The production step involving the Schedule 3 chemical took more than three days. One pair of inspectors arrived before the process was started and sealed the reaction vessel when the reaction had been started up. The remaining team arrived and carried out their inspection on the second day.

The inspection started with an opening conference between the facility staff and the inspection team. The leader of the inspection team explained the purpose of the inspection and invited comments on the limitations and problems foreseen by the staff. The Security Officer of the plant gave a brief description of the geographical location of the facility, security arrangements, regulations imposed by government agencies and a short

description with map of the entire facility. Thereafter the inspection team was taken on a guided tour of the facility and was shown storage tanks and depots, production plant, laboratories and administrative buildings. It was decided that the inspection should cover the entire facility, with emphasis on the reaction step. Each of the inspection team pairs was escorted by a member of the facility staff.

Results of the inspection

Production equipment

The inspected facility is a modern plant with multipurpose vessels of high quality. The multipurpose vessel inspected was also resistant to corrosion. The reaction vessel could be sealed immediately on arrival and it was possible to obtain samples at any time during the process. Organic solvents were supplied by a complex system of pipelines, parts of which were difficult to trace. There was a supply of fresh air to each manufacturing area and this air-supply could be connected to masks for the personnel operating the plant.

Analysis

Samples were collected from all the chemicals added to the reaction vessel and also from the reaction vessel at different intervals. During the reaction the waste from the scrubber could be collected and analysed. It was also possible to collect samples at the end of the process from the mother liquor before and after it had been neutralized. The amount of thionyl chloride could be estimated from flow rates and chloride-ion analysis.

The reaction mixture in the vessel was studied with thin-layer chromatography, which made it possible to follow the removal of reactants and the appearance of products.

The product of the reaction was tested by subcutaneous injection into rats as described in CD/961, Appendix 1, Annex on Chemicals, part V, and it was verified that the product was not one of the supertoxic compounds in Schedule 1 nor another supertoxic or harmful compound.

The reactants and products could be identified by infrared spectroscopy when necessary. Air samples were collected in the reaction plant to show the absence of Schedule 1 compounds. The reaction plant and storage room were also searched with a CAM to verify the absence of traces of certain Schedule 1 compounds.

Auditing

The production unit was accompanied by a chart showing dates, batch number, type and quantity of the chemicals scheduled for use, and the type and quantity of chemicals actually used in the process. All the chemicals used were also entered on a chart showing the input and output of chemicals from the storage depot. These charts were hand-written and this increased the inspectors' confidence in them. The production charts were kept at the facility for several years and allow good control of the use of Schedule 3 compounds. A computer based system is being developed, which will supplement the hand-written records. Manipulation of data may be easier in a computer-based system, which may therefore be less reliable than a hand-written one.

Trade union relations

In Norway the head of the local branch of the trade union is often a member of the board of directors and is therefore well acquainted with the company's plans and operation. He was interviewed to find out to what extent the leadership of the company could carry out a covert production without the knowledge of the working staff.

Two important factors emerged:

a) This is a facility with very few products. Any new process or application of unusual chemicals immediately attracts the attention of the employees.

b) According to the regulations, environmental and health hazards associated with new chemicals have to be discussed with representatives of the working staff before they are introduced.

Protection equipment

The aim was to examine to what extent the protection equipment provided could be used in the production of toxic substances. At this facility the protection equipment was excellent, ranging from suits with full air-supply and civilian gas masks to a supply of fresh air in the plant. A collective protection shelter equipped with filters etc was also found to be in excellent order. Such shelters in factories are obligatory in Norway.

The first aid or medical supply did not reveal drugs such as atropine, reactivators, BAL og corticosteroid spray, which could be used to counteract intoxication with Schedule 1 compounds.

Concluding conference

After a brief private meeting among the inspectors, the staff of the facility were informed of the findings of the inspection. The inspectors reported that they had verified that the Schedule 3 compound was used for legitimate production purposes and that the flow of thionyl chloride could easily be followed and was well accounted for. Later it was also ascertained

that the import of thionyl chloride was in accordance with government regulations. The inspectors and the staff then discussed the problems involved in the production of a Schedule 1 compound at the site.

Assessment of the inspection

- The success of the inspection was to a large extent due to the excellent cooperation of the staff of the company, both at head office and at the site. This is an internationally oriented pharmaceutical company used to inspections from national and international bodies. The facility had previously been inspected by the American Food and Drug Administration and by several Norwegian authorities concerned with environmental, fire and explosives controls, and being a pharmaceutical company of high integrity it is used to keeping detailed information both on the production process and products.

- A preliminary visit was not considered essential as long as we had experience of a similar pilot plant. The opening conference was regarded as necessary in order to discuss the limitations of the inspection, to work out a system of protection of confidential information, and to allow the facility to approve of the equipment belonging to the inspection team, with regard to the safety regulations at the plant.

- An inspection requires detailed information about the chemical process to be inspected. However, a complete analysis of all the products and reactants is not always necessary. The most important objective is to account for the mass flow of the restricted chemical compounds. The use of toxicity testing, as described in CD/961, may reveal presence of supertoxic and harmful compounds and such testing has the advantage that knowledge of the

chemical structure of the compounds is not essential. The limitations are that not all compounds listed in Schedule 1 are supertoxic and not all supertoxic compounds are in Schedule 1. In addition, the inspectors must be aware that problems in toxicity testing may be caused by diluting or toxic effects of solvents, or by interference with toxic side products or impurities. The absence of traces of certain Schedule 1 compounds at the plant after analysis by a CAM was an important confidence-building factor.

- Unrestricted access to all the company's business documents and production data can considerably facilitate a routine inspection. This necessitates strict measures to protect the company's business information. The document signed in our case does not give the company a full guarantee and cannot be regarded as adequate. The inspections carried out by the International Secretariat will need to build up confidence between the inspectors and the commercial firms. It will in the future be necessary to obtain a form of security clearance through the Secretariat and to use a standard document. These questions are dealt with in the Annex on the Protection of Confidential Information, CD/961, Appendix I.

- The information relevant to the inspection has to be selected from a mass of other data less relevant. In this case the flow of thionyl chloride in the single step is the most relevant information, and it is buried in the production chart and storage chart among other details. The use of hand-written production chart and storage chart in the format used by this facility increased the confidence of the inspectors. Computer-based systems can more easily be manipulated. Since the data appear in several different documents, unauthorized activities would involve two complete sets of all documents. Hand-written plant records may be a requirement for certain plants which are to be inspected on a routine basis.

- Discussions with the trade union representative convinced us that the national regulations for use of chemicals in industry made it difficult to carry out production without the knowledge of the working staff at this facility. The importance of the information obtained by discussions with the workers will differ between different facilities and different nations.

- The high standard of individual and collective protective measures in this facility means that the personnel are well protected against chemical hazards. For the production of a supertoxic Schedule 1 chemical, however, we would have expected to find a larger number of fully air supplied suits. The protection equipment present would only be sufficient for the personnel at the facility who are not directly involved in such a production. The geographical location, the distance from inhabited areas, did not exclude the possibility of such production. We did not find any antidotes to Schedule 1 compounds.

- The multipurpose vessel used for thionyl chloride was highly resistant to corrosion. Still, we observed a few weaknesses in the rubber and plastic seals, which would be destroyed by mustard gas at high temperatures. Such minor details may be important in an evaluation. In general the inspection concluded that mustard gas and other such chemicals can easily be produced in a modern chemical plant of high quality.

CONFERENCE ON DISARMAMENT

CD/CW/WP.286
11 April 1990

Original: ENGLISH

Ad hoc Committee on Chemical Weapons

AUSTRALIA

Ad Hoc Verification

Discussion Paper

1. Delegations have long been concerned that the verification régimes of the draft Chemical Weapons Convention do not deal adequately with the facilities that could be misused for chemical weapons production, but do not produce one of the commercial chemicals specified on the Schedules in the Convention. For that reason, earlier proposals have been made by both the Federal Republic of Germany and the United Kingdom for ad hoc verification (documents CD/CW/WP.183, 210 and 232). These two approaches, while based on a common underlying concern, provide different ways for ameliorating the concern.

2. This paper has developed from a series of discussions of the problem undertaken by Western Group delegations and while not representing the views of any one delegation puts forward for consideration an approach to ad hoc verification which attempts to combine the strongest elements of the proposals of both the Federal Republic of Germany and the United Kingdom. The proposed approach attempts to preserve the best features of both approaches and provide the most effective verification at the lowest cost and the lowest risk to sensitive military and commercial information.

3. The approach recognizes that provisions for ad hoc verification and challenge inspection complement each other and can be developed in parallel. Some refinement and additions to the provisions for ad hoc verification might eventually be necessary, based on the final form of Article IX of the Convention. Also a number of the elements of this approach need to be developed further, such as the level of the inspection quota and the criteria for inclusion of a chemical production facility on the proposed Register.

Proposed Approach to Ad Hoc Verification

A. It is necessary to supplement the framework for routine inspection as contained in the draft Convention (CD/961) in order to provide a means for routine monitoring of chemical production facilities that are capable of producing chemicals on Schedules 1, 2 or 3, but which do not currently do so and are, therefore, not covered by the declaration and monitoring régimes of Schedules 1, 2 and 3.

B. Each Party should be obliged to provide a national register of facilities on its territory for production of chemicals. The Register would include all plant sites, whether civil or military, based on agreed characteristics which, to the extent possible, should be defined so that plant sites that pose no risk to the objectives of the Chemical Weapons Convention are excluded from the Register.

C. Ad hoc visits could be requested for any plant site on the National Register, including declared facilities for production of Schedules 1, 2 or 3 chemicals. The inspection procedures would be designed to verify the absence of undeclared production of chemicals that are listed on Schedules 1, 2 and 3.

D. The ad hoc visit would be conducted on-site by a team of inspectors from the Technical Secretariat. As for other routine inspections, access would be mandatory and within agreed time frames. No national of a requesting Party would be present as an Inspector or Observer, except by consent of the Party receiving the visit. The results of the visit would be treated in the same manner as the results of other types of routine inspections.

E. Each Party would have a limit on the total number of ad hoc visits that it could request annually, (similarly, there would be a limit on the total number of ad hoc visits that could be requested annually by the Technical Secretariat.) It is for consideration whether there would also be a limit to the number of ad hoc visits that a Party would have to receive.

F. Ad hoc visits could be requested only for plant sites that have been placed on the National Register. Cases where a State Party has concerns about plant sites that it believes should have been placed on the National Register of another Party, but were not, could be resolved through the procedures specified in Article IX. A Party whose facility has been questioned shall be obliged to co-operate in resolving the issue.

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CONFERENCE ON DISARMAMENT

CD/CW/WP.287
11 April 1990

Original: ENGLISH

Ad hoc Committee on Chemical Weapons

ITALY

Production Capacity

I. INTRODUCTION

The most recent version of the draft Convention on chemical weapons, contained in document CD/961 of 1 February 1990, requires declarations of the production capacity related to facilities dealing with compounds on List 1 (Appendix I, pp. 81 and 82: Annnex to Article V - I: A.5 and I: B.7; Appendix II, p. 167: Model for an agreement relating to single small-scale facilities 1. (b) (ii); on List 2 (Appendix I, p. 104: Annex 2 to Article VI - 2. Facility (vi)) and on List 3 (Appendix I, p. 112: 1. (iv) (c)).

Following an attempt to arrive at a definition made by a Group of Experts (CD/961, p. 157), the conclusion was also drawn that "Although it was generally felt that it would be desirable to have one definition of 'production capacity' applicable all through the Convention, it was also concluded that this might not be possible".

The production capacity issue was also addressed in document CD/CW/WP.171 dated 14 July 1987, submitted by the United States delegation. The concept of "production capacity" is involved in at least three other cases:

- it is one of the factors to be declared in connection with "general plans for the destruction of chemical weapons" (CD/961 - Annex to Article IV, p. 75: V; 2 (c)).

- it can be adopted as a term of comparison between two chemical weapons production facilities, in connection with the problem of the "order of destruction". The aspect of "destruction capacity" has been addressed in document CD/CW/WP.148 (Cuba).

- it is one of the "possible factors identified to determine the number, intensity duration, timing and mode of inspections to facilities handling Schedule 2 chemicals" (CD/961 - Appendix II, p. 155 - 2 (c)).

A provisional indication of a means of calculating "an approximate production capacity" (CD/961, p. 159) is given by the formula:

$$\text{Production capacity (tonnes/year)} = \frac{\text{des. capacity} \times \text{op. factor} \times \text{no. of units}}{\text{pl. op. hours}}$$

where:

des. capacity = nameplate or design capacity of one unit (tonnes per year),

pl. op. hours = hours of planned operation to achieve the design capacity,

op. factor = operational factor: (which) should take into account the various facility-specific and process-specific factors which would affect the actual practical production capacity and could e.g. be determined during the initial visit. A need must exist for a provisional value of the operational factor to be applied before the initial visit has taken place.

The present document has the double aim of expanding the concept of production capacity in order to bring out the quantity of production obtainable from a plant and at the same time analysing the practical criteria which can be adopted in order to make available homogeneous data.

II. DEFINITION OF CAPACITY

The capacity of a chemical facility cannot be defined in unequivocal terms (this applies in particular in the case of batch processes where the number of manually controlled operations is greater than those of continuous processes), since the industrial efficiency of a production unit is not governed solely by its physical features but also by the professional competence of production operators. It has to be borne in mind that the capacity of a facility is not constant in time, but tends to increase. This is due to the process of collective learning (personnel become better aware of the margins adopted in the project for the critical dimensions of equipment and technical specialists improve their professional competence) which applies to all staff engaged in industrial activities and is commonly expressed by the concept of "learning curve" (curve of experience). It reflects at the same time the continuous advance of technology as a result of specific research programmes and general progress in the state of the art (introduction of automation, improvement in the properties of materials, etc.).

For practical purposes, three different indexes of capacity can be used for assessing a production facility:

(1) the nameplate capacity;

(2) the effective capacity;

(3) the programmable capacity.

1. Nameplate capacity

The product output of a facility reaches the peak value under particularly favourable conditions which at the same time remove any limiting factors, internal or external, militating against the production unit. These conditions are achieved within relatively short periods during the life-time of the facility. The optimal operating régime will normally be arrived at by means of test-runs which are normally carried out at the following times:

- at the start of production, when the project engineer's guarantees are verified;

- at the beginning and end of a restructuring period, when the decision has been taken to improve the efficiency of the production cycle.

The operational conditions corresponding to maximum production do not necessarily coincide with those affording maximum economic efficiency. It is in fact possible that negative effects, such as a deterioration in the quality of the final product, a reduction of the yield obtained from the process, excessive wear on the machinery or a higher unit consumption utilities may militate against operation of the plant under conditions of maximum production potential, except for short periods and in particular market situations.

The capacity of the facility is verified by means of test-runs and is normally expressed in quantities (tonnes or m^3) per operating day or hour and indicated by the term nameplate. The use of this term allows for the fact that, in practice, the corresponding annual output (daily capacity times 365 days) cannot be achieved as a result of the occurrence of obstacles of a technical, organizational or logistic nature. It is also to be noted that the maximum capacity may vary according to the purity of the reagents used and of the final product. The maximum capacity will in general increase whenever pure raw materials are used. On the other hand, a stringent specification for the final product will have a penalizing effect on the potential output of the plant.

The nameplate capacity is often taken to be synonymous with the design capacity; in fact, the nameplate capacity corresponds to the design capacity in the case where test-runs confirm the values expected by the project engineers. This equivalence is not always achieved in industrial practice.

2. Effective capacity

The effective capacity of a facility (normally expressed on an annual basis) is smaller than the nameplate capacity. The difference between the two is calculated by taking into account the internal technical limiting factors present in the production structure which prevent the values achieved in test-runs being maintained on a constant basis. Examples of such impediments are the fouling and incrustation of reactors, the breaking down of equipment and the aging of catalysts. Events of this type necessitate a shutdown of the facility and slowing of production for the time required to restore the conditions of technical reliability needed for a normal and safe production profile. The effective capacity may also be influenced by less than optimal seasonal or meteorological conditions. During the summer months, for example, the temperature of the cooling waters supplied to the heat exchangers may

become too high to be able to disperse the total amount of heat produced with the production unit operating on full load. The maximum production of the plant during the summer months will accordingly be lower than that obtained in the winter.

The effective production capacity of the facility can only be determined from a thorough-going knowledge of the facility characteristics, the management procedure and the technological processes used. In the great majority of industrial facilities the effective capacity will be slightly over 90 per cent of the nameplate capacity (on-stream factor 0.9). In the absence of specific data, the general practice is to assume that a chemical facility can operate under optimal conditions for 328 or 330 days in the year; an equivalent index often used in industrial practice is 8,000 hours per year running at nameplate capacity.

In the case of batch process units designed to produce compounds A, B and C (multipurpose plant), the capacity will be based on one of these products, as if the plant was engaged on the production of that compound throughout the year. The specification will state, for example, that the particular multipurpose production unit has a capacity of 100 tonnes of compound A, or 90 tonnes of compound B or 80 tonnes of compound C. In reality the overall annual output of the product mix will depend not only on the relative incidence of each individual product, but also on the frequency with which the production régime has to be changed. The passage from one production cycle to another (for example from A to B or from A to C) normally requires the replacement of some of the machinery or a modification of the connections between them. The plant shutdown interval between two different production runs will accordingly be greater than that normally required between two production runs of the same product.

The experience of plant management acquired over the years normally enables an increase to be made in the effective capacity. The difference between the nameplate and effective capacities tends to decrease and, as a result of the introduction of improvements, the design capacity is often exceeded.

3. Programmable capacity

Over a reasonable long time-span, plant production is also governed by external reliability limits affecting the production structure. Examples of such factors are the lack of sufficient product storage capacity, inadequate shipment facilities, maintenance factors in the heating plant or a temporary shutdown of one of the downstream plants processing the product. Events of this type necessitate halting or reducing the production load at times which frequently do not coincide with those scheduled for maintenance operations or for other technical reasons.

The programmable capacity as defined above expresses the maximum quantity of product which it is possible to produce and is normally shown in tonnes or m^3 per year. This value is fixed from year to year and the possibility cannot be excluded of its reduction as times goes by, even though the technical efficiency (as a consequence, the effective capacity) of the facility has increased in the meantime. If such is the case, it can therefore occur that external impediments affecting the physical structure of the facility can interfere with full exploitation of the available capacity. Only

a far-reaching knowledge of the characteristics of company programmes will enable the differences between the effective and programmable capacity values to be estimated.

III. COMMENTS AND PROPOSALS

1. The hypothesis of solution envisaged for calculation of the production capacity (CD/961, pp. 157-159) would appear to overlook some of the conditions in the industrial environment. A declaration of capacity based on such a formula might fail to supply data mutually compatible. For example, many managers of facilities working on batch process basis leave out of account shutdown periods during holidays, weekends and often even during the night hours. This type of management practice may induce production managers to declare a number of potentially usable hours during the year which are clearly lower than their real productive efficiency.

2. The indexes of nameplate, effective and programmable capacity enable the various prospects of the product efficiency of a facility to be assessed and at the same time the limitations on increased output identified. Taking into account that:

NAMEPLATE CAPACITY $>$ EFFECTIVE CAPACITY $\geq$ PROGRAMMABLE CAPACITY

the first index (and frequently also the second) over-estimates the real annual production capacity. For purposes of the Convention, on the other hand, the first value provides a less equivocal indication. Both the nameplate and effective capacity values, when available, should be declared in the initial declaration and confirmed at the time of the first visit of inspection to the facility and during examination of the production log books. On the other hand, the programmable capacity is of little use as far as the requirements of the Convention are concerned, since it takes into account also external impediments affecting the production structure which cannot always be verified during the auditing of records.

3. The acquisition of data on the nameplate and effective capacity of a facility implies a good knowledge of the relevant technological process, sound experience of plant management and the availability of relatively complex analytical equipment and techniques. Adequate managerial and specialist resources are required, namely, for the performance of test-runs under strictly controlled conditions (measurement of all the flow rates at plant input and output points, analysis of these flow rates, chemical-physical characteristics and thermodynamics, processing of analytical data and so on). If the plant manager does not have these data available, the International Authority cannot demand performance of test-runs to enable proper implementation of the Convention: in such a case the production data, as indicated in the following paragraph (4.C.), will have to be used.

4. Three cases may be assumed for purpose of calculating the effective capacity of a facility:

A. In the first case, the design capacity is known and furthermore a recent conducted test-run (carried out, for example, not beyond five years before the date of submission of the initial declaration), has certified that design capacity corresponds to the nameplate capacity. Under this assumption,

production log books will probably be available from which the "planned op. hours" and "the operational factor" can be extracted. The effective capacity of the facility is calculated in this way, making use of the formula proposed by the experts:

$$\text{Effective capacity (tonnes/year)} = \frac{\text{des. capacity x op. factor x no. of units}}{\text{planned op. hours}}$$

If the op. factor and planned op. hours factor are dubious or not known, the effective capacity may be conventionally assumed to correspond to 90 per cent of the design capacity.

B. In the second case, the nameplate capacity has been determined by means of a test-run, but the design capacity is not known, or is known but is lower than the nameplate capacity (this situation is typical of older plants which have been modified over the years to an extent that design characteristics were altered). In this case the formula contained in CD/961 will be modified, replacing the design capacity by the nameplate capacity:

$$\text{Effective prod. capacity (tonnes/year)} = \frac{\text{Nameplate cap. x op. factor x no. of units}}{\text{planned op. hours}}$$

If the planned op. hours and op. factors are dubious, the effective capacity may be conventionally assumed to be equivalent to 90 per cent of the nameplate capacity.

C. In the third case, no documentation certifying the maximum production capacity of the facility is available at all or such documentation is available but not very reliable (in this case it should not be excluded that the design capacity may be available but its authenticity may not have been demonstrated by production log books). The nameplate capacity can only be estimated on the basis of production data, as suggested in the above-mentioned document CD/CW/WP.171.

In particular, the following formulae may be used.

(a) for continuous process:

$$\text{ESTIM. NAMEPLATE CAP. (tonnes/year)} = Q \times 365 \times \text{no. of production lines}$$

where Q is the daily output over a 24 h period;

(b) for batch process:

$$\text{ESTIM. NAMEPLATE CAP. (tonnes/year)} = \frac{Q}{\Delta} \times 24 \text{ h} \times 365 \times \text{no. of production units}$$

where Q is the number of tonnes produced in a batch;
Δ is the duration (in hours) of the complete batch production cycle.

In this case again the effective capacity may be assumed to be equivalent to 90 per cent of the estimated nameplate capacity.

5. The application of the above procedures to facilities with limited production (e.g. with an output of less than five tonnes/year) does not appear likely to provide reliable data, as has already been pointed out in document CD/961, page 158.

In any case, whatever production capacity is assigned to such facilities allowance will have to be made for a very high error margin (even of the order of 100 per cent).

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CONFERENCE ON DISARMAMENT

CD/CW/WP.288
11 April 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

AUSTRALIA; CANADA; FINLAND; FRANCE; GERMANY, FEDERAL REPUBLIC OF; THE NETHERLANDS; NORWAY; SWEDEN; SWITZERLAND; AND THE UNITED KINGDOM OF GREAT BRITAIN AND NORTHERN IRELAND

International Interlaboratory Comparison (Round Robin) Test

1. Introduction

The Finnish Blue Book of 1988 on Standard Operating Procedures for the Verification of Chemical Disarmament expressed a hope that it would initiate an international collaborative effort to develop Standard Operating Procedures for verifying the future Chemical Weapons Convention. Subsequently experts from several laboratories were approached for their views on an international test. The suggestion was enthusiastically received and in January 1989 an initial meeting was held among the experts present in Geneva to plan the experiment. The plans were finalized in the following June and Finland was nominated as the coordinator of the test. It was decided to table a preliminary report on the results in the CD as soon as the results were available and to provide a more detailed report in a Blue Book during the summer session of 1990.

2. Objective

The aim of the exercise was to test existing procedures for sample pretreatment and analysis to determine whether standard operating procedures would be required for the future Convention. It was not the intention of the exercise to choose laboratories for verification purposes; this will be done first by the Preparatory Commission.

Accordingly, all laboratories used their own methods for sample pretreatment and analysis and provided detailed descriptions of these methods and of the instruments with which the analyses were performed. The main objective was to identify qualitatively agents and their degradation products. Additionally, some quantative estimation of the extent of contamination was sought. If more than one analytical method was used for the analysis, results with each technique were to be reported.

Each participating laboratory was provided with a username and password allowing access to the Finnish VERIFY database. For this purpose a separate "Round Robin" file was created in the database to enable participating laboratories to add their results and experimental details to it.

3. Types of samples

At an early planning meeting it was agreed that the test should comprise the analysis of two types of spiked air samples at different concentrations and one type of soil sample. Further, for each type of sample (air and soil) one agent that was mentioned by name in Schedule 1 of the then draft rolling text CD/881, together with two or three of its degradation products would be used. Each laboratory would receive similar samples. The anticipated concentration range in the samples were:

1. Tenax air samples; 50 - 500 ng of agent/tube (Chrompack)
2. XAD air samples; 50 - 1000 µg of agent/tube (Supelco)
3. Soil samples; 10 - 100 mg/kg of agent on 3 samples of 50 - 100 g of soil, and one background soil sample. Laboratories would not know which of the four soil samples were spiked ones and which was the background one.

4. Sample preparation

France prepared the samples and analyzed one set of samples to act as a reference. In the spiked air samples the concentration

of sarin (97%) was between 270 and 340 ng in the Tenax tubes and 110-120 µg in the XAD tubes. VX and its degradation products, N,N-diisopropylaminoethanethiol, O-ethyl methylthiophosphonate and ethyl methylphosphonate were used to spike the soil samples and the concentration of VX (94%) and the degradation products were 3 - 5 mg per 50 g of soil. All samples were coded and individually packed in Mylar bags and placed in an insulated chest together with refrigerant units for transportation. Finland recoded the samples before delivery to the participating laboratories. No details of the spiking methods were distributed to the laboratories.

5. Analytical methods

The main analytical methods used in the exercise turned out to be gas chromathography (GC) with selective detectors and mass spectrometry (MS), most often as a hyphenated technique (GC-MS). One laboratory additionally used high performance liquid chromatography-mass spectrometry (HPLC-MS) and micro liquid chromatography in combination with flame photometric detection (Micro-LC-FPD). Another laboratory tested the potential of a mobile mass spectrometer and a further participant tested retention spectrometry. In addition to GC-MS three laboratories used nuclear magnetic resonance (NMR) spectrometry and one laboratory used infrared (IR) spectrometry.

6. Timeframe

The samples were delivered to the laboratories in late September - early October 1989 except for Australia who first received the samples in late October. Owing to various time constraints some laboratories requested that the timeframe for the analyses be referred to the end of November. At that time three laboratories asked for the timeframe to be postponed further to the end of the year. The first reports from certain participants were in the coordinating laboratory at the end of October.

The laboratories were asked to record their results on the VERIFY database themselves or through the coordinating laboratory by 5 January 1990. In the event four laboratories added their results directly and four sent their reports to the coordinating laboratory. When all laboratories had reported their findings France described how the samples were prepared and which chemicals at what concentrations were used to spike them. The results were discussed for the first time toward the end of the resumed session in January 1990 in Geneva.

7. Results

Table 1 shows the results from the analysis of the Tenax and XAD air samples and Table 2 the results from the analysis of soil samples. The spiking chemicals are marked with an asterisk. The reference results obtained by France are shown in column 10 (laboratory 10).

7.1. Air samples

All laboratories undertaking these analyses found sarin in the samples. Many of the laboratories found even sarin impurities in the XAD samples.

7.2 Soil samples

Table 2 shows the results from the analysis of soil samples. Six laboratories found VX in soil samples. Eight laboratories found either impurities or degradation products of VX.

It is interesting to note that the two laboratories (Numbers 2 and 5 in Table 2) which did not detect intact VX analysed the soil samples in December, that is, more than two months after the samples had been prepared. Previous studies have shown that degradation of VX is rapid in a range of soil types (for example, in one study only 0.1% of intact VX was left after three weeks). On that basis, it is not surprising that these two laboratories could not detect intact VX. It is fortuitous, from the

verification viewpoint, that VX decomposes to give degradation products which are sufficiently stable in soil to enable a valid conclusion to be drawn. Thus laboratories may be able to conclude that a soil has contained VX, without detecting the intact VX.

8. Conclusions

The concentration of sarin in the air samples was sufficiently hight to permit identification of the spiked agent. Identification of VX in soil samples was evidently more problematic because VX degrades in soil. The storage of samples, the time between preparation of the samples and their analysis, and the sample pretreatment methods would appear to be critical factors contributing to the differences between the results.

Six of the laboratories were able to identify clearly the spiking agent and eight laboratories found degradation products of VX which, as they could not result from other than VX itself, serve as fingerprints. No laboratories recorded agents that were not present; in other words there were no false positives.

The Round Robin Test provided some confidence that the analytical methods available are capable of providing valid information for verification purposes. However, there is a continuing need to develop and review procedures for the storage of samples, for sample pretreatment and for sample analysis. Criteria of identification was recognized as an area which needs further discussion.

Direct contact with the VERIFY database necessitated the use of modems and telephone lines. Communication would be facilitated if the hardware and software were to be standardized. The incomplete status of the VERIFY database limited its usefulness in the identification of the degradation products. Further the need for more stringent timeframes for any future interlaboratory comparison tests was recognized.

Table 1: Compounds identified in Tenax and XAD samples in Round Robin interlaboratory test.

	laboratory									
	1	2	3	4	5	6	7	8	9	10
TENAX:										
⋆ Sarin	x	x	x	x	x	x	o	x	x	x
XAD:										
⋆ Sarin	x	x	x	x	x	x	x	x	x	x
Isopropyl methylphosphonate				x			x			
Methylphosphonic acid							x			
Diisopropyl methylphosphonate		x	x			x	x	x	x	
Ethyl isopropyl methylphosphonate			x	x		x		x	x	

⋆ spiked compound

o not analyzed

Table 2: Compounds identified in soil samples in Round Robin interlaboratory test.

	laboratory									
	1	2	3	4	5	6	7	8	9	10
SOIL SAMPLES:										
* VX	x		x	x		x		x	x	x
* O-Ethyl methylthiophosphonate	x		x			x		x	x	x
O-Ethyl S-methyl methylphosphonothiolate	x	x	x		x					
* Ethyl methylphosphonate	x		x	x		x		x	x	x
Ethyl methyl methylphosphonate									x	
Methylthiophosphonic acid										x
Methylphosphonic acid				x				x		x
O,S-Diethyl methylphosphonothiolate	x	x	x	x					x	x
Diethyl methylphosphonate	x	x	x	x	x	x		x	x	
* N,N-Diisopropylaminoethanethiol										x
Bis[2-(diisopropylamino)ethyl] disulphide			x	x					x	x
N,N-Diisopropyl aminoethanethioisopropane										x
N,N-Diisopropyl aminoethanethioethane										x
N,N-Diisopropyl aminoethyl-2-chloride			x							
N,N-Diisopropylethylamine					x					

* spiked compound

Poland	CD/CW/WP.289	Provision of data relevant to the Chemical Weapons Convention NOT REPRODUCED (see WP volume)	Also issued as CD/985 17 Apr. 90

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Canada	CD/CW/WP.290	National trial inspection at a single small-scale facility NOT REPRODUCED (see WP volume)	Also issued as CD/987 19 Apr. 90

* * *

India	CD/CW/WP.291	Letter dated 19 April 1990 from the Permanent Mission of India addressed to the Secretary-General of the Conference on Disarmament transmitting a document entitled "Report of the national trial inspection conducted by India" NOT REPRODUCED (see WP volume)	Also issued as CD/988 20 Apr. 90

* * *

GDR	CD/CW/WP.292	Report on a trial challenge inspection in a chemical industry plant NOT REPRODUCED (see WP volume)	Also issued as CD/996 12 Jun. 90

* * *

GDR	CD/CW/WP.293	Inspection methodology for challenge inspections in industrial chemical plants NOT REPRODUCED (see WP volume)	Also issued as CD/997 12 Jun. 90

GDR	CD/CW/WP.294	Application of trace analysis to exploit memory effects in challenge inspections NOT REPRODUCED (see WP volume)	Also issued as CD/998 12 Jun. 90

* * *

Austria	CD/CW/WP.295	Report on a national trial inspection NOT REPRODUCED (see WP volume)	Also issued as CD/999 12 Jun. 90

CONFERENCE ON DISARMAMENT

CD/CW/WP.296
18 June 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

UNITED KINGDOM OF GREAT BRITAIN AND NORTHERN IRELAND

Addition of Chemicals to the Schedules

1. The attached flow chart illustrates the type of assessment process which the UK believes will be required when a chemical is submitted to the Technical Secretariat by a State Party which considers the chemical a sufficient risk to merit assignment to a schedule of the Convention. The chart reflects discussions held in the Working Groups of the Ad Hoc Committee and has been developed from the flow chart in the informal working paper tabled by the French delegation on 6 April 1989.

2. The first step proposed in the chart addresses the question of whether or not there is any legitimate civil production of the substance named. If there is none, the substance can only be assigned to Schedule 1 or be declared no risk to the convention. If there is civil production, the substance can only go on Schedule 2B or Schedule 3, or be declared after subsequent assessments to be of no risk.

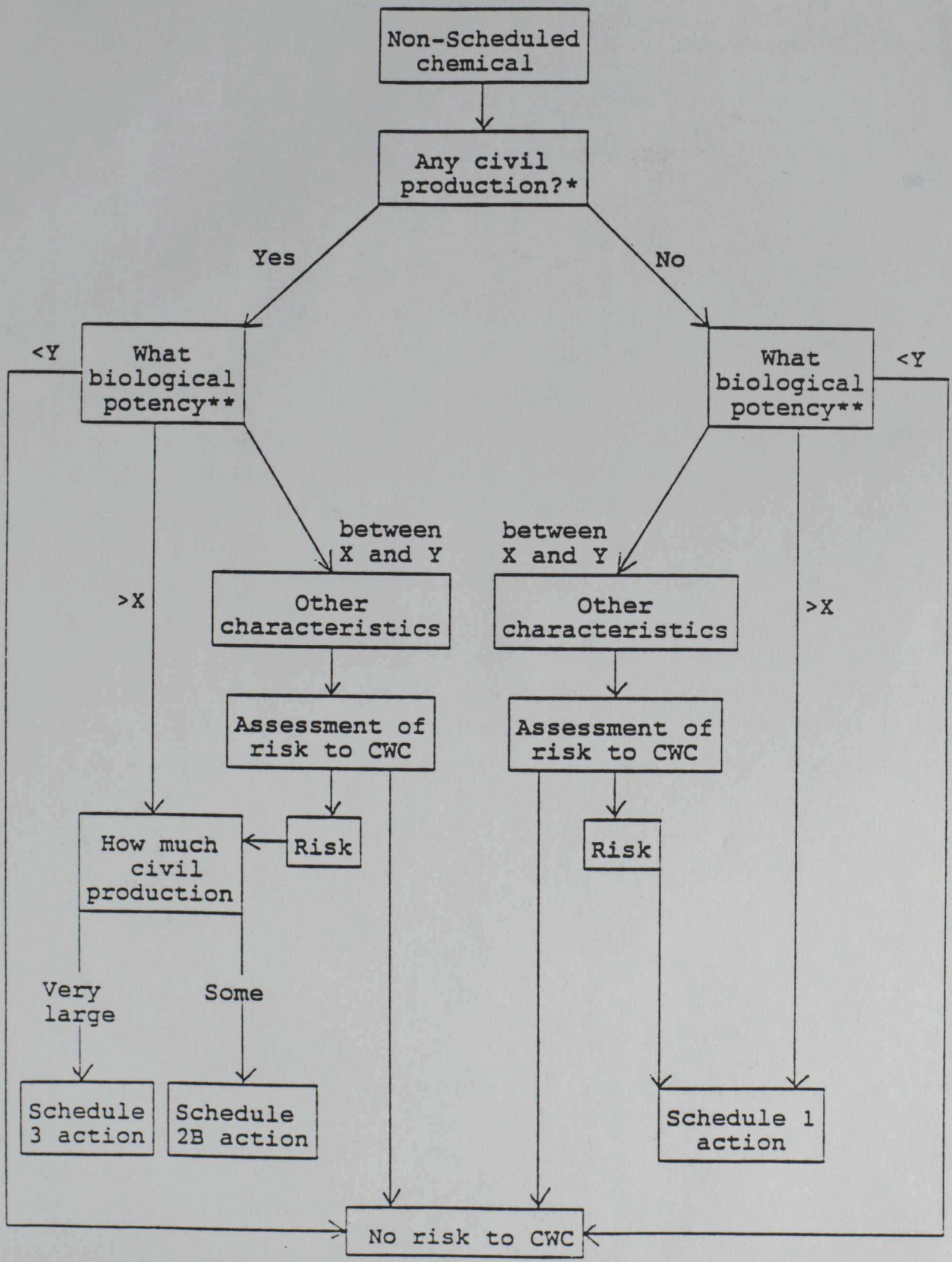
3. The second stage examines the biological potency (be it lethality or incapacitation) and proposes three ranges for examining such potency. Some substances will have such a low potency (<Y) that they will not constitute a risk to the Convention. At the other end of the spectrum there are substances that are so potent (>X) that they are indisputably a risk. There are those of intermediate potency (between X and Y) that will need to be further assessed in terms of characteristics other than biological potency to determine what level of risk they pose. These characteristics might include properties related to weaponizability.

4. This approach is intended to overcome the difficulties encountered in earlier discussion on the Annex to Article [...] (CD/881) which envisaged a sharp cut-off for the lethal dose (LD50) at which provisions of that putative Annex were to begin to apply. The problem recognised in discussion was that substances just slightly above that cut-off lethal dose would nevertheless be ignored for the purposes of inclusion in the Schedules.

5. The flow chart suggests that there could be values of X and Y agreed at levels above and below which, respectively, there should be no uncertainty about the risk posed by the substance names. Substances with potencies between the values of X and Y would be subject to further scrutiny for risk to the Convention based upon criteria not related solely to biological potency. This approach should provide a more flexible and realistic method of assessment of whether a new substance should be added to the Schedules.

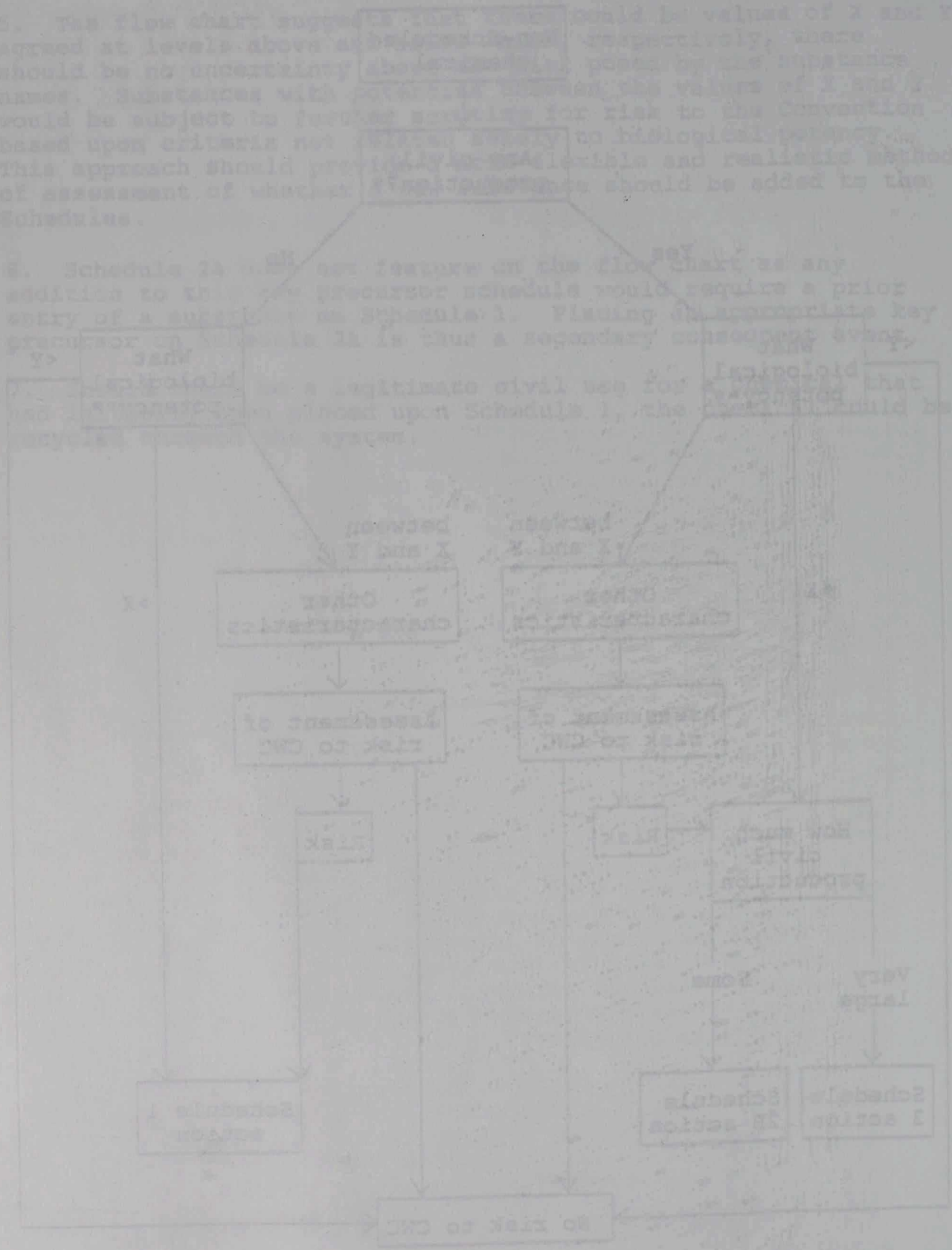
6. Schedule 2A does not feature on the flow chart as any addition to this key precursor schedule would require a prior entry of a substance on Schedule 1. Placing an appropriate key precursor on Schedule 2A is thus a secondary consequent event.

7. Should there be a legitimate civil use for a chemical that had initially been placed upon Schedule 1, the chemical could be recycled through the system.



* Intentional or otherwise

** X and Y are measures of biological potency. For each new substance they would be measured by parameters appropriate to its perceived military application.



CONFERENCE ON DISARMAMENT

CD/CW/WP.297
20 June 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

FINLAND

Provision of data relevant to the Chemical Weapons Convention

In order to contribute to the negotiations on the Chemical Weapons Convention, Finland presents below data relevant to the future Convention in accordance with the format proposed in document CD/828 of 12 April 1988.

The data provided is based on information collected with the co-operation of the Finnish Chemical Industry Federation, National Board of Labour Protection (its Register of Hazardous Chemical Products) and the Finnish Defence Forces.

The survey includes the chemicals listed in Schedules 1, 2 and 3 in document CD/961 of 1 February 1990 and reflects the situation in Finland in the first half of 1990.

Schedule 1 chemicals were produced for protective purposes in quantities above 100 grams/year at one single laboratory.

For the purpose of this declaration the threshold for schedule 2 and schedule 3 chemicals has been established at 1 tonne/year. The data on these chemicals appear in Tables 2 and 3.

Table 1

1.	Presence of CW on own territory	None
	Possession of CW on territory of another State	No
2.	Aggregate number of facilities for the production and storage of CW	None
	Aggregate number of facilities for the producton, processing and consumption of chemicals on Schedules 1, 2 and 3	4 */
3.	Types and names of CW agents	None
	Types of CW munitions stored; CW agents stored in bulk	None
	Number and names of chemicals on Schedules 1, 2 and 3 produced in the chemical industry	None
4.	Plans and methods for the destruction of CW	None

*/ The aggregate number of facilities does not include the single laboratory synthesizing schedule 1 chemicals for protective purposes.

Table 2

Schedule 2 chemicals	No. of companies	
	Import	Production
N,N-Dimethyl aminoethyl-2-chloride	1	0
N,N-Diisopropyl aminoethyl-2-chloride	1	0

Table 3

Schedule 3 chemicals	No. of companies	
	Import	Production
Phosphorus oxychloride	3	0

Norway	CD/CW/WP.298	Use of sorbent extraction in verification of alleged use of chemical weapons	Also issued as CD/1008 26 Jun. 90

NOT REPRODUCED
(see WP volume)

* * *

	CD/CW/WP.299	Withdrawn

CONFERENCE ON DISARMAMENT

CD/CW/WP.300
27 June 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

UNITED STATES OF AMERICA

Revisions to Article VI, Permitted Activities

"2. Toxic chemicals and their precursors listed in Schedules 1, 2A, 2B and 3 in the Annex on Chemicals, which could be used for purposes prohibited by the Convention as well as facilities which produce, process or consume, or which are capable of producing these toxic chemicals or precursors, shall be subject to international monitoring as provided in Annexes 1, 2, 3 and 4 to this Article ... ."

"3. Within 30 days of the entry into force, each State Party shall declare data on relevant chemicals and the facilities which produce them, or which are capable of producing them, in accordance with Annexes 1, 2, 3 and 4 of this Article."

"5. Each State Party undertakes to subject chemicals listed in Schedule 1 and facilities specified in Annex 1 to this Article to the measures contained in that Annex and in Annex 4."

"6. Each State Party undertakes to subject chemicals listed in Schedule 2, Parts A and B and facilities declared under Annex 2 to this Article to monitoring by data reporting and routine systematic international on-site verification, through on-site inspection and use of on-site instruments as long as production and processing are not impaired, as well as to ad hoc visits, in accordance with Annexes 2 and 4."

"7. Each State Party undertakes to subject chemicals listed in Schedule 3 and facilities declared under Annex 3 to this Article to monitoring by data reporting and ad hoc visits, in accordance with Annexes 3 and 4."

Additions to Article VI, Permitted Activities

(to follow current para. 7)

" . Each State Party undertakes to list all facilities which produce, process or consume any of the chemicals listed on Schedules 1, 2A, 2B and 3, or which are capable of producing them, in the National Register, and agrees to subject all such facilities to the procedures for ad hoc visits."

Ad Hoc Verification

1. The agreement shall not provide for access to or participation in the Ad Hoc Visit by any national of the State Party which selected the facility, except by consent of the host State Party. The selecting State Party shall make any requests for the presence of such a national representative directly to the host State Party.

On-Site Visits

2. The Technical Secretariat shall conduct all Ad Hoc Visits within [ ] hours of a request for an Ad Hoc Visit.

3. The Members of the Visiting Team shall, in accordance with the Facility Access Agreement executed for the visit:

- have unimpeded access to all areas that have been agreed for the visit;
- comply with the safety regulations at the facility;
- select items to be inspected;
- bring with them and use any agreed instruments to assist them during the visit;
- request and receive samples taken by representatives of the facility authority in the presence of the Team and the representative[s] of the host State Party;
- perform on-site analysis of samples;
- transfer, if necessary, samples for analysis off-site to a laboratory designated by the Technical Secretariat;
- afford the opportunity to the host State Party to be present when samples are analysed, whether on-site or off-site;
- ensure, in accordance with agreed procedures, that samples transported, stored unprocessed are not tampered with;
- in the event of ambiguities or anomalies, review records directly related to procurement of Schedules 1, 2 and/or 3 chemicals at the facility being visited, or other data the facility authority provides to address the ambiguities or anomalies; and

  communicate freely with the Technical Secretariat.

4. The host State Party for the Ad Hoc Visit shall, in accordance with the Facility Access Agreement executed for the visit:

- have the right to accompany the Visiting Team at all times during the Visit and observe all their activities at the facility;
- have the right to retain duplicates of all samples taken, as well as to be present when samples are analysed;

- have the right to inspect any instruments used by the Visiting Team and to have them tested in the presence of its own personnel;

- have the right to receive copies of the report of the Ad Hoc Visit, as well as copies of any information and data that was gathered on the facility visited; and

- have the obligation to assist the Visiting Team, upon its request, in collecting and analysing samples on-site;

- have the obligation to make available, in the event of ambiguities or anomalies identified by the Visitng Team, records directly related to procurement of Schedules 1, 2 and 3 chemicals at the facility or other data to address the ambiguities or anomalies.

Submission of Visiting Team Report

5. After each Ad Hoc Visit, the Visiting Team shall submit a report with the members' findings to the [Director-General of the] Technical Secretariat, which will transmit a copy of this report to the host State Party. The results of such visits shall be treated in the same manner as the results of other types of routine inspections.

6. The Visiting Team may request clarification of any ambiguities arising from the Ad Hoc Visit. In the event that any ambiguities arise which cannot be resolved in the course of the visit, the Visiting Team shall inform the [Director-General of the] Technical Secretariat immediately.

Consultation and Co-operation

7. Each State Party shall use the procedures specified in Article IX, including procedures for consultation, clarification, and Challenge Inspection, to resolve any concerns it may have about facilities that were not included in the National Register of any other State Party.

8. In accordance with Article IX, each State Party shall be obligated to co-operate in resolving the matter.

AD HOC

Addition to Article VII, National Implementation Measures

(to follow current para. 1)

. Each State Party shall maintain a National Register of all relevant civil or military facilities */ on its territory which are capable of producing any of the toxic chemicals or precursors listed on Schedules 1, 2A, 2B and 3 in the Annex on Chemicals. The National Register shall also include all facilities which have been declared under Annexes 1, 2 or 3 to Article VI.

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*/ The characteristics of chemical production facilities should be set forth in the Annex on Chemicals.

AD HOC VISITS

Obligation and Frequency

1. An Ad Hoc visit may be requested by any State Party, or by the Technical Secretariat, of any facility included in any other State Party's National Register. Each State Party may request up to [ ] Ad Hoc visits each year. The total number of requests per year by the Technical Secretariat shall be limited to [ ]. Ad Hoc visits to facilities on the territory of any State Party shall not exceed [ ] per year.

Agreement on Facility Access for Visit

2. As for the other routine inspections, access to the facility selected for an Ad Hoc visit shall be mandatory and shall be provided within [ ] hours of notification of selection.

3. Immediately prior to each Ad Hoc visit, a Facility Access Agreement shall be signed among the facility's management or other entity responsible for the facility, the host State Party, and the Technical Secretariat.

4. Such agreement shall be based on a model agreement, but shall be tailored to govern the conduct of the Ad Hoc visit to the particular facility. At a minimum, it shall provide for visual inspection and sample collection, as well as specify any analysis procedures to be used.

CONFERENCE ON DISARMAMENT

CD/CW/WP.301
27 June 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

UNITED STATES OF AMERICA

Report on the Second United States Trial Inspection Exercise

Introduction

The United States Government, working together with the chemical industry, is continuing to develop inspection procedures which will contribute to monitoring and verification measures for a chemical weapons convention (CWC). The United States views these early trial inspections as the beginning of the process to refine the inspection procedure, not as a test of procedures that are close to final form. In February 1989, United States experts conducted a National Trial Inspection (NTI) at the facility operated by Akzo Chemicals Inc., in Gallipolis Ferry, West Virginia. On 20-21 March 1990, the United States conducted the second NTI at Alcolac Inc., in Baltimore, MD. The objectives of this latest NTI were to test updated routine inspection procedures, test new sampling techniques and various analytical options, consider process monitoring techniques, assess the extent of mandate necessary to ensure compliance and assess the number of inspectors required for an effective inspection team.

Alcolac Inc. produces thiodiglycol, Schedule 2 chemical, which can be converted to the Schedule 1 mustard agent in one step by reaction with hydrochloric acid. Thiodiglycol has numerous legitimate commercial and industrial uses and is produced by several manufacturers in the United States. It is widely used in textile dyeing processes, in elastomers, in stabilizers and antioxidants, as a lubricant additive, and as a solvent for ballpoint pen ink.

This inspection was characterized as a "routine inspection" which means the systematic, on-site inspection of declared facilities. The results of the second trial inspection are presented in this working paper, using the format elaborated during the Conference on Disarmament open-ended consultations in 1988 and CD/CW/WP.213. The results of the first United States National Trial Inspection were reported in CD/922, 22 June 1989.

A. GENERAL APPROACH

1. Objectives of the national trial inspection

The main objectives of this second trial inspection were as follows:

- to determine whether Schedule 1 chemicals were produced in this facility;
- to determine whether the declared thiodiglycol production could be verified and the resulting inventory accounted for;
- to determine whether the currently available sampling, instrumentation and analytical techniques are suitable for achieving the objectives of a routine inspection;
- to determine whether the procedures are adequate and effective for conducting a routine inspection;
- to consider whether continuous process monitoring instrumentation techniques could be used to augment or even substitute for routine inspection;
- to determine the time, team size and cost associated with routine inspections.

2. Provisions in the draft convention under which the national trial inspection took place

The national trial inspection was based on Article VI and on the provisions governing monitoring of Schedule 2 chemicals as set forth in Annex 2 to Article VI and "Model for an Agreement Relating to Facilities Producing, Processing or Consuming Chemicals listed in Schedule 2" contained in CD/961.

3. Type of on-site inspection

This inspection was a simulated routine inspection of a facility which would be declared as a Schedule 2 production facility under the proposed CWC and included an initial visit.

4. Advance information

(a) Declarations

The facility provded an initial declaration which included data for 1989 in the format specified in Annex 2 to Article VI.

(b) Agreement on inspection procedures

A facility agreement was negotiated during and after the initial visit. The agreement contained in CD/961 (pp. 161-166) served as the starting point for the drafting of the facility agreement with additions and changes as appropriate. The agreement included a "Premises Liability Release Agreement" which was required by the facility officials. The facility agreement was

broad in scope, allowing almost total access to the site and the plant records. This was deliberately done, as will be discussed below, because of the risk to the objectives of the convention posed by the relevant chemical and the characteristics of the facility.

5. Type of facility inspected

The declared facility inspected is a multipurpose industrial chemical facility using batch processing. It is part of a larger plant which contains many operating process units in two areas of a single building. The products produced at the site are chemical specialties for cosmetics, soaps and detergents and industrial uses.

6. Type of declared activity at the facility

The facility inspected declared the following activities:

- production of a Schedule 2 chemical, thiodiglycol;
- production of another chemical, 2-mercaptoethanol (2ME) which is made from the same raw materials as thiodiglycol;
- consumption of Schedule 2 chemical, thiodiglycol, to produce sulphone diglycol.

7. Actual activity at the facility

During the inspection, 2ME was being distilled and thiodiglycol was present in a storage tank and in 55 gal drums in two different warehouses. In the declared production reactor a non-related product was being produced.

The declared facility was found to consist of a production facility, three separate raw material and product storage areas, an analytical laboratory, an effluent treatment system and raw material and product storage tanks. The declared reactor was found to be used for the production of 52 different products in 1989.

B. DETAILED APPROACH

1. The inspection mandate

The inspection was governed by the facility agreement which specified the drawings and records to be examined, the equipment to be inspected and points where samples were to be taken. It incorporated, by reference, a separate document containing detailed inspection procedures for routine inspection of Schedule 2 facilities. The facility agreement was deliberately broad in scope because this site contained all of the necessary chemicals and equipment to produce mustard, a Schedule 1 chemical. It is believed that in this type of facility, the Technical Secretariat must have the authority to broaden the mandate to ensure compliance.

2. Composition of the inspection team

The inspection team was composed of:

- Team leader: a chemical engineer experienced in production of Schedule 1 and 2 chemicals from the United States Arms Control and Disarmament Agency (ACDA);

- three chemical engineers knowledgeable in production of Schedule 1 and 2 chemicals, one from ACDA and two from the United States Department of Defense (DOD);

- a physicist from DOD;

- an analytical chemist from DOD;

- two inspector assistants from DOD.

3. Inspection equipment

Inspection equipment consisted of sampling bottles, sampling pumps and absorption tubes, size measuring equipment (tapes and rulers) and safety equipment. Safety equipment included hard hats, safety glasses, safety shoes or boots and escape respirators.

4. Activities prior to the arrival of the inspection team on-site

Inspection procedures applicable to any Schedule 2 facility were developed by a controller group, based on provisions of Annex 2 to Article VI and Protocol on Inspection Procedures in CD/961 (pp. 117 to 149). A facility agreement and detailed inspection plan were then prepared, based on the initial declaration, the initial visit and the general procedures.

The exact inspection date was established four weeks in advance with the facility officials. A date was selected when the facility would not be producing thiodiglycol.

5. Advanced preparations on-site

No special preparations were made on-site.

6. Escort and points of contact arrangements

Facility personnel served as escorts. The Alcolac Director, Environmental Health and Safety Affairs was the designated point of contact at the site. No entry/exit procedures at a point of entry were included in the scope of the inspection.

7. Other participants

The trial inspection exercise was managed by an inter-agency group with representatives from ACDA, the Office of the Secretary of Defense, the Office of the Joint Chiefs of Staff, the State Department, the On-Site Inspection Agency and other interested agencies. This group included several members of the United States chemical weapons convention negotiating team. Members of the inter-agency group participated in the trial inspection as observers,

along with a representative of the Chemical Manufacturers Association (CMA). CMA is a non-profit trade association representing a major portion of the basic chemical manufacturing capacity in the United States. CMA served as a liaison in arranging this beneficial trial inspection between the chemical industry and the United States Government.

8. Duration of initial visit and inspection (Based on 9 hours/man day)

- initial visit: 3 days (18 man-days);
- preparation of facility agreement: 12 man-days;
- inspection: 2 days (17 man-days);
- NTI inspection report preparation: 30 man-days.

9. Measures to protect confidential business information (CBI)

It was agreed that all information to which Government officials were given access would be treated as CBI. A special repository was established at the site for sensitive documents and drawings used by the inspection team. Some CBI data computations and sketches were removed from the site for the inspection report. No CBI facility drawings, documents or operating procedures were removed.

10. Opening Conference

During the opening conference, the inspectors established their credentials and specified their planned activities. A facility representative provided a safety briefing. The conference was short because the same team had conducted the initial visit about six weeks before and participated in the facility agreement negotiations. The opening conference required about 15 minutes. A "normal" opening conference should entail about one hour.

11. Types of records needed and/or audited

Calendar year 1989 records were required for material balances involving thiodiglycol and 2-mercaptoethanol production and hydrogen sulphide usage. Railcar logs, inventory details, batch book (daily logs), production sheets (with analysis) and shipping papers were needed. Sulphone diglycol production and scrubber liquor log sheets and analyses for sulphide were required. Back-up records to verify information above were audited including: material transfer sheets, warehouse records, product shipping records, suppliers invoices, bills of lading, sales records, waste manifests, freight bills and weigh tickets.

Because of the concern for the possible production of mustard, chlorine-containing compounds (chlorosulphonic acid, hydrochloric acid and by-products) were also audited and material balances computed.

The hydrogen sulphide data would provide a basis for estimating thiodiglycol production and key raw material usage. The back-up records should provide the basis for estimating inventory and disposition of thiodiglycol. Any variances in production and inventory would be helpful in

establishing potential for disparity or diversion of Schedule 2 material for prohibited purposes. The chlorine computations would provide information on the possible production of Schedule 1 chemicals.

12. Plant orientation tour

Immediately after the opening conference, the entire inspection team would normally tour the areas subject to inspection. Because this team toured the facility during the initial visit, the orientation tour was omitted.

13. Inspection of areas and facility equipment

Immediately following the opening conference, the two gate inspectors were deployed to ensure all gates were closed and sealed except the two gates they would be monitoring. They recorded the type of vehicle, contents, weight in/out and purpose of visiting the site. Although no samples of contents were taken, the inspectors should be authorized to verify what is entering or leaving. Any thiodiglycol departing should be sampled to ensure this is not another substance while the actual thiodiglycol has been diverted elsewhere. Samples should also be taken to ensure Schedule 1 chemicals are not being removed from the site. Similarly, raw materials entering should be substantiated to ensure no key precursor, such as hydrogen sulphide, is entering the plant mislabelled.

Equipment inspection started with the survey of the piping and instrument diagram (PID) drawings. These drawings which were not available during the initial visit were essential to understand the process and its controls. These were followed as the basis for the size of the equipment, materials of construction, controls and interconnection of the various components. They displayed how each vessel was constructed with every pipeline and control system identified. The drawings were made a part of the inspectors security file for maintaining a record of the equipment as it was presently configured.

After thoroughly reviewing and understanding the PID drawings, the equipment inspection was started in the raw material storage areas where the key feedstock is handled. Hydrogen sulphide is received in 22,000 gal railcars and transferred under its own pressure to the reactor (3MR). The line was traced carefully and found to go only to 3MR through various flow controllers, vaporizers and a venturi mixer. It would be possible to pipe this material to other reactors in the facility, but there was no evidence that this had ever taken place. Alcolac stated that 3MR is the only reactor where hydrogen sulphide is used and only in the production of thiodiglycol and 2ME. The other raw material is received in approximately 24,000 gal railcars, but is pumped to a storage tank. This material is only used in this facility, but in three possible reactors (including 3MR) and for the production of many other products in addition to thiodiglycol and 2ME. The inspection efforts concentrated on the 3MR reactor system and all equipment connected to it. This included controls, holding tanks, storage tanks, pumping systems and venting systems. An associated still pot for distilling 2ME was also inspected. Physical measurements were made on several of the vessels to assist in verification of the actual size and volume of the equipment and comparison with the PID drawings.

It was found that by the adaptation of hoses and use of various piping systems, the thiodiglycol could be pumped to any reactor in the plant. Therefore, the inspection also included the undeclared surfactant production area of the plant where chlorosulphonic acid is used as a raw material and by-product hydrochloric acid is generated.

Visual observations of the two product storage warehouses and effluent treatment facility were made. Drums of thiodiglycol were counted to assist in the inventory. In addition, samples were taken from these areas to validate chemical content and seek possible evidence of the Schedule 1 chemical, mustard.

14. Inspection of operating procedure

Production and ancillary equipment in both operating areas of the plant were examined in detail for suitability for production of Schedule 1 chemicals. Particular attention was paid to presence/absence of equipment and safety devices for containment and handling of toxic chemicals.

Interviews were conducted with personnel involved in both operating areas to verify types of operations and degree of hazardous materials handled during their employment at the site.

15. Sampling and sample-taking procedures

Samples were taken by facility personnel as requested by the inspectors as follows:

- sample of the 3MR reactor taken at the pump sample port;
- sample of the digester/sweetening tank, T17;
- sample from the 2ME distillation unit;
- sample of contents of pilot plant reactor;
- a gasket from the 3MR eductor mixing loop.

Additional samples were taken by the inspectors as follows:

- vapor samples above and under the 3MR reactor;
- vapor samples in the lower surfactant area;
- wipe samples around the 3MR reactor system (five sample points)
- wipe sample from cover of storage vessel D-24;
- wipe samples from the three plant drumming stations;
- wipe sample from the surfactant floor drain;
- wipe samples from the pilot plant and an R & D lab hood;
- water samples from four points in the effluent treatment system.

16. Handling of samples

Each sample was given a code number, labelled and the data recorded in a log-book. The samples were maintained by the inspectors or locked in a cabinet. Later, in the facility laboratory, the liquid samples were opened, subdivided into four sample portions, relabelled and sealed. Care was taken to maintain a secure chain of custody for the samples at the facility. The samples were then shipped by commercial carrier from the facility to the off-site analytical laboratory.

17. Analysis of samples

On-site analysis: Samples of process liquids were analysed on site by facility personnel in the presence of inspectors. Gas chromatography (GC) was used.

The inspectors used Gas-Tek (TM) tubes to check for the presence of hydrogen sulphide, and chlorine vapours.

Off-site analysis: Process liquid, water, soil, wipe, and vapour samples were analysed off-site at United States Government laboratories. The followng analytical methods were used:

- gas chromatography (GC): for checking the on-site analysis (process liquid samples);

- gas chromatography - mass spectrometry (GC-MS): to identify chemicals present at trace levels (process liquid, water, soil, wipe, and vapour samples);

- high performance liquid chromatography (HPLC): to identify trace chemicals in aqueous solution (water, soil, and wipe samples).

18. Purpose of analyses

Samples were analysed for the purpose of attempting to detect an indication of the presence of the Schedule 2 chemical, thiodiglycol in undeclared areas. The Gas-Tek and vapour samples were taken to check for the presence of the starting materials needed for the production of thiodiglycol. The process liquid, water, soil, wipe, and vapour samples were analysed to check for the presence of thiodiglycol. Representative samples of each type were analysed for mustard agent.

19. Documentation of the Inspection

The trial inspection was documented through still photographs of the site and notes taken by the inspectors and observers.

20. Evaluation by inspectors

The inspectors' evaluations covered the following aspects:

- deviations from initial plans;

- problems encountered;

- usefulness of inspection procedures;

- results of the inspection;

- conclusions that could be drawn about the facility's activities;

- matters or concerns which should be brought to the attention of the CD.

21. Closing Conference

During the closing conference, the inspectors reviewed their activities and indicated their findings. This conference required approximately 40 minutes.

22. Anomalies, disputes and complications

Several discrepancies in the initial declaration were resolved in the initial visit. However, during the routine inspection, a new set of computer production records was provided which was never totally satisfactory. The records audit inspectors resorted to reviewing the production log sheets for every day of the year on which the 3MR reactor was operated. This was a more valuable and accurate source of data but also served to identify types of discrepancies which experienced inspectors will have to be prepared to deal with. For example:

- inclusion of a 6,600 pound "heel" in one lot which was included in new production;

- blending of lots and drums which should not have been reported as new production;

- readjusting the starting raw material weight expenditures after weighing the drummed product. (Actually the drummed product weight was the most accurate basis for accounting for the raw material usage. The feed totalizers were not as accurate as "advertised" by their manufacturer.);

- lots of 2ME were sometimes redistilled and blended which should not be reported as new production.

There were other complications. The railcars could not be weighed at this plant. Therefore, the exact usage of hydrogen sulphide is not known until the car is returned to the supplier, weighed to determine the tare, and the invoice returned to the plant. This process can take several months.

Another source of dispute arises if the plant records and the inspectors' calculations are not in precise agreement. Most discrepancies were resolved to within 1 per cent which may seem reasonable. However, if the discrepancy is 1 per cent of 1 million pounds, the actual quantity is 5 tons of key precursor which could be considered significant.

No anomalies were intentionally introduced.

23. Report of the inspection team

The inspectors' report was prepared during the weeks following the inspection as a part of a detailed evaluation of the trial inspection. The inspection team report fully documents all activities and findings of the second NTI exercise.

24. Impact of the inspection on facility operations

Total costs to the facility were estimated at $39,500 based primarily on the time and effort required of the facility officials for preparation of the initial declaration, negotiation of the facility agreement and participation in the trial inspection. The inspection had minimal impact on plant operations.

C. RESULTS

1. Inspection team rights

The facility officials required the inspectors to sign a "Premises Liability Release Agreement". This released Alcolac of any responsibility due to death, disease, illness or injury as a result of being exposed to safety hazards at the facility and other similar indemnifications as a result of the inspection. This issue must be dealt with by the Convention or by the State party implementing legislation to ensure that the inspectors' legal rights are protected.

2. Inspection team mandate

As a result of the initial visit it became obvious that facilities of this type require an extensive monitoring/inspection régime. All of the chemicals and equipment were available at this site to produce mustard, a Schedule 1 chemical. The negotiated facility agreement was deliberately broad in scope to allow the inspection team to "search" the entire site to ensure compliance. There was some criticism of the agreement indicating it was not realistic. It is believed that the Technical Secretariat must have the broad mandate in a facility where the declared material is one step away from a Schedule 1 chemical, to acquire the widest latitude in the subsidiary arrangements for inspection of the site. The alternative would be to have more frequent inspections or full-time inspectors at such a facility. In either situations, there may be difficulty in obtaining such comprehensive access.

For the long range, the use of tamper resistant continuous process monitoring instrumentation would minimize the intrusiveness and the frequency of routine inspections at such facilities.

3. Inspection planning

One of the conclusions of the first NTI was the importance of the initial visit and good planning. This lesson was demonstrated during this exercise. Because of the comprehensive initial visit and facility agreement, the actual routine inspection was expeditiously completed with few deviations from the initial plans. Even though considerable problems were encountered in the

records audit, the inspection was completed in two days (9 hours/day). This was also facilitated in time and accomplishment by familiarity of the inspectors with the site and full co-operation of the facility officials.

4. General inspection approach

The first NTI demonstrated that inspection visits and material balances cannot provide complete assurance that the quantity or type of Schedule 2 chemicals produced is correctly declared primarily because the inspectorate does not have an independent means of monitoring plant operations. These measures could be circumvented by fabricated records or simply by not recording any data in the permanent books of the facility. Not recording receipt of raw materials, plant operations or product produced would falsely indicate that the equipment was idle. This exercise demonstrated that poor data or record errors can lead to confusion or inconclusive resolution which could result in a large variance.

To ameliorate both of the above situations, consideration needs to be given to development of simple, tamper proof, reliable instruments that could monitor the process equipment on a continuous basis. The declared reactor 3MR was used to produce 52 different products in 1989. The process variables of temperature, pressure, reaction time, reactants, concentrations and mixing options as well as the composition of the output are parameters that may be monitored. An instrument that can monitor a combination of these key variables and be specific as to what, when and how much is being produced would be most valuable between inspections. The United States is initiating a programme for assessment of this type of monitoring equipment.

The trial inspection demonstrated that a combination of equipment inspection, records audit and sample analyses are all essential components of an effective inspection régime. The exercise showed that the inspection procedures are generally effective for conducting a routine inspection.

5. Equipment inspection

Examination of the equipment together with a knowledge of the chemicals being produced at this facility, showed that Schedule 1 mustard could be produced in several of the operating areas. Visual examination is not sufficient to determine whether such chemicals have been produced in the past. (Sample analysis may help to some degree in this aspect.) Equipment inspection discloses much about the potential ability of the facility and is also a basis for determining the production capacity. Equipment inspection combined with types of products produced, enables the inspectors to determine the capability of the facility to produce illicit chemicals. From this information, the Technical Secretariat has the basis to negotiate a broad scope for future inspections, commensurate with these factors. Also, the frequency of visits which should be based on the potential for engaging in prohibited activities will be determined using these observations of production capability, production capacity and proximity to Schedule 1 chemistry.

During the initial visit, piping and instrumentation diagrams (PID) were not available. The latest set of drawings (12 March 1990) were available for the routine inspection and represented the "as-built" equipment. These were extremely helpful in understanding the process, determining the size and

materials of construction of equipment and explaining the instrumentation and controls of the entire process. This information is absolutely essential to an inspection team and should be available and up to date. Establishing a standard format for PID drawings which would be available at all Schedule 2 producers' facilities would greatly assist all inspection teams and save time, effort and cost. Even where language differences are a problem, the use and availability of standard drawings would enhance the inspection.

6. Records audit

Chemical production practices generate a multitude of interlocking records that can be audited as a means of monitoring declared chemical production. However, the data available can have its limitations, particularly if it is not timely, has errors or creates confusion. With respect to this trial exercise, there were considerable problems even though we had the full co-operation of the facility officials. The Company had recently changed to a new computer system for their inventory. The change was initiated during the initial visit and was still being worked on when the NTI was conducted six weeks later. Much time was spent trying to sort out computer runs before it was concluded that it was a wasted exercise. The alternative was to go to the daily plant log sheets and other daily records and reconstruct what took place in reactor 3MR for the entire year. Another related issue was that the Company produces two chemicals, thiodiglycol and 2ME, from the same raw materials. This meant that both materials had to be totally accounted for to verify declared thiodiglycol production. Of particular interest for future inspections is the fact that daily logs contain errors, are sometimes incomplete and are prepared by different employees who interpret guidance slightly differently, all of which must be sorted out. This resulted in more effort than planned which was resolved by enlisting several of the observers to participate in the auditing process.

The records auditing process is one of the elements used for examining declarations of the production taking place at the facility. When language differences and format differences are added to normal errors that will always be present in records, the problem of resolving and accounting for a year's activity is formidable. A recommended solution to this problem is to establish model documents for uniform record keeping for all Schedule 2 producers. The format could be developed in consultation with the chemical industry and its associated trade associations for world-wide use.

Another area of concern is the ultimate disposition of high risk Schedule 2 chemicals. Thiodiglycol is one step away from mustard agent. The routine inspection will only examine and calculate quantities declared and produced in a given period. It provides no information on the distribution and subsequent use of this chemical. To effectively achieve the treaty objective of detecting prohibited use of thiodiglycol, it would seem that the tracking of this chemical to its ultimate destination should be part of the inspection procedure. This would increase the intrusiveness of the inspection but seems warranted for certain listed chemicals, such as thiodiglycol. Tracking the distribution would create additional burdens for the Technical Secretariat to trace the shipments to their destinations.

With respect to this trial inspection, the records audit produced an inventory and production accountability within 1 per cent. Based on the key feedstock, a closure of 0.8 per cent was found. The maximum production capacity was verified. The records were consistent and indicated that thiodiglycol production had been fully accounted for. The recorded production and consumption were essentially the same as the declared production within the 1 per cent error limits allowed in measuring the weight of feedstock and products. This seems reasonable agreement. However, with respect to larger production quantities, for example, 1 per cent of 1 million pounds could result in as much as 5 tons of product being unaccounted for. For Schedule 2 chemicals, such as thiodiglycol, agreement should be reached on quantities which would be considered significant if unaccounted for.

7. Sample Analysis

The first NTI concluded that samples should be analysed off-site "to obtain the most precise and quantitative results". However, if analytical instrumentation is available or can be developed to bring with the inspection team, a number of problems could be avoided and the inspection could be achieved most expeditiously. More samples could be taken, the data would be timely, the inspection would be less intrusive, samples would not have to leave the premises and many sample handling and shipping problems could be avoided.

Discussions with instrumentation experts indicate that considerable progress is being made in the development of analytical equipment that is portable and could be carried in a suitcase. Sophisticated analytical equipment, manned by experienced analysts and brought to an inspected facility, could provide an adjunct to off-site analysis of samples. The equipment would produce quick results and could possibly minimize or eliminate the need to ship samples off-site for some inspection scenarios. The United States has a programme to thoroughly investigate this area.

Water, wipe and soil samples which now require off-site work-up, do not pose the same handling problems and are taken for other reasons. The vapour and liquid process samples reveal the real time activities, whereas, the water, wipe and soil samples provide information on past practices. Both are equally important, but the process samples' identifications are more timely in completing the immediate objectives of the routine inspection. Analysis of the other samples off-site will provide the Technical Secretariat with information that, if "positive", will have to be dealt with in a different manner.

This inspection reinforced many of the conclusions drawn from lessons learned during the first NTI experience. It was demonstrated again that sample taking, sample preparation and shipment, and sample analysis require considerable planning and expertise. The facility agreement should specify what samples will be taken and the sampling locations. The agreement should also provide for optional or random sampling of the vessels interconnected with the declared reactors to ensure that the inspection is not totally predictable. Planning for tamper-proof, safe packaging and shipment of samples to the Technical Secretariat's laboratory under both the State party and international laws must be well thought out and standardized to avoid problems.

Once the samples are obtained, a continuous chain of custody must be maintained until they are analysed. Tamper-indicating seals should be applied and the samples must be properly labelled. A log must be maintained to identify the samples' sources. A coded numbering system should also be utilized to protect the identity of the producer once the samples have left the site.

Because the Schedule 2 chemical, thiodiglycol was not in production during this NTI, all samples were analysed for the purpose of attempting to detect an indication of prior production. Thiodiglycol was found in a liquid sample of the reactor contents (different product) taken one week after thiodiglycol production in wipe samples taken at the drumming station, and in the effluent water leaving the site. Hydrogen sulphide, the key starting material, was found in the air samples taken one week after thiodiglycol production. Representative samples of each type were analysed for mustard agent. None was detected. All sample types were useful in determining what other chemicals might normally be present.

8. The inspection team

Similar to the first NTI, this exercise demonstrated the need for extensive expertise in chemical engineering, chemical production and analytical chemistry. Even for a relatively simple process, the minimum number of inspectors is six based on the three pairs of inspectors concerned with equipment, records and sampling respectively. It also has become apparent that there should be a separate team leader who must resolve issues and keep the inspection process moving forward. There is also a need for various assistants, such as interpreters, analytical assistants and gate monitors. The manning of the team should be based on conducting the inspection in about three days (minimum 9 hour working days). This should be feasible after a comprehensive initial visit and a good facility agreement.

The inspection team should personally gather the data which is necessary for verification, such as, counting drums, watching sampling of tanks, checking labels, randomly checking drum contents and reviewing the daily production logs. On-site analysis can contribute to timely completion of an inspection and the teams should be equipped to accomplish this task. In a large complex site with numerous separate plant areas, the team should be equipped with the necessary location-finding equipment to be sure they are in the correct location. This could also be confirmed by photographs obtained during an initial visit to the declared facility.

9. Confidentiality

Protection of confidential business information must be guaranteed to the maximum extent possible consistent with verification requirements. During the inspection of a broad mandate multipurpose facility, such as this thiodiglycol plant, a substantial amount of proprietary information must be disclosed. Both thiodiglycol and 2ME production were examined, but in addition, information on their other products was necessary to determine the use of chlorosulfonic acid and the hydrochloric acid produced. Investigating the possibility of thiodiglycol and a chlorinating material being utilized to make mustard was the primary concern of the inspection. Therefore, considerably

more of this site had to be "opened" for the inspection than if the Schedule 2 product were two or three steps away from a prohibited Schedule 1 chemical. The inspection of this type facility is intrusive, but the use of this information should be managed such that access is only available to the inspectors on the site.

Another difficulty related to a material such as thiodiglycol is its ultimate destination. The Technical Secretariat must know where and how it is being used. Access to the list of users appears to be consistent with the risk of this commodity to the convention.

10. Areas requiring further work

This inspection has continued to make clear the need to conduct additional trial inspections in the chemical industry as well as at government installations. Routine, challenge and ad hoc inspections must be practised to determine how best to conduct an inspection as well as to learn what security and proprietary information is subject to possible loss.

A need exists for development of technology for portable, real time, sensitive analytical equipment and continuous on-site process monitoring instrumentation.

There is a need for the development of standard PID drawings of facility equipment and model documentation, format and records for auditing purposes.

Inspection of this type of facility will require extensive access to and use of confidential business information. It is necessary to develop reliable means to protect the CBI as well as classified State party information.

11. Conclusions

It is concluded from this inspection that the Technical Secretariat will be inspecting a limited number of facilities in the civil chemical industry which will demand a broader mandate. This facility was an excellent example, where both the chemicals and the equipment were present to produce a Schedule 1 chemical. Extensive freedom at the site is required for verification. Access to the ultimate destination of the Schedule 2 chemical is warranted to assure no prohibited Schedule 1 chemicals are being produced.

CONFERENCE ON DISARMAMENT

CD/CW/WP.302
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Ad Hoc Committee on Chemical Weapons

THE NETHERLANDS

Analytical chemical results of the second trial inspection on verification of non-production of chemical warfare agents in a civil chemical industry in the Netherlands

SUMMARY

At the request of the Ministry of Foreign Affairs the Prins Maurits Laboratory TNO (PML-TNO) participated in a trial inspection of a civil chemical industry in the Netherlands. The purpose of the inspection was to check the procedural as well as the analytical chemical aspects and to report the results in a working paper to the Conference on Disarmament in Geneva. PML-TNO took part in the inspection to support the analytical chemical aspects.
The inspection was divided into two parts:
- a routine inspection to prepare the inspection procedure in consultation with the facility management. The declared data have to be verified by routine checks.
- an unannounced (ad-hoc) inspection of a whole industrial complex to verify the non-production of chemical warfare agents.

In the routine inspection the chemical process to produce a steroid-intermediate was checked. In this process triphenylmethylphosphonium bromide (TPMPB) is used as a reagent. This PCH_3-containing compound is a schedule [2] compound (precursors of chemical warfare agents). A number of samples were taken from the chemical process for analysis at PML-TNO. From the results it could be derived that the declared transformation of TPMPB into triphenylphosphinoxide (TPPO) was only partly correct.

In the ad-hoc inspection a number of water samples and atmospheric samples were taken at relevant locations in the industrial complex. The water samples were analysed at PML-TNO to determine the occurrence of PCH_3-containing compounds. Additionally, the Water Testing Kit was used to determine the presence of chemical warfare agents.

Both in the routine inspection as well as in the ad-hoc inspection atmospheric samples were analysed on application of the Gas Reconnaissance Kit to determine the presence of chemical warfare agents.

CONTENTS

1 INTRODUCTION

In the Netherlands a national trial inspection under the direction of the Ministry of Foreign Affairs was carried out in the first half of 1989. The multipurpose production installation inspected was part of a complex preparing pharmaceutical products.
The scope of the inspection was divided into two parts:

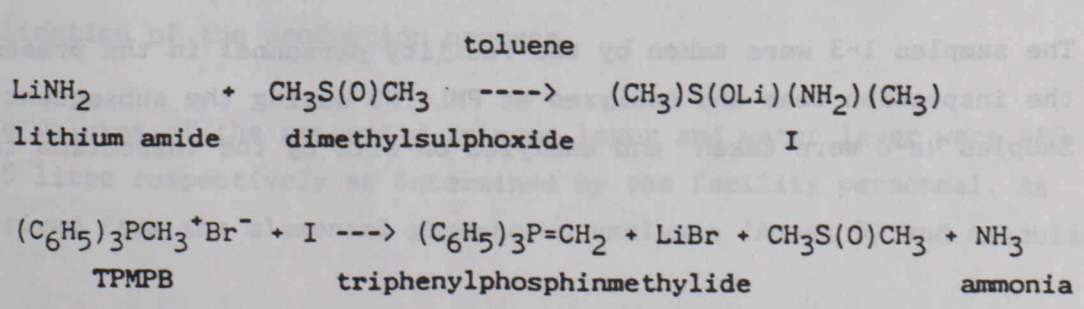
- a routine inspection to verify that the processing of the Schedule [2] chemical triphenylmethylphosphonium bromide (TPMPB) is consistent with needs for non-prohibited purposes;
- an unannounced (ad-hoc) inspection of the whole production complex to verify non-production of representatives of the categories 1-6 on Schedule [1] and chemicals 1-3 on Schedule [3].

This report contains the analytical chemical results of the routine and the ad-hoc inspection separately. Parts of these results will be incorporated in documents to be published as CD/WP-papers by the Ministry of Foreign Affairs.

2 ROUTINE INSPECTION

2.1 Objective of the inspection

The production installation is used for a batch process transforming a ketonic pharmaceutical intermediate into the corresponding methylide. According to the declaration of the facility management TPMPB is transformed into triphenylphosphinoxide (TPPO). The reaction scheme of the prccess including a so-called Wittig reaction is as follows:



$(C_6H_5)_3P=CH_2$ + keto-steroid ---> $(C_6H_5)_3P=O$ + methylide-steroid
TPPO

$(C_6H_5)_3P=CH_2$ + H_2O ----> TPPO + CH_4
excess water methane

The objective of the inspection was to verify the above-mentioned declaration.

2.2 Sampling

Samples

1 About one gram of the starting material (137 kg) TPMPB was taken to verify the declared identity

2 About one gram of the starting material (7,6 kg) $LiNH_2$ was taken to verify the declared identity.

3 A quantity of 3-4 ml of the toluene layer and water layer of the reaction mixture were taken after the reaction had taken place to validate the declaration of the reaction process.

4 The atmosphere in the facility was sampled at three locations to verify the non-production of representatives of categories 1-6 on Schedule [1] and chemical 1-3 on Schedule [3]:

a. right over the reaction vessel in which TPMPB was processed;

b. inside an enamel vessel close to the before-mentioned reaction vessel;

c. inside a separate part of the building to be entered by passing a lock. In this room airtight vessels and related safety utilities were used.

The samples 1-3 were taken by the facility personnel in the presence of the inspection team and analyzed at PML-TNO during the subsequent days. Samples 4a-c were taken and analyzed on-site by the inspection team.

2.3 Inspection equipment

For on-site analysis of samples 4a-c the Dutch Gas Reconnaissance Kit was used to detect representatives of categories 1-6 on Schedule [1] and chemicals 1-3 on Schedule [3].
A more detailed description of this kit is given in Annex I.

2.4 Analytical equipment

Sample 1 (TPMPB) and sample 3 (toluene layer and water layer) were analysed by means of mass spectrometry (MS).
Sample 2 ($LiNH_2$) was analysed by means of X-ray diffraction (XDF).
Sample 3 (toluene layer and water layer from the reaction mixture) was analysed by means of gas chromatography in combination with flame ionisation (GC-FID) and phosphorus specific NP-detection (GC-NPD). The identity of the volatile components was determined by gas chromatography using reference substances as well as in combination with mass spectrometry (GC-MS). The total phosphorus content was determined by a wet chemical colour reaction (elemental-P).
The principles of the different analytical techniques are given in Annex 2 for MS, Annex 3 for XDF, Annex 4 for GC-FID, GC-NPD and GC-MS, and Annex 5 for elemental-P.

2.5 Results and discussion

Starting materials

The mass spectrometric analysis (Annex 2) of the sampled starting material TPMPB and the X-ray diffraction analysis of $LiNH_2$ (Annex 3) verified in the light of additional data derived from the inspection sufficiently the declared identity of the respective compounds.

Validation of the production process

The volumes of the separated toluene layer and water layer were 480 and 445 litre respectively as determined by the facility personnel. As derived from the elemental phosphorus analysis (Annex 5) and calculated

as the original starting material TPMPB these layers contained respectively :

toluene layer	31.5 kg	TPMPB
water layer	101.5 kg	TPMPB
total	133 kg	TPMPB

Based on the 137 kg amount of TPMPB added to the reaction vessel the above-mentioned total amount indicates a recovery of 97 %.

Gas chromatographic analysis (Annex 3) indicated the presence of mainly two "relatively volatile" phosphorus containing compounds both in the toluene layer and in the water layer. The compounds were triphenylphosphinoxide (TPPO) and diphenylmethylphosphinoxide (DPMPO). The toluene layer contained additionally only a small amount of triphenylphosphine (TPP). The identity of these compounds could be confirmed by gas chromatographic-mass spectrometry as well as by gas chromatographic analysis of reference compounds. The water sample contained another non-volatile compound identified by mass spectrometry as non-transformed starting material TPMPB.
Besides these qualitative results the respective amounts of the different phosphorus-containing components were calculated as derived from experiments with reference compounds.

toluene layer	TPPO	11	kg	calculated as TPMPB
	DPMPO	15	kg	"
	TPP	0,5	kg	"
water layer	TPPO	3	kg	"
	DPMPO	49	kg	"
	TPMPB	50	kg	"
	total	128,5	kg	"

This result indicates a recovery of approximately 94% based on the total amount of TPMPB added to the reaction vessel. It seems wise to present this result only as a quantitative recovery and not in the form of an exact value (e.g. 94%), because there appear some uncertainties which may give rise to inaccurate quantitative results:

- on continuing the gas chromatographic analysis the performances of the column decreased. An increasing peaktailing of the different

components was found probably due to non-volatile residues (e.g LiBr and TPMPB) present in the toluene and water samples deposited in the injection port and the capillary column.

- the exact composition of the reaction mixture was unknown. As a consequence it was impossible within the time-schedule of one week to carry out more accurate recovery experiments.

From the above-mentioned results it can be concluded that the essential part of the phosphorus content can be ascribed to the presence of three phosphorus-containing compounds instead of one declared by the management. Besides the declared endproduct (TPPO) of the production process, the by-product DPMPO was found as well as a considerable amount of non-transformed starting material TPMPB. The formation of DPMPO can probably be explained by a base hydrolysis (ref.1) of TPMPB.

Air samples 4a,b and c were analysed on-site by means of the Gas Reconnaissance Kit. The detection results were negative in all cases indicating the absence of concentrations of compounds belonging to categories 1-6 on Schedule [1] and chemicals 1-3 on Schedule [3] above the detection limits as given in Annex 1.

3 AD-HOC INSPECTION

3.1 Objective of the inspection

Test of procedural and analytical chemical aspects of an ad-hoc or unannounced inspection of the production complex to verify the non-production of representatives of categories 1-6 on Schedule [1] and chemicals 1-3 on Schedule [3].

3.2 Sampling

Atmospheric samples were taken and analysed on-site by the inspection team at the following locations:

1 inside an empty hydrogen fluoride resistant reactor;

2 right over a man-hole of a 4000 litre enamel reactor that was used to process acetic anhydride;

3 inside a man-hole of a storage vessel containing organic waste originating from the whole complex.

Two water samples (each composed of two bottles of 100 ml) were taken by the inspection team and analysed off-site at PML-TNO. The locations were:

4 outlet of waste water of the building in which the TPMPB processing was going on but at the moment of sampling devoid of related waste.

5 outlet of waste water originating from the whole complex.

3.3 Inspection equipment

For on-site examination of samples 1-3 the Gas Reconnaissance Kit was used to detect representatives of categories 1-6 on Schedule [1] and chemicals 1-3 on Schedule [3]. A more detailed description of this kit is given in Annex 1.

Samples 4 and 5 were investigated by using the Water Testing Kit developed by PML-TNO to detect representatives of categories 1-6 on Schedule [1] and some chemicals related to chemicals 1-3 on Schedule [3]. More details are given in Annex 1. Normally this kit is used on-site the sampling location but due to a crowded program during the on-site inspection the detection was carried out at PML-TNO the day after the inspection.

3.4 Analytical equipment

Samples 4 and 5 were analysed on application of micro liquid chromatography in combination with a phosphorus specific detector (Annex 6) to detect methylphosphonic acid and alkyl methylphosphonic acids related to nerve gases such as Sarin, Soman and VX. For the time being this method was not prepared to analyse pinacolyl methylphosphonic acid, the hydrolysis product of Soman. In this case liquid chromatography-thermospray-mass spectrometry was used (Annex 7).

3.5 Results and discussion

Samples 1-3 were investigated on-site with the Gas Reconnaissance Kit.

On sample location 1 (sample 1) an ambiguous result on the detection of cyanogene chloride was found. Two of the four atmospheric samples taken showed a faint positive detection; the other samples were negative. The management of the production facility stated to expect the presence of residual amounts of acetonitrile vapour which indeed showed a positive reaction later on in the PML-TNO laboratory. The other detection reactions were negative.

Sample 2 from the enamel 4000 l reaction vessel gave a positive nerve gas detection. However, according to the explanation of the management of the production facility cholinesterase inhibitors should be absent. In this case the positive detection was due to the presence of acetic acid formed from the acetic anhydride vapour at the sampling location. An excess of acid will interfere with the detection reaction because it gives rise to an unacceptable change of the pH (3.7) of the inhibition reaction medium. All other detection reactions were negative. At this sampling location it was found that some components of the detection kit were not resistant to the acidic vapour.
Sample 3 taken from the vapours above the organic waste gave in all cases a negative detection.

In the samples 4 and 5 methylphosphonic acid and a number of alkyl methylphosphonic acids could not be detected above the detection levels of 0.05 µg/l and 1 µg/l respectively (see Annex 6). In case of the waste water outlet of the whole production complex the sensitivity of the micro LC-method was decreased probably due to the presence of other anions (e.g. Cl^-). These anions interfere in the precolumn performance.

4 CONCLUSIONS

The first application of the Gas Reconnaissance Kit as well as that of the Water Testing Kit in the field of verification of non-production of compounds belonging to categories 1-6 on Schedule [1] and chemicals on Schedule [3] ran smoothly. Within a short time period a number of detection reactions were carried out and interferences could be accounted for. However, from these experiments carried out in an industrial environment it may be stated that additional research will be necessary to obtain a foolproof detection equipment. During sampling it may be advisable to have available a personel protection equipment.

The application of micro liquid chromatography in combination with flame photometric detection was introduced to replace the conventional PCH_3 verification procedure (ref.2). The method has better prospects because smaller amounts of samples are required, a more rapid analysis time is attained and a wider scope of PCH_3-containing compounds e.g. alkyl methylphosphonic acids can be detected besides methylphosphonic acid. From the experience derived from the results presented in this report it may be stated that more research is required but the main objectives of the micro liquid chromatography method are within reach.

The production process in which the Schedule [2] compound TPMPB is consumed could only be validated completely after the detection of two additional chemicals besides the declared transformation product TPPO. It was found that an extensive analytical equipment and experience were required. For this reason problems may be expected on-site the facility when analysing such mixtures sampled from chemical processes.

One week after the trial inspection the complete analytical results could be presented to the inspection team.

5 ACKNOWLEDGEMENTS

The author grateful acknowledges the technical assistance of M.S.Nieuwenhuizen, R.C.M.Olivier and H.F.G.Oudmayer (detection equipment), E.R.J.Wils, A.H.Hulst and J.van Laar (mass spectrometry), R.Eerligh, W.Duvalois, J.A.M.Romeijn and M.A.Schrader (X-ray diffraction, elemental-P), C.E.A.M.Degenhardt (gas chromatography), A.L.de Jong and Ch.E.Kientz (micro liquid chromatography), H.L.Boter for critical reading of the manuscript, and W.B. de Bruin for manuscript preparation.

6 AUTHENTICATION

Datum: juni 1989

A. Verweij
(Project Manager/Author)

7 REFERENCES

1.R.C.Harden, G.A.Harvey, and D.M.Rackham, J.High Res.Chrom.& Chrom. Comm., 11(1988)587
2.A.Verweij, H.L.Boter and C.E.A.M.Degenhardt, Science 204,616(1979

ANNEX 1

DESCRIPTION OF THE GAS RECONNAISSANCE KIT

The kit developed by PML-TNO was used to determine whether the surrounding atmosphere at the sampling location contains representatives of the chemical warfare agents categories 1-6 on Schedule [1] and the chemicals 1-3 on Schedule [3] above a threshold level. A number of consecutive simple wet chemical colour tests has to be carried out according to the instruction manual in the kit. The results concerning the presence of the chemical warfare agents are obtained within 15 minutes. The following minimum gas concentrations (mg/m3) can be detected:

Sarin	0.02	HCN	5
Soman	0.01	ClCN	1
Tabun	0.02	Phosgene	5
VX	0.01		
mustard HD	0.27		
Lewisite	3.5		

DESCRIPTION OF THE WATER TESTING KIT, CHEMICAL WARFARE AGENTS

The kit has been developed to determine whether water is fit for human consumption. By a number of consecutive simple tests, conclusions can be reached within 20 minutes. The following concentrations (in mg/l) can be detected:

Sarin	0.02	arsenics	1-2
Soman	0.02	CN	8
Tabun	0.04	ClCN	5
VX	0.02	Cl_2	5
Mustards HD	4	(pH	6.5-9.0)
HN-3	2		

ANNEX 2

DESCRIPTION OF MASS SPECTROMETRY

Principle

The components of the sample in very small amounts are introduced into an evacuated part of the instrument and subsequently ionised and fragmented by bombardment with high energy electrons. The resulting positive ions are removed. The negative ions are accellerated in an electric field and subsequently, depending on the respective masses, these ions are deflected in a magnetic field and detected. The resulting mass spectrum of a compound is very specific. The structure of compounds can be elucidated using comprehensive data storage and retrieval systems.

Result

The sample of triphenylmethylphosphonium bromide was analysed at a source inlet temperature of 300°C. The resulting mass spectrum was quit similar to the spectrum of triphenylphosphine (M=262) as published by H.Williams in J.Amer.Chem.Soc.,90(1968)966. This compound can be expected as derived from the pyrolysis of the phosphonium salt. At 350°C the spectrum of methylenetriphenylphosphine M=276 is predominantly obtained (Fig.1). The formation of methylbromide could not be ascertained. Only in the spectrum determined at 350°C ions of HBr (m/z=80, 82) were observed.

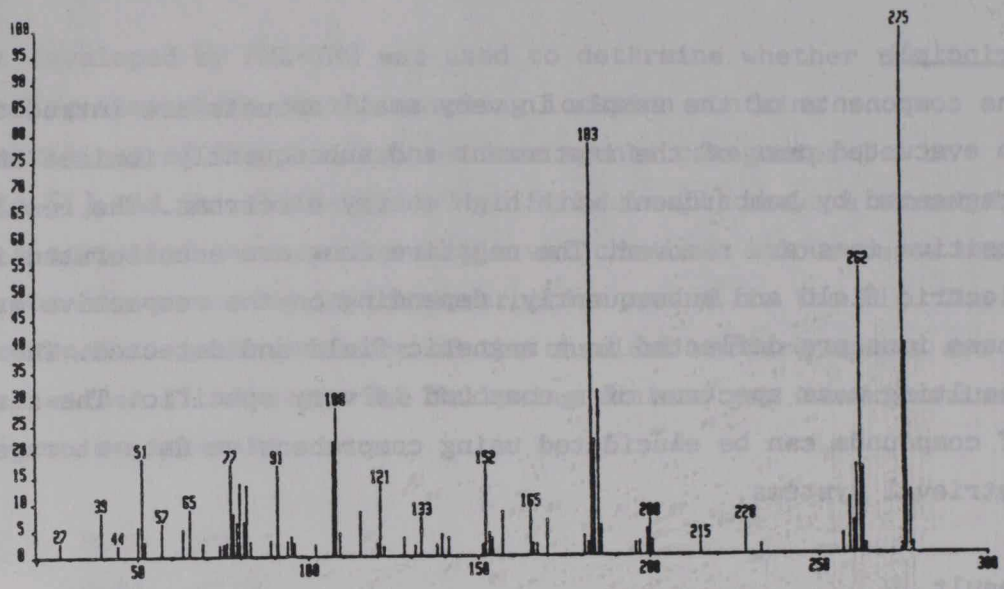


Figure 1 Mass spectrum of triphenylmethylphosphonium bromide sampled at the trial inspection. Mass spectrometer VG70-250S. Ion source temperature 350 C. x-axis = m/z; y-axis = intensity, peak at m/z = 275 normalized at 100%. Further details in text.

ANNEX 3

DESCRIPTION OF X-RAY DIFFRACTION

Principle

A solid sample is irradiated with a small band of parallel X-rays composed of one wavelength. The ray is diffracted at specific angles depending on the distance between the layers of atoms in the crystals of the sample. The angle of reflection is determined by an electric detector adjusted in a half circle around the sample. To elucidate the identity of the solid material the resulting spectrum can be evaluated using a comprehensive data storage and retrieval system.

Result

As can be derived from the spectra presented in Fig.1 the sample is composed of a mixture of lithium amide (6-418), lithium hydroxide hydrate (24-619) and lithium hydroxide (32-564). Both lithium hydroxide compounds are hydrolytic degradation products due to moisture in the atmosphere during sampling and storage of the solid material. The reference spectra are derived from the JCPDS-file (Joint Committee on Powder Diffraction Standards).

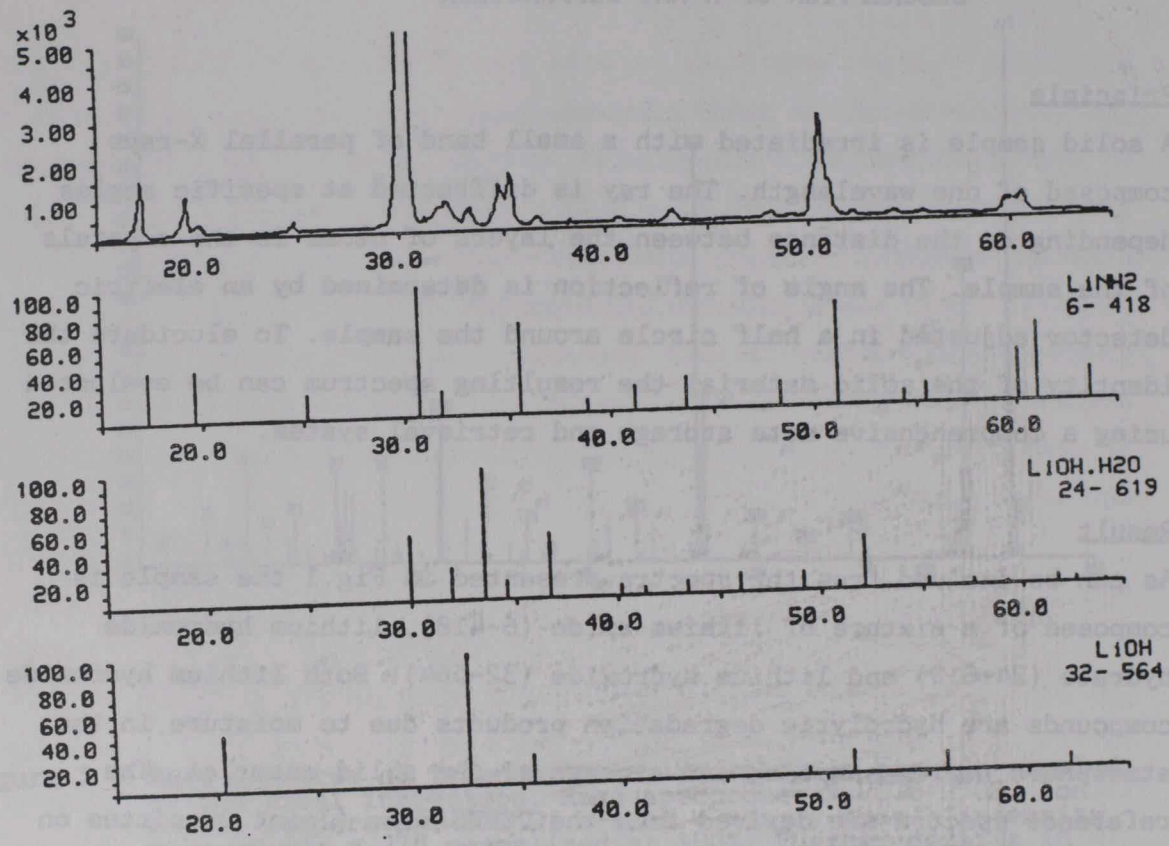


Figure 1 X-ray diffraction spectrum of lithium amide sampled at the trial inspection presented together with reference spectra. x-axis = angle of diffraction; y-axis = intensity.

ANNEX 4

DESCRIPTION OF GAS CHROMATOGRAPHY-FLAME IONISATION DETECTION. DESCRIPTION OF GAS CHROMATOGRAPHY-NP DETECTION. DESCRIPTION OF GAS CHROMATOGRAPHY-MASS SPECTROMETRY

Principle

At a certain temperature and pressure volatile components of the sample are introduced into a capillary tube (gas chromatographic column) which contains a thin wall-coating composed of a high-boiling liquid (stationary phase). An inert gas (mobile phase) flows from the injection or introduction point towards the detector at the end of the tube. Based on a compound-specific partition of the sample components which is continuously adjusted between the mobile gas phase and the stationary liquid phase a separation will be obtained.

All volatile organic compounds can be detected in a sensitive but non-specific way by the **flame ionization detector** (FID) measuring the amounts of ions formed while burning the compounds. In a gas chromatogram each compound is characterised by its specific retention time and the height of the signal (peak area) represents the amount of the concerning compound. For standardisation purposes the retention indices of substances can be calculated as derived from the retention times (RI) correlated with those of n-hydrocarbons. Representative gas chromatograms are given in Fig.1 and 3.

Organophosphorus compounds can be detected in a sensitive as well as a specific way using a **NPD-detector** (P-mode); N and P stand respectively for nitrogen and phosphorus. A rubidium salt tablet is placed in the flame and the gas flows (hydrogen and air) are adjusted for phosphorus detection. Representative gas chromatograms are given in Fig.2 and 4.

A gas chromatographic column can be connected with a **mass spectrometer** giving a sensitive and specific detection of the separated compounds. Additionally the identity of each compound can be elucidated. The spectra of the three phosphorus containing compounds derived from the gas chromatographic analysis are presented in Fig.5,6 and 7. The principle of mass spectrometry has been described in Annex 2.

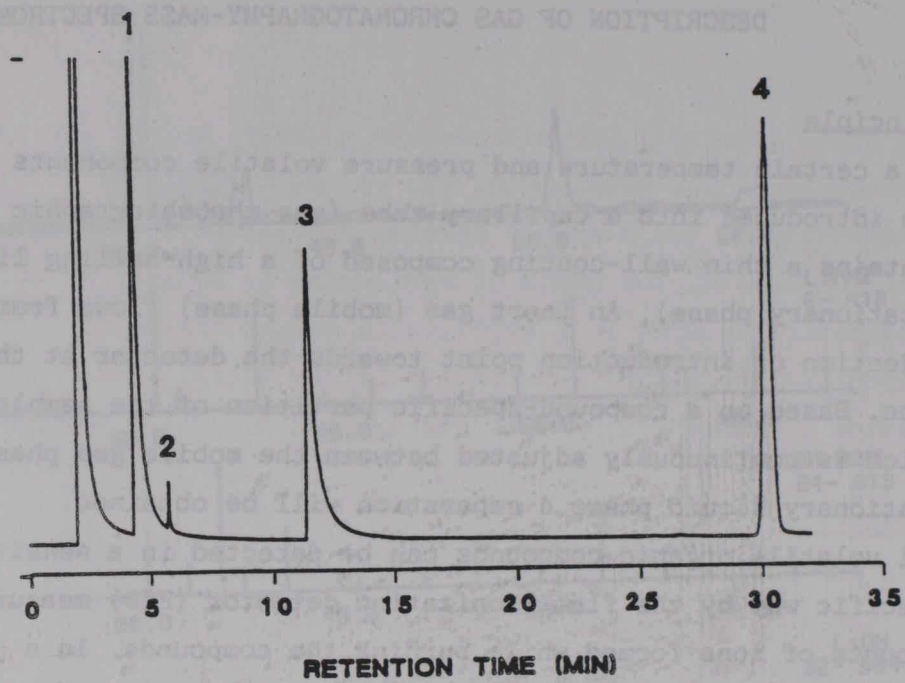


Figure 1 Gas chromatogram-FID. Sample 3, toluene layer from the reaction mixture. Column SE-30, i.d. 0.7 mm; temp. 240 C. Retention data: 1 = diphenylmethylphosphinoxide ret.time 4.30 min., RI=1991; 2 = triphenylphosphine ret.time 5.71 min., RI=2143; 3 = triphenylphosphinoxide ret.time 11.42 min., RI=2449; 4 = the non-organophosphorous compound at ret.time 30.26 min. was not further identified.

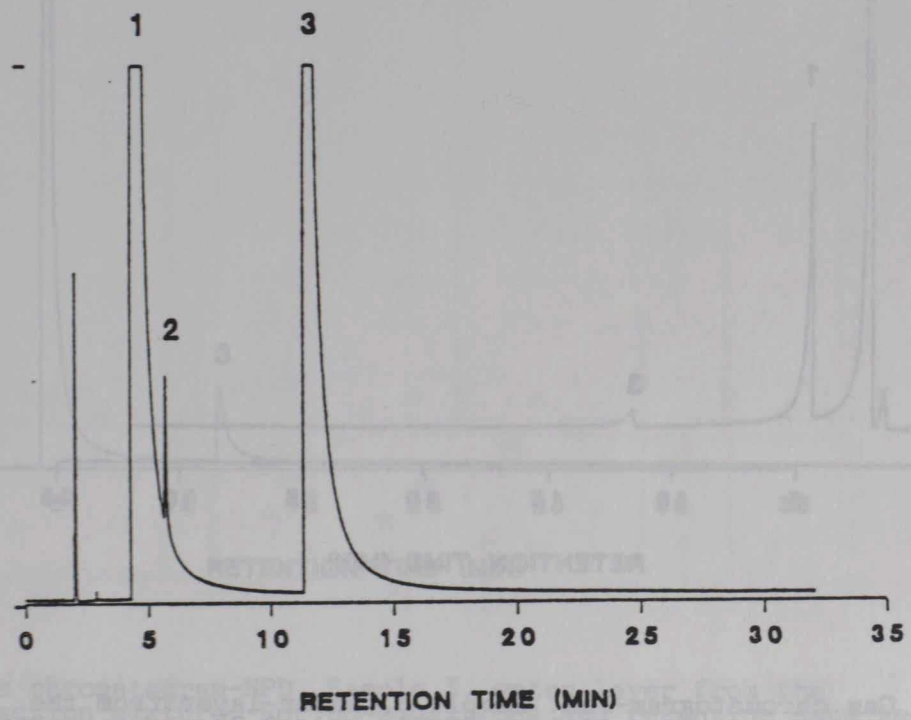


Figure 2 Gas chromatogram-NPD. Sample 3, toluene layer from the reaction mixture. For GC-conditions and retention data see Fig.1.

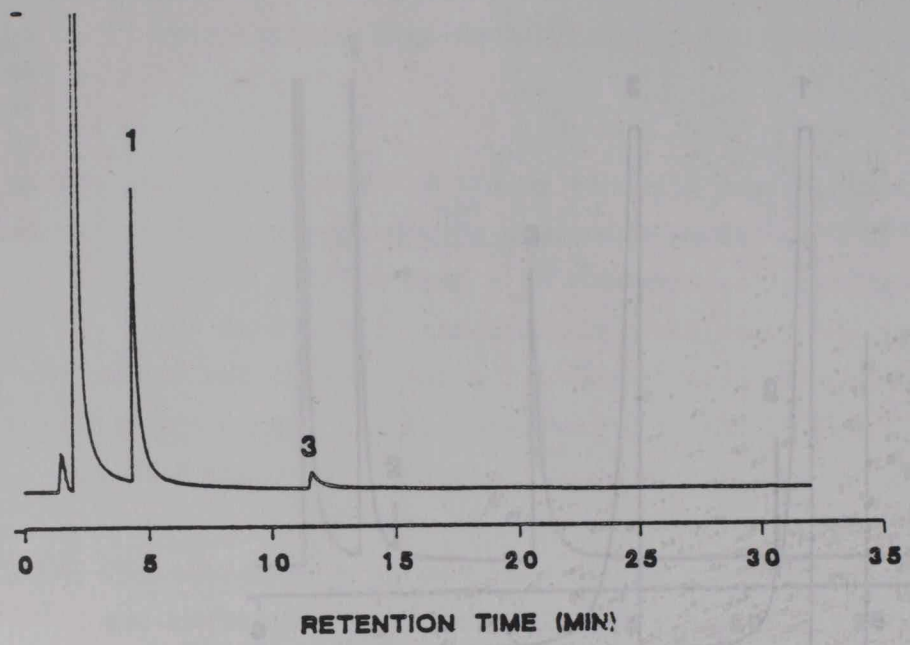


Figure 3 Gas chromatogram-FID. Sample 3, water layer from the reaction mixture. Column SE-30, i.d. 0.7 mm; temp. 240°C. Retention data: 1 = diphenylmethylphosphinoxide ret.time 4.30 min., RI=1991; 3 = triphenylphosphinoxide ret.time 11.42 min., RI=2449.

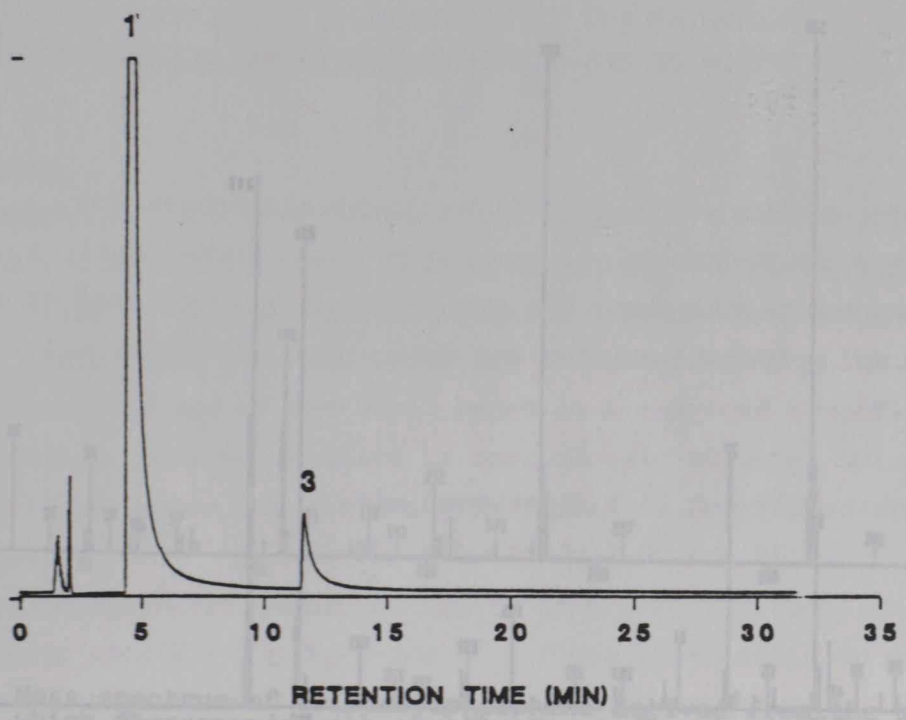


Figure 4 Gas chromatogram-NPD. Sample 3, water layer from the reaction mixture. For GC-conditions and retention data see Fig.3.

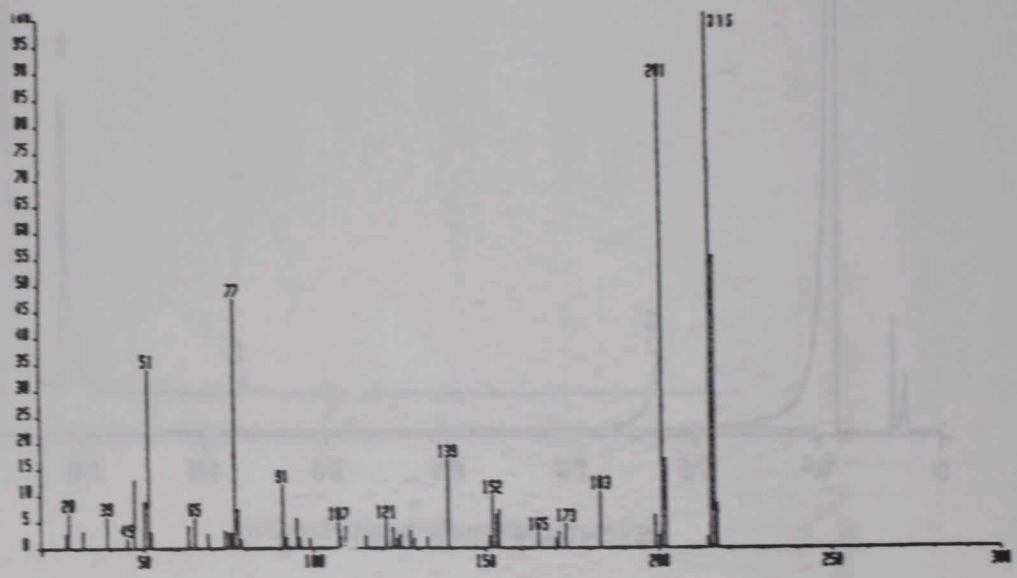


Figure 5 Mass spectrum of diphenylmethylphosphinoxide derived from the peak which corresponds with that in Fig.1 ret.time 4.30 min.

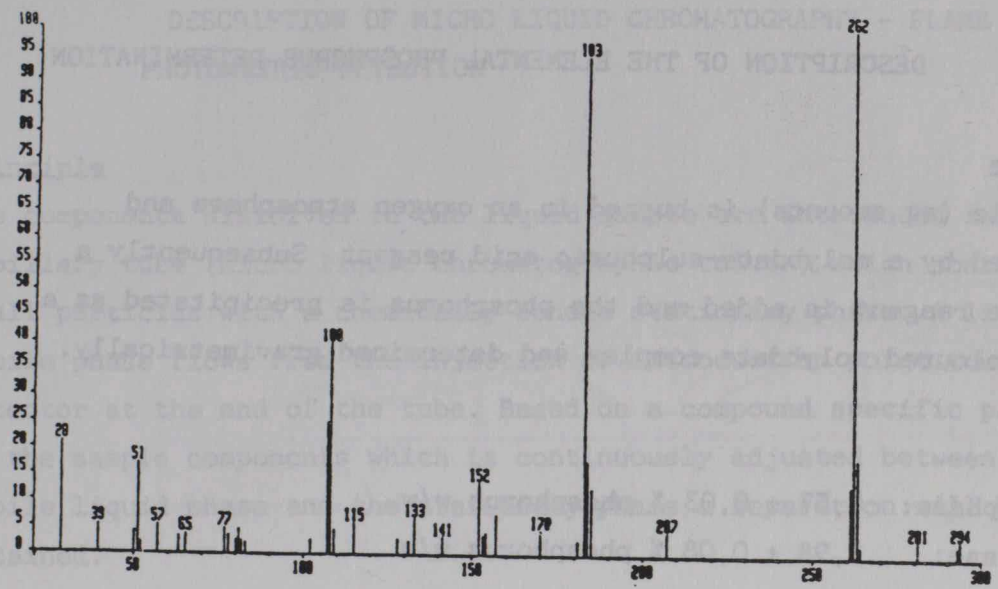


Figure 6 Mass spectrum of triphenylphosphine derived from the peak which corresponds with that ir Fig.1 ret.time 5.71 min.

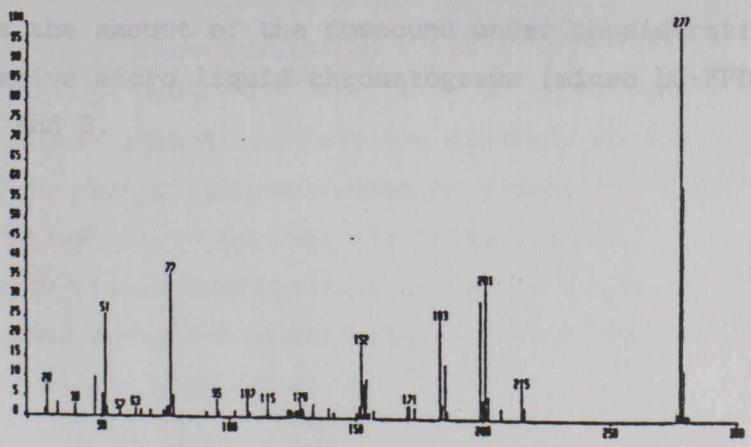


Figure 7 Mass spectrum of triphenylphosphinoxide derived from the peak which corresponds with that in Fig.1 ret.time 11.42 min.

ANNEX 5

DESCRIPTION OF THE ELEMENTAL PHOSPHORUS DETERMINATION

Principle

The sample (mg amounts) is burned in an oxygen atmosphere and destructed by a molybdate-sulphuric acid reagent. Subsequently a molybdate reagent is added and the phosphorus is precipitated as a yellow coloured molybdate complex and determined gravimetrically.

Result

Toluene-phase: 0,57 ± 0,03 % phosphorus w/w

Water-phase: 1,98 ± 0,08 % phosphorus w/w

ANNEX 6

DESCRIPTION OF MICRO LIQUID CHROMATOGRAPHY - FLAME PHOTOMETRIC DETECTION

Principle

The components dissolved in the liquid sample are introduced into a capillary tube (micro liquid chromatographic column) which contains small particles with a chemically bonded stationary phase. A liquid mobile phase flows from the injection or introduction point towards the detector at the end of the tube. Based on a compound specific partition of the sample components which is continuously adjusted between the mobile liquid phase and the stationary phase a separation will be obtained.

The organophosphorus compounds can be detected in a sensitive and phosphorus specific way by means of a **flame photometric detector** (FPD) measuring the amount of excited POH fragments formed while burning the compounds. In a chromatogram each compound is characterised by its specific retention time and the height of the signal or peak area represents the amount of the compound under consideration.

Representative micro liquid chromatograms (micro LC-FPD) are given in Figures 1 and 2.

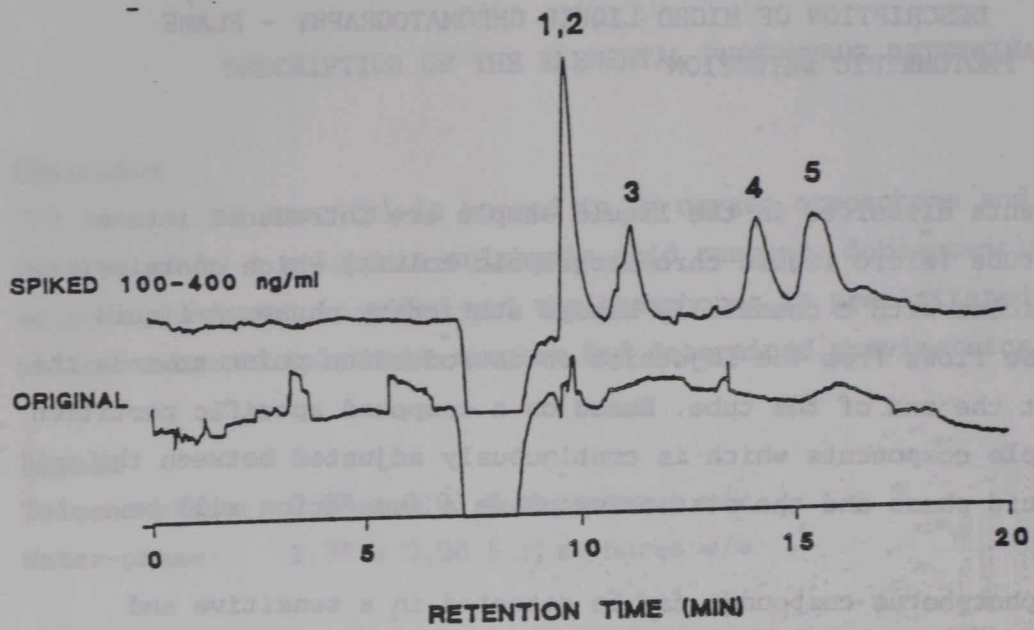


Figure 1 Micro LC-FPD chromatogram. Sample 4, taken from the waste water outlet of the TPMPB-processing. Columns i.d. 0.3 mm Partisil SAX and PRP-1 (10 µm particles). Mobile phase: formic acid + acetic acid + conc. ammonia + water, 1+1.5+3+94.5; 8 µl/min. Precolumn: Partisil SAX in Valco valve. For comparison purposes the original sample was spiked with 1 = methylphosphonic acid, 2 = dimethyl phosphoric acid, 3 = ethyl methylphosphonic acid, 4 = isopropyl methylphosphonic acid, 5 = diethyl phosphoric acid.

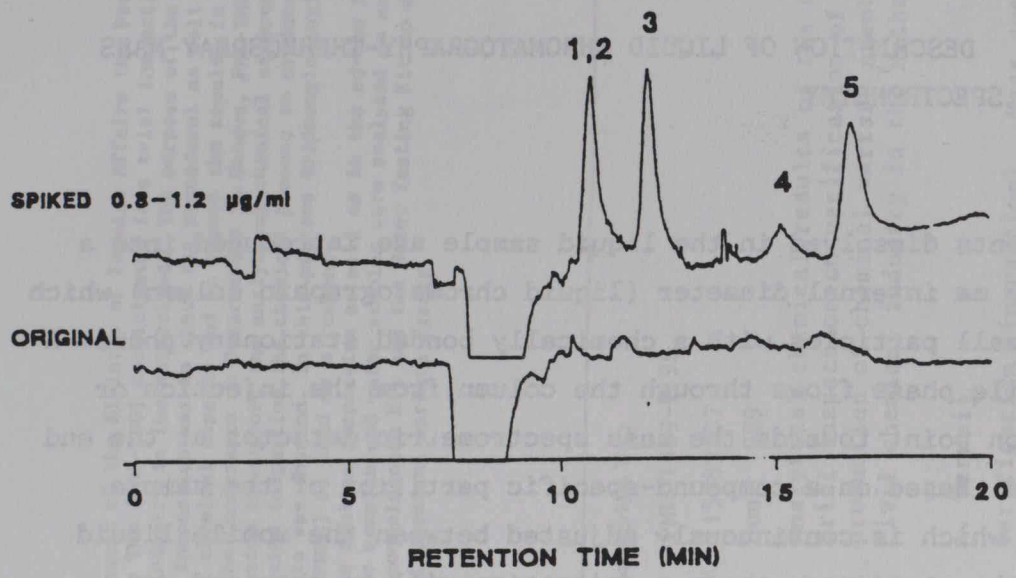


Figure 2 Micro LC-FPD chromatogram. Sample 5, taken from the waste water outlet of the whole production complex. For further details see Fig.1.

Results

In the process water sample methylphosphonic acid, ethyl methylphosphonic acid, isopropyl methylphosphonic acid could not be detected above the detection level of 0.05 µg/litre. For reference purposes dimethyl phosphoric acid and diethyl phosphoric acid were included. Each sample was pretreated by precipitating phosphoric acid as calcium phosphate to exclude its interference.

In waste water sample representing the whole facility complex the above-mentioned methylphosphonic acids was not found above the detection level of 1 µg/litre.

ANNEX 7

DESCRIPTION OF LIQUID CHROMATOGRAPHY-THERMOSPRAY-MASS SPECTROMETRY

Principle

The components dissolved in the liquid sample are introduced into a tube with 5 mm internal diameter (liquid chromatographic column) which contains small particles with a chemically bonded stationary phase. A liquid mobile phase flows through the column from the injection or introduction point towards the mass spectrometric detector at the end of the tube. Based on a compound-specific partition of the sample components which is continuously adjusted between the mobile liquid phase and the stationary phase a separation will be obtained.
The compounds can be detected in a sensitive and specific way by the mass spectrometric detector (see Annex 2). In a chromatogram each compound is characterised by its specific retention time and the height of the signal or peak area represents the amount of the concerning compound. The method and characteristic chromatograms are given in ref.1.
The method was used to analyse the water samples for pinacolyl methylphosphonic acid. In both cases this hydrolysis compound related to Soman could not be observed above the detection level of 0.1 µg/litre.

Ref.1 . E.R.J.Wils and A.G.Hulst, J.Chromatogr.,454(1988)261

Result

SUMMARY:

At the request of the Ministry of Foreign Affairs the Prins Maurits Laboratory TNO (PML-TNO) participated in a trial inspection of a civil chemical industry in the Netherlands. The purpose of the (routine and ad - hoc) inspection was to check the procedural as well as the analytical chemical aspects and to report the results in a working paper to the Conference on Disarmament in Geneva. PML-TNO took part in the inspection to support the analytical chemical aspects.
In the routine inspection the chemical process to produce a steroid-intermediate was checked. In this process triphenylmethylphosphonium bromide (TPMPB) is used as a reagent.
Both in the routine inspection as well as in the ad-hoc inspection atmospheric samples and water samples were analysed on application of the Gas Reconnaissance Kit and the Water Testing Kit to determine the presence of chemical warfare agents.

Prins Maurits Laboratorium TNO

Report no.: PML 1989-C 96

Assignment no(s): 115189137

Date: June 1989

Title: Analytical chemical results of the second trial inspection on verification of non-production of chemical warfare agents in a civil chemical industry in the Netherlands.

Author: A. Verweij

Descriptor(s):

Verification (inspection)
kits (detection)
Chem.agent detection
Mil.chem. agents
Sampling
Waste water
Gas chromatography
Mass spectroscopy
X-ray diffraction

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X-ray diffraction

CONFERENCE ON DISARMAMENT

CD/CW/WP.303
28 June 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

UNION OF SOVIET SOCIALIST REPUBLICS
UNITED STATES OF AMERICA

Proposed Revisions to the Rolling Text

Article IV

Revise paragraph 5 (as in CW/Group B/5/Rev.3; 9 April 1990) to read as follows:

"5. Each State Party shall:

(a) destroy all chemical weapons pursuant to the Order of Destruction specified in the Annex to Article IV, beginning not later than one year from the date the Convention enters into force for it, and finishing not later than 10 years after the Convention enters into force or as determined by the special conference of States Parties to be held pursuant to Article VIII, section B, subsection (b), paragraph 4 bis. However, a State Party is not precluded from destroying its chemical weapons at a faster pace;"

Article VIII

B. Conference of the States Parties

(b) Powers and functions

Add a new subparagraph 4 bis as follows:

"A special conference of States Parties shall be held at the end of the eighth year after the date of entry into force of this Convention to discuss the implementation of the principles and objectives of the Convention. This special conference shall, inter alia determine in accordance with the procedures specified in the Annex to Article IV, whether the participation in the Convention is sufficient for proceeding to the total elimination of all remaining chemical weapons stocks over the subsequent two years. The conference shall not have the authority to amend the Convention."

Annex to Article IV

(text as in CW/Group B/5/Rev.3; 9 April 1990)

Revise paragraph 2, first tick, in section III, subsection B to read as follows:

"- shall start the destruction of Category 1 chemical weapons not later than one year from the date the Convention enters into force for it, and shall complete it not later than 10 years after the entry into force of the Convention, or as determined by the special conference of States Parties to be held pursuant to Article VIII, section B, subsection (b), paragraph 4 bis." (remainder of text unchanged)

In Section III, subsection B, add a new paragraph 3 as follows:

"3. At the special conference of States Parties to be held pursuant to Article VIII, section B, subsection (b), paragraph 4 bis, an affirmative decision that participation in the Convention is sufficient for proceeding to the total elimination of all remaining chemical weapons stocks over the subsequent two years shall require the agreement of a majority of the States Parties that attend the special conference, with such majority including those States Parties attending the special conference that had taken the following three steps:

(a) presented officially and publicly, before 31 December 1991, before the Conference on Disarmament, a written declaration that they were at the time of that declaration in possession of chemical weapons;

(b) signed the Convention within thirty days after it was opened for signature; and

(c) became a party to the Convention by no later than one year after its entry into force."

UK	CD/CW/WP.304	Verification of the Chemical Weapons Convention: practice challenge inspections of government facilities: analysis of results NOT REPRODUCED (see WP volume)	Also issued as CD/1012 11 Jul. 90

* * *

Romania	CD/CW/WP.305/ Rev.1	Data relevant to the Chemical Weapons Convention NOT REPRODUCED (see WP volume)	Also issued as CD/1014/ Rev.1 16 Jul. 90

CONFERENCE ON DISARMAMENT

CD/CW/WP.306
17 July 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

REPORT OF THE TECHNICAL GROUP ON INSTRUMENTATION

The Ad Hoc Committee on Chemical Weapons decided on 9 April 1990 to reestablish 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 18 to 22 June 1990.

The group discussed extensively issues related to
- detection devices
- sampling equipment
- types of samples
- transport of samples to an off-site laboratory
- on-site analyses
- use of a mobile laboratory
- novel agents
- non-destructive measurement technology
- instrumental databases

Background papers were submitted by the delegations of France, the German Democratic Republic, the Netherlands, and the United States. A list of all background papers is included as an annex to this report. The German Democratic Republic also demonstrated a prototype for an ion mobility spectrometer. The group appreciated the workshop hosted by the Federal Republic of Germany in 14-15 June 1990.

The results of the discussions are presented in this report. The report does not repeat views presented in the previous report (CD/CW/WP.272) but adds substance on technical issues related to non-routine inspections. The first part discusses the verification by instruments and other technical means in the absence of facility agreement. The second part is a summary on the conclusions and recommendations as well as the views on the needs for further development of instrumentation. The first part of the report was discussed in the closing session of the group's meeting on 22 June 1990. The second part has been compiled by the chairman of the group. The group also reviewed the conclusions, recommendations and the views presented in the group's previous report.

GE.90-62136

VERIFICATION BY INSTRUMENTS AND OTHER TECHNICAL MEANS IN THE ABSENCE OF FACILITY AGREEMENT

The draft Convention foresees that facility agreements will be made with the facilities that will be covered by systematic international verification. The purpose of the facility agreements will be to provide the Technical Secretariat and the inspectors with the necessary advance information of those facilities in order to be able to plan the verification activities and to enable the inspectors to perform their on-site task. The importance of these agreements has been highlighted also in several reports on national trial inspections. According to the latest rolling text (CD/961) facility agreements will be made for the following declared facilities:

- storage facilities (p.68)
- destruction facilities (p.77)
- CW production facilities (p.88)
- single small scale facilities (p.99)
- facilities covered by paragraph 2 of the section on production in the Annex 1 to Article VI (p.102)
- facilities where schedule 2A and 2B chemicals are produced, processed or consumed in excess of the threshold determined in the Convention (p.107)

Facilities which will not be covered by facility agreements will therefore be:

- schedule 3 facilities
- schedule 2A and 2B facilities producing, processing or consuming less than the threshold
- laboratories where schedule 1 chemicals are synthesized in aggregate quantities less than 100 grams per year
- facilities which are undeclared under any provision of the Convention

In addition, investigations of alleged use of chemical weapons will not have any facility or other comparable supportive agreements.

The inspection types that can be foreseen for these targets are challenge inspections, other non-systematic inspections (e.g. ad hoc verification) and investigations of alleged use. The challenge inspections and investigations of alleged use have been discussed in the Ad Hoc Committee in detail, while the concept of ad hoc verification is in an early stage of development. As a guidance to ad hoc inspections the group used the Chairman's summary of consultations held during the spring session of 1990 (CW/Group A/8). While the triggering mechanisms to start the inspections differ, the technical aspects, when applicable, seem to be quite similar.

It was emphasized that in these inspections the inspectors will have to transport and use their own equipment for on-site analysis because there is no previous information on the inspected site. The time requirement was identified by the group as a serious constraint; aspects of particular concern to the group in this respect were the time taken for the inspection team and its equipment to arrive at the site as well as the time taken to carry out the inspection on-site.

DETECTION DEVICES

The group considered detection techniques such as ion mobility spectrometry to be valuable in finding the best sampling points and thus reducing the time required for sampling. Detection can, however, never be a substitute for chemical analysis but may in some cases reduce the number of samples taken. There are several detectors commercially available and under development. The group preferred detection devices over detection kits as far as the response time is concerned.

Detection instruments (e.g. CAM) are also necessary for personal safety of the inspectors, especially when investigating military stocks and cases of alleged use.

SAMPLING EQUIPMENT

The group considered the existing sampling equipment for environmental samples to be suitable when properly assembled in kits. Various types of equipment have been tested during national trial inspections. Similar equipment can be used for collection of environmental samples for all target problems.

If munitions are found in undeclared storage facilitites or in the case of alleged use they may be inspected by non-destructive measurement techniques (see Table 1) to distinguish chemical munitions from conventional ones. Chemical munitions must be sampled to allow unambiguous identification of the chemical fill. The equipment required for sampling munitions are more difficult to define due to the possible hazards during sampling and the fact that there are many different types of munitions. The group felt it advisable to include among the inspectors explosive ordnance experts. It was also suggested that once the decision has been made to open a munition the receiving state should be responsible for the opening of the munitions and for sampling in storage facilities in presence of inspectors; the group recognized, however, that it may be impracticable for the host nation to sample unexploded munitions in the investigation of alleged use.

In inspections to chemical production facilities the samples from the process could be collected using the equipment of the facilility.

TYPES OF SAMPLES

Because these inspections are carried out in unpredictable conditions the exact types of samples cannot be defined. Typical examples of samples which could be collected during challenge inspections are listed in Table 2. Environmental samples may include air, water, and soil. These samples can be collected both in the facilities and outdoors. Artefacts were considered as valuable samples since some materials retain chemicals.

The group discussed the need to take samples from all chemicals found during inspections of undeclared storages in order to find possible novel agents. Due to the large number of different chemicals found in military sites (incl. lubricants, solvents, fuels etc.) it was considered sufficient to restrict sample taking to those chemicals which on preliminary examination may have relevance to the Convention. Another possibility might be to include into the database those chemicals which are often found in different sites for purposes not prohibited by the Convention to facilitate their identification as unimportant under the CWC.

Any chemical found in munitions and suspected to be a CW agent should be subject to most careful analysis.

Samples collected for trace analysis in undeclared storages may become necessary if the inspection mandate includes retrospective elements or if the inspectors find indications that chemical weapons may have been removed before the inspection.

The group identified the problem of verifying possible production of undeclared schedule 2 chemicals, especially in multi-purpose plants; similar problems arise in demonstrating non-production of schedule 1 chemicals. The reasons for this are that not only is the number of chemicals to be identified potentially very large but, in multi-purpose plants, the number of possible sampling points may also be large. It is therefore necessary to use sampling/analytical techniques which may provide an overall picture of the chemicals being produced at the site at the time of the inspection in order to overcome the possibility that undeclared compounds may remain undetected during the monitoring of declared compounds. Such overall sampling/analytical techniques will not necessarily give definitive information but may lead the inspectors to, for example, request an extension to the time allowed for the inspection or to identify additional investigation techniques which they may wish to use. Sampling techniques proposed include the sampling of the air environment, waste water and organic waste and analysis of such samples for schedule 2 chemicals; in the case of organophosphorous compounds analysis for compounds containing a P-C bond may be carried out.

It should be noted that if the aim is to verify the absence of overproduction or diversion the target problem itself cannot be solved by sampling and chemical analysis but mainly by auditing methods. However, chemical analysis may become necessary to confirm the identity of the chemicals in question.

TRANSPORT OF SAMPLES TO AN OFF-SITE LABORATORY

The group emphasized that transportation and proper handling of samples is a serious problem and should be addressed at an early stage.

During the transport due care must be taken to secure the samples against tampering and to preserve an unbroken chain of custody.

ON-SITE ANALYSES

The role of on-site analysis is to detect the absence of listed chemicals and to ensure that in case of tentative identification, best possible samples are brought to off-site laboratories.

During inspections to undeclared facilities the detection of scheduled compounds may indicate a breach of the Convention. The identification of trace levels of schedule 2 and 3 chemicals found in chemical compounds of legitimate civilian interest, such as organophosphorous pesticides, whilst not necessarily indicating non-compliance, should cause the inspectors to seek an explanation for the presence of such trace levels of the particular compound or to send appropriate samples off-site for confirmatory analysis in order to ensure that a balanced appreciation is made of the significance of the trace levels in the light of all relevant available information. The analytical results have to be unambiguous and, accordingly, samples giving an indication of listed chemicals with the analytical techniques used on-site will normally have to be brought to at least two off-site laboratories for confirmatory analyses (see CD/CW/WP.272).

The analytical tasks identified in the previous report of the group (CD/CW/WP.272) for scheduled chemicals were considered. For on-site analysis the monitoring of known compounds, monitoring of compounds for structure elucidation and semiquantification are the most appropriate ones. Unambiguous identification and structure elucidation would be desirable but may prove to be possible only in off-site laboratories, at least for the immediate future.

These tasks can be performed with such presently available techniques as gas chromatography with retention index monitoring using temperature-programmed indexes and selective detectors, GC-RIM, micro liquid

chromatography with flame photometric detection (LC-FPD/NPD) and other specific detectors, and gas chromatography-mass spectrometry (GC-MS). The miniaturization of instruments and improvement of sensitivity may lead to new techniques becoming applicable for on-site analyses.

Scheduled chemicals can be detected by GC with specific detectors followed by GC-MS analysis using electron ionization (EI) in selected ion monitoring (SIM) mode observing at least three ions. Positive results can be confirmed by recording the whole EI mass spectrum and chemical ionization (CI) to give the molecular mass of the detected compound. Information from the GC run can be used to select the proper SIM method. The degradation products can be detected with LC-FPD/NPD and confirmed by LC-MS. Toxins may require other analytical techniques than those referred to here.

In case trace analysis is considered necessary the monitoring method should be as sensitive as possible. At present GC-FTIR is not sensitive enough for trace analysis.

The same techniques can be used during challenge inspections and ad hoc verification although their applications may vary.

The group emphasized the importance of calibration and sensitivity tests performed on-site before analyses. Standard operating procedures and calibration standards for these tasks were considered necessary (see ref. CD/CW/WP.272).

USE OF A MOBILE LABORATORY

Whilst miniaturization of equipment in the future may reduce the need for a mobile laboratory such miniaturization of equipment will not eliminate the need for the provision of a safe working environment which may be provided by a mobile laboratory.As mentioned in CD/CW/WP.272 the equipment needed on-site may be brought as containerized modules instead of a fully equipped, general purpose mobile laboratory, and set up as a laboratory on-site. This does not exclude, however, use of mobile laboratories when found more suitable than containerized equipment.

NOVEL AGENTS

When a chemical becomes suspect (e.g.found in munitions or on the battle field) its structure can be elucidated and its toxicity determined in an off-site laboratory. In all other circumstances the question of novel agents poses a serious problem for which the group has not been able to define a solution. This issue should be subject to further detailed study.

Development and testing of chemical weapons is prohibited under the Convention. Verification of compliance with this provision will be a very difficult task. However, when encountered, the verification requires a variety of techniques including analytical methods which at this point in time remain undefined. For field testing the methods may be analogous with those used in cases of alleged use.

NON-DESTRUCTIVE MEASUREMENT TECHNOLOGY

Non-destructive measurement techniques can provide a useful tool for the inspectors to minimize sample taking and thus increase both speed and safety during the inspection. In challenge inspections their main use is to distinguish probable chemical munitions from other munitions. They cannot, however, replace detailed chemical analysis for identification of compounds. The techniques that have relevance to CW verification are radiography, neutron activation, neutron transmission, acoustic techniques (ultrasonics, pulse-echo) and examination of physical properties (see Table 1, for more detailed description of the techniques see also CD/CW/WP.272).

Neutron activation and neutron transmission techniques provide information on the elements present and in some cases on their relative ratios in the chemical and would thus be the preferred techniques. Their adaptation to the specific needs of CW verification as well as the reduction of the weight and size of these instruments to man-portable levels require considerable further development. Several countries are already engaged in efforts to this end.

Acoustic techniques coupled with an adequate computerized database can be developed to provide data on the physical state of the filling and possibly on some physico-chemical characteristics. Laser-based acoustic techniques may enable remote inspection. Both of these techniques are under study.

The non-destructive measurement techniques were considered most useful for inspections of undeclared stocks and inspections of alleged use (unexploded munitions). They may also have applications for inspections to commercial production plants (containers and drums).

Further development of these techniques could be encouraged. It can also be noted that these techniques have useful applications for pollution control.

TABLE I. Non-destructive measurement techniques.

Measurement	Determination	Interrogator	Complexity
Radiography	Structural image	neutrons	Requires complex field instrumentation and a strong source. Complex analysis.
		gamma-rays	Requires moderate source and separate film development or complex field instrumentation and analysis.
Activation	Structural Image, Elemental contents	neutrons	Requires complex field instrumentation and a strong source or an accelerator. Complex analysis for elemental identification. Post analysis preferred. Moderate analysis for signature comparison.
Transmission	Structural Image, Elemental contents	neutrons	Requires weak source and moderately complex field instrumentation. Complex analysis for elemental identification. Post analysis preferred. Moderate analysis for signature comparison.
Acoustic	Structural Image, Density Signature	sound	Requires physical contact and the absence of external object contact. Moderate field instrumentation. Complex analysis for imaging. Moderate analysis for signature comparison.
Physical Properties	Weight, liquid filled	mechanical interaction	Requires physical contact or movement of the system under interrogation.

INSTRUMENTAL DATABASES

Some general ideas of the governing principles of the databases and information systems needed by the Organization were discussed in the previous report of the Group (CD/CW/WP.272). In its June 1990 meeting the Group discussed in more detail one specific subitem of the problem area i.e. the instrumental databases. These databases are needed both for analytical and non-destructive measurement techniques.

The role of the instrumental databases in verification in the absence of facility agreement is to facilitate identification of those samples which need to be transported to a minimum of two off-site laboratories. Verification of undeclared activities will require final confirmatory analyses to be done using authentic fully validated reference compounds spiked into the samples.

The analytical databases should include sufficient data to enable identification of all possible chemicals listed in the schedules of the Convention. In addition, data on their degradation products, impurities, reaction artefacts, and derivatized products should be included in the database.

It is very important to record the identification data according to standard operating procedures and from pure fully validated compounds to ensure correctness of the data.

The analytical data produced by the instruments used on-site should be compared with the data in the central database to detect any significant differences between the two databases. This would be needed in order to evaluate the need to record new data specific to the instrument. Priority would be given to those instruments producing identical data in order to avoid repetition of a considerable amount of work.

Gas chromatography

The GC database should include temperature-programmed retention indexes measured for different standard stationary phases and for different lengths of the columns (e.g. 15, 25, and 50-m). It would be advantageous to also have response factors for the listed chemicals.

Liquid chromatography

A similar database as for gas chromatography will be needed for liquid chromatography.

Mass spectrometry

The mass spectrometric database should contain electron ionization spectra and chemical ionization spectra recorded using different reactant gases such as methane, isobutane, and ammonia. The database should also contain, for each compound, information on the three specific ions which would be used during the selected ion monitoring of that compound. In addition, high resolution data and selected reaction monitoring data should be included as well as negative ion spectra for those compounds for which this method is more sensitive than positive ion spectra. Complete mass spectra should be recorded as well as eight most abundant ions.

Infrared spectrometry

The infrared spectrometric database should contain spectra of all possible chemicals recorded in different matrices. Liquid state spectra could be recorded in carbon tetrachloride and as a nujol mull, solid state spectra in potassium bromide, and gas phase spectra in gas cuvettes. Separate gas phase libraries should be recorded for GC-FTIR spectra recorded with a light pipe, tracer, and matrix isolation techniques. For the central database the spectra should be stored as interferograms to allow processing of the spectra.

Nuclear magnetic resonance spectrometry

The nuclear magnetic resonance spectrometric (NMR) database should contain spectra observing ^{1}H, ^{13}C, ^{31}P, and ^{19}F nuclei. The spectra should be recorded in different deuterated solvents considering deuterated chloroform and deuterium oxide as the primary solvents. In addition to the spectra recorded at the appropriate magnetic field strength, the database should also include the most important spectral parameters, chemical shifts and coupling constants. For the central database the free induction decay (FID) signal should be stored to allow processing of the spectra.

The group discussed the task of recording all analytical data in the analytical database. Three options were presented which should all be further explored:

1. The Technical Secretariat could accept analytical data from various laboratories. This would, however, lead to considerable nonuniformity in the data.

2. The workload could be distributed between accredited laboratories which have obtained accreditation through interlaboratory comparison tests and shown to follow standard operating procedures and good laboratory practice (GLP).

3. The work should be done solely by the laboratory of the Technical Secretariat to guarantee the integrity of the data. No previous data would be accepted.

In addition to the analytical identification data the inspectors may benefit from additional supporting information such as toxicity data, information on declared munition types, physicochemical properties of the agents and their degradation schemes. Care must be taken not to include confidential information in the data.

The group had the view that the analytical database and the recommended operating procedures could be provided to all laboratories, both international and national, performing verification analyses. It should be noted, however, that system-related incompatibilities may cause individual user problems. Regular updating of the databases would be required. An alternative would be to allow the laboratories to use the database of the Technical Secretariat.

CONFIDENTIALITY ISSUES

The group considered the confidentiality issues especially in the context of commercial chemical industry. The recommended analytical methods have been selected to demonstrate the absence of listed chemicals instead of identifying current production. It was suggested that the instrumental databases used specifically for this purpose should contain identification data of only those chemicals listed in the schedules of the Convention. This would leave other production unidentified. A step by step approach was also considered starting with the least intrusive technique available to the inspectors and progressing to more intrusive techniques as required by the situation.

Confidentiality problems might arise in the case of mass spectrometric analysis due to the fact that the instrument may retain original analytical data in its computer. The inspectors would have to find a convincing method to demonstrate that information on chemicals not under the convention would be deleted from the computer memory after the analysis.

TABLE 2. Challenge Inspection Scenarios On-site

Target problem	Site	Procedure	Samples e.g.	Analysis
Non-declared stocks	military storage filling e.g.	detect any scheduled chemicals and chemicals stored as cw	bulk, munitions, environmental ventilation, wipe, waste, artefacts	non-destructive, GC-MS, GC-RIM,LC, trace, degradation products
Non-declared production of sch 1 chemicals	military commercial	detect any sch 1 chemicals	reactors, storage sites, wipe, combined waste water and organic waste from the production site, environmental	GC-MS, GC-RIM, LC, trace, semi-quantification, degradation products
Non-declared production of sch 2 & 3 chemicals	commercial, military	detect any sch 2 & 3 chemicals	see above	see above, but traces only for sch 2, no semiquantification
Overproduction of sch 2 & 3 chemicals	commercial	not detectable by chemical analysis		
Diversion of sch 2 chemicals	commercial	not detectable by chemical analysis		
Alleged use		a) collection and preservation of samples, b) detection/ identification of chemical agents	environmental, munitions, biomedical, artefacts	non destructive, GC-MS, GC-RIM, enzymatic, degradation products

NB:- Positive results are confirmed in at least two off-site laboratories with two spectrometric methods.
- Special care is taken during transportation and for preservation of confidential information.
- Development, testing and novel agents, when encountered, require presently undefined analytical methods.

Part II

CONCLUSIONS, RECOMMENDATIONS AND NEEDS FOR FURTHER DEVELOPMENT

There was a general agreement that the most important task in the inspections without a facility agreement was the identification of the best sampling points to reduce the number of samples to be analyzed on-site. This task can be facilitated by the use of detection devices.

Detection devices

Detection can never be a substitute for chemical analysis. There are several detectors commercially available and under development which could be encouraged.

Sampling equipment

The group considered the existing sampling equipment for environmental samples to be suitable when properly assembled in kits. The equipment needed for sampling of munitions are more difficult to define due to possible hazards during sampling and the fact that there are many different types of munitions. It was suggested that during the inspection to an undeclared storage facility when the decision has been made to open a munition the receiving state would be responsible for the opening.

Types of samples

Because these inspections are carried out in unpredictable conditions the exact types of samples cannot be defined. To detect possible novel agents during inspections to undeclared storages samples could be taken from those chemicals which on preliminary examination may have relevance to the Convention.

To enable detection of possible undeclared schedule 2 chemicals use of sampling and analytical techniques which may provide an overall picture of the chemicals being produced at the site at the time of inspection was considered necessary; in the case of organophosphorus compounds analysis for compounds containing a P-C bond may be carried out.

The group noted that there are no analytical methods available for the detection of overproduction or diversion of scheduled chemicals.

Transport of samples to an off-site laboratory

The group emphasized that transportation and proper handling of samples is a serious problem and should be addressed at an early stage.

During the transport due care must be taken to secure the samples against tampering and to preserve an unbroken chain of custody.

On-site analyses

It was agreed that the role of on-site analysis is to identify those samples which will have to be transported to at least two off-site laboratories for confirmatory analyses. This task is facilitated by the instrumental databases.

It was emphasized that identification of trace levels of undeclared schedule 2 and 3 compounds should cause the inspectors to seek an explanation for the presence of such compounds. Samples could be sent to an off-site laboratory to ensure balanced appreciation of the significance of the trace levels in the light of all relevant information.

For on-site analyses the monitoring of known compounds, monitoring of compounds for structure elucidation, and semiquantification are the most appropriate ones. These tasks can be performed with such presently available techniques as gas chromatography, liquid chromatography, and mass spectrometry. The miniaturization of instruments and improvement of sensitivity may lead to new techniques becoming applicable for on-site analyses. The same techniques can be used during challenge inspections and ad hoc verification.

It was emphasized that in these inspections the inspectors will have to transport and use their own equipment for on-site analyses because there is no information on the inspected site. The group also emphasized the importance of calibration and sensitivity tests performed on-site before analyses. Standard operating procedures and calibration standards for these tasks were considered necessary.

Novel agents

When a chemical becomes suspect (e.g. found in munitions or on the battle field) its structure can be elucidated and its toxicity determined in an off-site laboratory. In all other circumstances the question of novel agents poses a serious problem for which the group has not been able to define a solution. This issue should be subject to further detailed study.

Verification of the compliance with the nondevelopment provision of the Convention may require a variety of techniques including analytical methods which at this point in time remain undefined. For field testing the methods may be analogous with those used in cases of alleged use.

Non-destructive measurement technology

In challenge inspections the main use of the non-destructive techniques is to distinguish probable chemical munitions from other munitions. They cannot replace detailed chemical analysis for identification of compounds. The techniques which have relevance to CW verification are radiography, neutron activation, neutron transmission, acoustic techniques and and examination of physical properties.

Neutron activation and neutron transmission techniques would be preferred as they provide information on the elements contained in the chemical fill. Their adaptation to the specific needs of CW verification as well as the reduction in size to man-portable levels require considerable further development. The techniques require extensive databases to support their use in verification. Further development of these techniques could be encouraged.

Instrumental databases

The role of the instrumental databases in verification in the absence of facility agreement is to facilitate identification of those samples which need to be transported to a minimum of two off-site laboratories. Verification of undeclared activities will require final confirmatory analyses to be done using authentic fully validated reference standards spiked into the samples.

The databases should include sufficient data to enable identification of all possible chemicals listed in the Convention. In addition, data on their degradation products, impurities, reaction artefacts, and derivatized products should be included in the database.

It is important to record identification data according to standard operating procedures and from fully validated compounds to ensure correctness of data. Databases should be created for gas chromatography, liquid chromatography, mass spectrometry, infrared spectrometry, and nuclear magnetic resonance spectrometry.

The group discussed the task of recording all analytical data in the database. Three options were presented which should all be further explored:
1. The Technical Secretariat could accept analytical data from various laboratories.
2. The workload could be distributed between accredited laboratories.
3. The work should be done solely by the laboratory of the Technical Secretariat to guarantee the integrity of data.

The group had the view that the analytical database and the recommended operating procedures could be provided to all laboratories, both international and national,

performing verification analyses.

Confidentiality issues

The group considered the confidentiality issues especially in the context of commercial chemical industry. The recommended analytical methods have been selected to demonstrate the absence of listed chemicals instead of identifying current production. For this purpose the instrumental databases should contain data on only those compounds listed in the Convention. A step by step approach was suggested starting with the least intrusive technique available to the inspectors and progressing to more intrusive techniques as required by the situation.

ANNEX 1

BACKGROUND PAPERS

The following background papers were presented in the Technical Group on Instrumentation.

France: Sealed tubes neutron generators for detection and analysis

France: Chemical sampling unit
Portable contamination test apparatus

German Democratic Republic: Application of a portable PC-supported ion mobility spectrometer for verification purposes under a chemical weapons convention

The Netherlands: A chemical sampling kit

The United States: Non-destructive measurement techniques (Table 1 of the report)

TECHNICAL GROUP ON INSTRUMENTATION, 1990

Verification by instruments and other technical means in the absence of facility agreement

Tentative agenda 18-22.6.1990, Geneva

18 June-

- Review of conclusions and recommendations of CD/CW/WP. 272
- Review of need for further developments/areas
- Special problems for verification by instruments in the absence of a facility agreement
- Types of inspections anticipated (ad hoc, challenge, alleged use, etc.)

19 June-

- Role of detection, sampling and analysis
- Types of detection equipment available
- Types of portable sampling equipment available
- Types of samples required and sampling points
- Analytical needs for samples/calibration/transport
- Use of a mobile laboratory
- Selectivity, sensitivity, reliability, and portability of instruments

20 June-

- Non-intrusive analytical technology available
- Radiographic techniques
- Ultra-sonic techniques
- Neutron activation techniques
- Other possible techniques
- Combination of technologies
- Portability/sensitivity of instruments

21 June-

- Instrumental databases
- Confidentiality issues (Protection of business information)
- Scope and use of data (Undeclared production/diversion)
- Organization of data for analytical purposes
- Users of data and data protection

22 June-

- Review of discussions
- Availability of instruments to meet above needs
- Further developments needed to meet the needs
- Possible cooperative developments
- Future plans to address needs
- Preparation of the draft report to Chairman

Results of the open-ended consultations on re-establishment of the Technical Group on Instrumentation on 5 April 1990 under the chairmanship of Dr. Marjatta Rautio

Suggested programme of work: Verification by instruments and other technical means in the absence of a facility agreement

1. Facility agreements will be made for the following facilities: (CD/961)

- Storage facilities (p. 68)
- Destruction facilities (p. 77)
- CW production facilities (p. 88)
- Single small scale facilities (p. 99)
- Facilities covered by paragraph 2 of the section on production in the Annex 1 to Article VI (p. 102)
- Facilities where schedule 2A and 2B chemicals are produced, processed or consumed in excess of the threshold determined in the Convention (p.107)

2. Facilities not covered by facility agreements:

- Schedule 3 facilities
- Schedule 2A and 2B facilities producing, processing or consuming less than the threshold
- Laboratories where schedule 1 chemicals are synthesized in aggregate quantities less than 100 g per year

3. Special cases where no facility agreements exist:

- Facilities which are undeclared under any provision of the Convention
- Alleged use. Due to the profound differences between inspections to facilities and investigations of alleged use the discussion could be confined to the relevance the instrumentation used during inspections to facilities may have in cases of alleged use.

4. Types of inspections foreseen for the targets:

- Challenge inspections
- Other non-systematic inspections

The present rolling text does not include any 'other non-systematic inspections' but suggestions that come under this

definition have been presented (spot checks, ad hoc checks, ad hoc inspections).

5. Verification aims:

- non-production or non-storage of Schedule 1 chemicals either at the time of the inspection or also retrospectively
- undeclared production of Schedule 2A , 2B and 3 chemicals
- compliance with thresholds

6. Focus of discussion:

Relevant equipment used on-site include instruments for sampling and analysis, detection, non-destructive analytical techniques, protection, communication, preservation of information and location finding equipment. Discussions could be focused on the first three groups and their use on-site in circumstances where no facility agreement exists. The problems encountered during these inspections seem to be more related to the on-site activities than to the analytical tasks which would be performed in an off-site laboratory.

7. Topics for discussion:

- Identification of the special features of the inspections which are not based on facility agreements
- Requirements for sensitivity, specificity, reliability and portability of the instruments
- Types of samples, determination of sampling points
- Need for instrumental databases
- Conclusions: what is realistically achievable and needs for further development

8. Background papers:

National background papers are invited on the topics to be discussed and on the research and development programmes related to instrumentation and other technical means for verification.

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Timeframe: One week, from 18 to 22 June 1990. Report finalized and presented to the ad hoc committee before 20 July 1990.

TECHNICAL GROUP ON INSTRUMENTATION

18-22 June 1990

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Nether-lands	CD/CW/WP.307	Report on a Trial Challenge Inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1018 19 Jul. 90

CONFERENCE ON DISARMAMENT

CD/CW/WP.308
19 July 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

THE NETHERLANDS

Criteria for confirmation of chemical warfare agents identification

1. Introduction

Laboratories which will operate under a Chemical Warfare (CW) Convention must possess the capability for the unambiguous identification of CW agents in samples taken during an inspection or in the event of an alleged use investigation. This task is more or less comparable with that of laboratories which control environmental laws or regulations set up by the International Olympic Committee (doping control). The chemical analytical evidence must be such that it cannot be refuted in a court of law or questioned by experts. Due to the complexity of analytical chemistry it is not always clear to non-experts on which grounds identification is based. Allegations of the use of CW agents are sometimes founded on unsubstantial evidence. This is for instance the case with an allegation of the use of CW agents in Angola [1], where the whole analytical evidence is based on chemical detection reactions which are sensitive to interferences. In sharp contrast herewith stands the identification of CW agents in the samples taken on the battlefield by the UN team of experts during the Iran-Iraq conflict in the period 1984-1987 and analysed by specialised laboratories in Sweden and Switzerland [2]. In these cases the chemical analytical evidence, based on the results of spectrometric and chromatographic techniques, was never questioned by experts. These techniques form the basis of the analytical criteria described in paragraph 3. Generally a number of spectrometric and chromatographic techniques should be applied. However, if the amount of material to be identified decreases, the number of available chemical analytical techniques will be reduced. Moreover the degree of difficulty of the identification problem will generally increase.

In view of the political and possibly military implications of a violation of a treaty, identification of CW agents must be absolutely certain. To achieve this certainty procedures may be described in detail and validated as standard operating procedures (SOP's). This approach is suggested by Finland [3]. As pointed out by De Ruig et al [4] for the analysis of veterinary drugs in test samples this approach has a number of drawbacks :

- A SOP cannot accommodate itself to new developments and is likely to become old-fashioned.
- Modern methods, involving highly sophisticated equipment, may operate according to unique procedures which are not transferable to other laboratories.
- The information present in an analytical result may not be clearly accessible. Spectra from an analyte isolated from a matrix may differ to a certain extent from a reference spectrum.
- Because of the many possible analytes and matrices validation of SOP's will become impractical, unworkable and too expensive in terms of costs and laboratory resources.

Therefore an alternative approach was proposed by setting up identification criteria. The same approach is proposed by The Netherlands for the identification of CW agents. In this working paper criteria for confirmation of CW agents identification are presented. The wish for having such a set of identification criteria was expressed during the meeting of experts which discussed the results of the first Round Robin verification exercise carried out in the fall of 1989 [5]. The criteria presented here are based on reports in the literature [4, 6-8] and on experiences in The Netherlands and other NATO nations. This approach does not imply that a detailed description of the chemical analytical procedures used by a particular laboratory is no longer necessary. Such an activity must be part of the general quality procedures of the laboratory. Also this approach does not preclude the establishment of recommended methods that may or may not be utilized.

Primarily, this paper deals with criteria for confirmation of identification. However, for fulfilling these criteria other factors should also be taken into account. Analysis and sampling are to a certain extent linked. Air samples taken by sucking air through adsorbent tubes followed by thermodesorption analysis with equipment specially designed for the treatment of these tubes is a well-known example of such a link. Laboratories are only capable of performing the identification task if sample collection and transportation has been carried out correctly. Most likely, any mistakes made during the sampling cannot be corrected later on. Furthermore, compounds such as CW agents are subjected to decomposition during transportation. Therefore, in addition to the identification criteria guide-lines and procedures should be set up for sampling. Some general sampling criteria are summed up in paragraph 2.

The analysis of samples should be carried out in specialised laboratories. In addition to general rules which go by the name of Good Laboratory Practice the laboratory must fulfil other prerequisites. First of all it must be equipped with modern chemical analytical instruments and must have a well-trained team of technicians to operate them. Additionally the team must have the necessarily experience in the field of identification of CW agents. The importance of the last mentioned item cannot be emphasized enough. Approximately five years ago several victims of alleged attacks with mustard gas were treated in European hospitals. Several reports from hospital and forensic laboratories appeared in literature describing the identification of mustard gas in the urine of victims 5-10 days after the alleged attacks [9,10]. In view of the hydrolytic instability of mustard gas under physiological conditions [11] this seemed highly unlikely. One may presume that laboratories with experience in the field of CW agents should at least question such a finding.

2. Sampling criteria

The following criteria should guarantee the sampling integrity and quality of the laboratory.

- samples should be taken, labelled and transported to an analytical laboratory according to accepted procedures
- in cases where it is possible and appropriate control samples should be taken
- samples should be sent to at least two specialized laboratories trained for the analysis of CW agents

3. Analytical criteria

The analytical criteria are arranged according to the amount of material available. They are based on general accepted identification techniques of which the abbreviations are presented in paragraph 6. It is obvious that techniques providing more information (e.g. high resolution MS or MS/MS) or new developed methods (e.g. LC/MS) than the ones mentioned in the criteria may provide alternatives. In order not to restrict the identification to those laboratories which are equipped with such highly sophisticated instruments no criteria have been set up for these techniques.
The first two sets of criteria (see 3.1 and 3.2) are considered to be appropriate for the unequivocal identification of a CW agent. The third set of criteria (see 3.3) forms a basis for a severe accusation that CW agents are present and that further investigations have to be performed. These last criteria are of particular value for the analysis of biological samples.

3.1. Large amount of material available

The amount of material is sufficient to perform every chromatographic or spectrometric analysis.

- agreement between recorded spectrum (IR or Raman, NMR, mass) and reference spectrum (see 3.5.1).
- at least two types of spectra should be recorded of which preferably one is an electron impact (EI) mass spectrum
- if the molecular ion is not present in the EI mass spectrum chemical ionization (CI) should be carried out
- GC retention indices or times and/or TLC Rf values may provide additional evidence (see 3.5.2 and 3.5.3)

3.2. Small amount of material available

In this case the compound will most likely be present in an extract or an adsorption tube and identification has to be carried out on a (sub-)microgram level using combined methods (e.g. GC-MS).

- GC retention indices or times must agree (see 3.5.2)
- the EI mass spectrum must correspond with that of the reference (see 3.5.1)
- in case of an absent molecular ion a CI mass spectrum must be recorded
- GC specific detection (NPD, FPD), GC-IR or TLC Rf values (see 3.5.3) may provide additional evidence

3.3. Very small amount of material available

Generally in this case the amount of material is so small (picogram level) that no complete EI or CI mass spectrum can be obtained. Consequently multiple ion detection (MID) has to be carried out. Because of the limited information provided by this technique the following set of criteria is considered to be insufficient for an unequivocal identification of CW agents, but forms a sound indication for their presence.

- GC retention indices or times must agree (see 3.5.2)
- GC specific detection (NPD, FPD) must be carried out in appropriate situations
- GC/MID has to be performed on at least three individual selected ions (see 3.5.4).
- the peaks obtained during GC/MID should have co-incidental maxima, the same peak width at half height, a signal-to-noise ratio of at least three and their ratio should fall within 10 % of the values of the reference (see 3.5.5)

3.4. General

- at least two samples should be analysed leading to comparable results
- the results of the identifying laboratories should correspond with each other
- the analytical procedures applied to blank control samples should not result in a positive identification
- in order to check the analytical procedure as well as to obtain an impression of the recovery a blank control should be spiked and analysed
- a rough estimate of the quantity must be carried out and reported along with the identified CW agent(s)
- the detection limit should be indicated in case no CW agents were found

3.5. Remarks

3.5.1. Spectral data from commercially available libraries or literature may be consulted. However, they should be used with discretion, as blind acceptance of library data matches could lead to erroneous results. Nothing surpasses comparison of sample data with data from authentic reference compounds recorded with the same instrument and under identical conditions.

3.5.2. GC retention indices must agree within ± 5 units, when analysed on the same stationary phase and under standardised conditions.
GC retention times must agree within ± 5 seconds in case a direct comparison is made between a compound and a reference. This can only be achieved if the analyses are performed with the same column and under the same conditions.

3.5.3. In addition to the specific detection reactions standardised TLC values must agree with reference data within the limits which are determined by the standard deviations of the reference data.

3.5.4. No general rules can be given for the selection of the ions, except from the fact that they should correspond with characteristic fragments. If appropriate the (pseudo)molecular ion should be selected.

3.5.5. GC/MID criteria can only be achieved if the compound to be identified is compared with a reference analysed with the same equipment and under the same conditions.

4. Final considerations

4.1. The above described criteria are only applicable to those compounds which are not naturally occurring such as the organophosphorus nerve agents. For compounds such as toxins which are found in nature additional criteria have to be fulfilled. Factors such as concentration level and combination of compounds are of importance.
Evidence provided by decomposition products or precursors is less compared to the CW agents themselves. No general rules can be given. For example the identification of pinacolyl alcohol will strongly point to the nerve agent soman, as will thiodiglycol do in the case of mustard gas. However, thiodiglycol is widely used in industry, whereas pinacolyl alcohol is considered to be solely used for the production of soman.

4.2. By considering the criteria one should bear in mind the statement made by Gilbert et al [6] in a paper concerning confirmation and quantification of pesticide residues: "No universally-agreed rules have been established as to the criteria that should be fulfilled to establish identification, and what might be appropriate in one situation were unequivocal identification is required may be inappropriate elsewhere where less rigour is necessary". Less rigour is perhaps required in cases where many samples and a large amount of material is available. However severe rigour will be necessary when the confirmation of identification has to be carried out at a low level. As the amount of material to be identified decreases the number of possible errors during the identification process increases. Artifacts, cross-contamination or other interferences may lead to erroneous results. For correct analyses at the low ppb level in complex matrices a quality assurance plan will be necessary [7].
Finally despite all the analytical criteria there is still the human factor. It will be difficult to believe that CW agents have been used if the presence of these compounds has been confirmed in only a few samples at a low ppb level.

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6. Abbreviations of analytical techniques

CI : chemical ionization
EI : electron impact
FPD : flame photometric detection
GC : gas chromatography
IR : infrared spectrometry
LC : liquid chromatography
MID : multiple ion detection
MS : mass spectrometry
NMR : nuclear magnetic resonance spectrometry
NPD : nitrogen phosphorus thermionic detector
TLC : thin-layer chromatography

CONFERENCE ON DISARMAMENT

CD/CW/WP.309
25 July 1990

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

SWITZERLAND

National Trial Inspection

(Documents and Annexes to CD/CW/WP.247)

INSPECTION REPORT

1. Tasks of the inspection

The inspectors were requested to establish whether at the time of the inspection the process and the book-keeping corresponded with the declaration.[1] The result was to be laid down in a report.

A detailed instruction document was worked out by a member of team A, assisted by the management of the production facility and the organizers of the exercise.[2] This contained proposals regarding methods of carrying out the inspection and consisted mainly of check-lists and forms. These were intended to assist inspectors without prior experience and special training to plan and carry out the inspection. These proposals were closely related to the content of the facility attachment[3] and, as far as possible, to the verification aims contained in CD/874 (p.81). They were also elaborated with specific reference to the process which was to be inspected. The team of inspectors was to regard this as a document which they were free to use as and how they thought fit.

2. The inspection

2.1 Chronology

First day, afternoon: four hours in which representatives of the company briefed the inspecting team in the presence of the organizers of the exercise. The briefing included:

- an explanation of the declaration;

- a description of the production plant and those of its surroundings which were to be inspected, with the aid of plans and layouts;

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[1] for the declaration see Annex I

[2] for the instruction document see Annex II

[3] for the facility attachment see Annex III

- a description of the process in regard to its chemical content, processing and surveillance, together with the logistic activities connected with it;

- a tour of the production plant;

- a description of the aims of the inspection and formal assignment of the inspection task by team A.

Second day, afternoon: second briefing with the aid of voluminous detailed documentary information provided by the company. The briefing included:

- study of the documentation;

- questions to representatives of the production facility;

- commencement of the inspection of the production plant by team sub-division "facility";

- commencement of the inspection of the logistic documents by team sub-division "logistics".

Third day, whole day: continuation of the inspection:

- coordination talks between the sub-divisions;

- intermediate discussions with representatives of the production facility;

- inspection of stores, continuing the process of controlling the logistics;

- inspection of the production plant and the process;

- final discussions with representatives of the company.

2.2 Inspection process

After the initial talks with representatives of the production facility, the following inspection tasks were carried out in accordance with the assignment:

- inspection of all the written documentation provided;

- consecutive control of each process step in the process;

- random checking of quantities processed, delivered to and retrieved from storage as recorded by the computer system;

- control of all the apparatus involved, together with auxiliary appliances;

- control of all containers on the site in respect of raw materials, intermediates and final products;

- taking separate samples from different process steps - these to be analysed within the production facility - followed by examination of the methods used for the analysis.

3. Description of the process

The process inspected was a section of a multi-step synthesis:

Process step 5:

Reaction E1 + E2 ——————————> A crude
auxiliary compounds
solvents

Process step 6:

Purification A crude ——————————> A pure
recrystallization
from organic solvents

Process step 7:

Reaction A pure ——————————> B
transformation final product

The quantities of A and B are within a magnitude range of 1-10 metric tons per year.

All these process steps - steps 5 and 6 as well as final step 7 - are run in consecutive programmes and in parallel with a time shift, so that on any particular day only isolated parts of the process can be observed. These observations may be best described as a series of snapshots.

Process step 5 represents an organic reaction with a yield of 75-80 % in respect of the key product E2. Any by-products formed, together with auxiliary chemicals not incorporated in the reaction product and recycled solvents, were not of relevance for the inspection. In step 6, however, there do appear relevant by-products from the mother liquor which are only worked up in the overlapping batches and which had to be taken into account when establishing a mass balance. If the process takes place as declared, there will appear no perceptible quantities of product A or B in waste waters, exhaust gases or solid wastes. An inspection of these would become necessary only if the mass balance should show discrepancies and be inconsistent with the declaration.

The following plan shows the flow of material for product A:

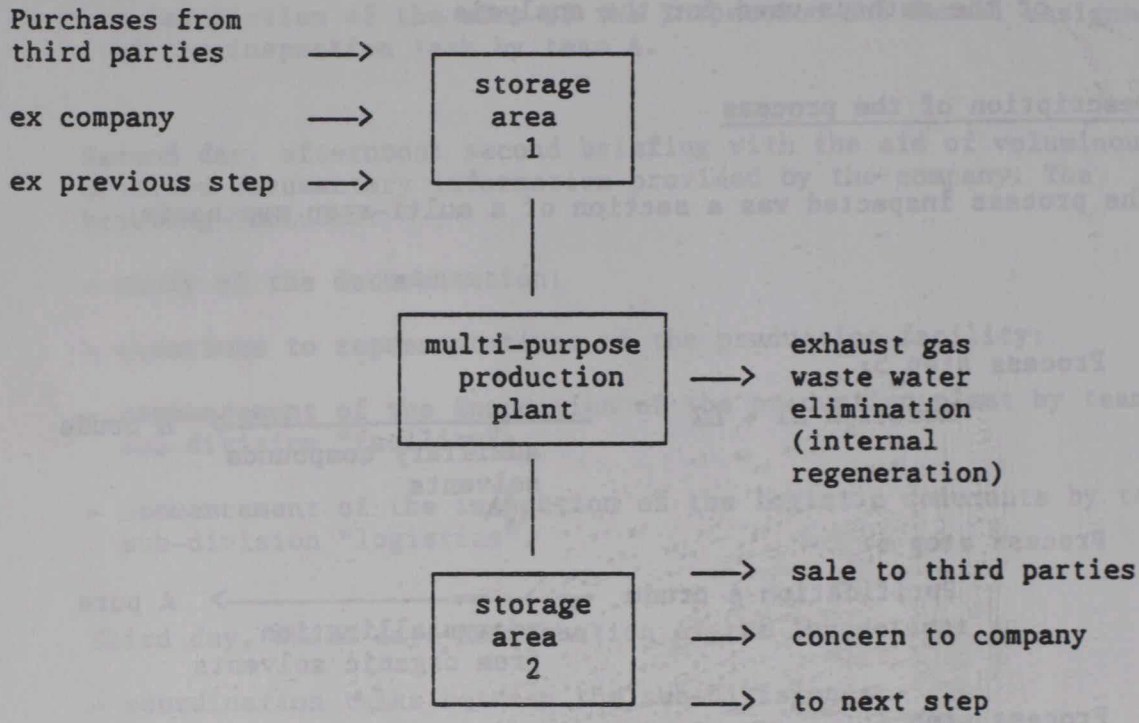

Figure 1: Flow of material

Process steps 5 and 6, which are essential for the inspection of the production plant, are carried out in plant sections as shown in the following plan:

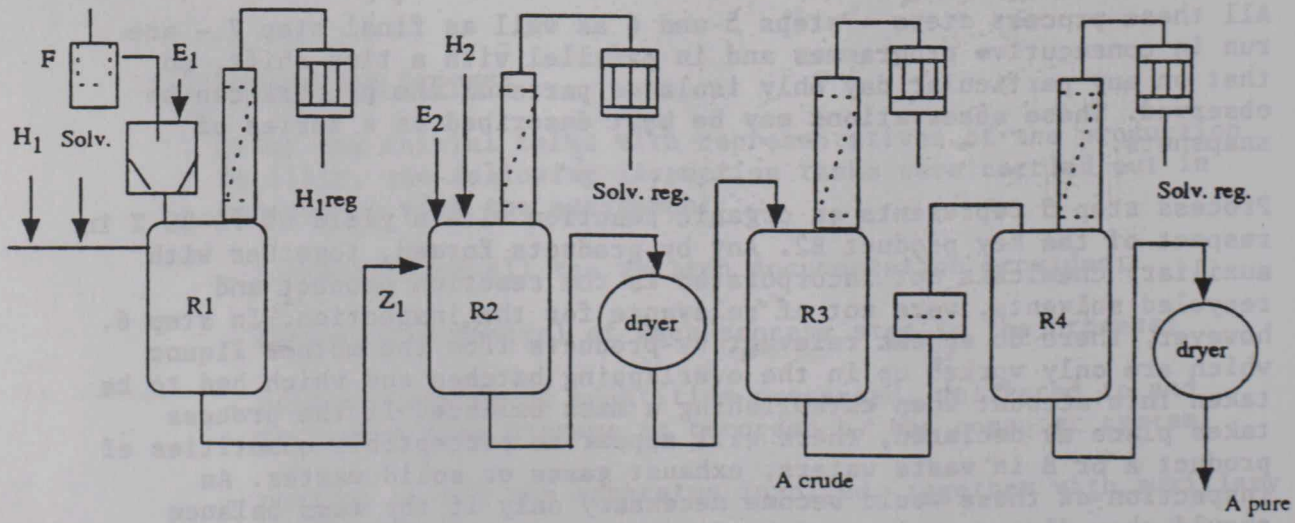

Figure 2: Schematic layout of the process

In reactor R1, an auxiliary compound H1 is bound to the basic molecule E1, thus forming the intermediate Z1. Any excess of the auxiliary compound H1 is distilled before the next reaction. After the transfer of product Z1 from reactor R1 to reactor R2, the starting material E2 is coupled to Z1, accelerated by the catalyst H2. In the next stage, auxiliary group H1 is split off by hydrolysis, and product A is dried. Purification of A crude is achieved by recrystallization in the two reactors R3 and R4. The solvent used is then distilled, and product A pure is dried in the drier.

4. Team sub-division "logistics"

4.1 Controls carried out

The computer programme used for the movement of goods is a commercial system widely used in the car industry. The company had it adapted for its own needs. By using this proprietary computer system, the inspectors were able to check on the video display terminal all substances at any time during any step in the production process.

It was thus possible to control the quantity and the precise location of any relevant product by use of a single computer terminal. This comprehensive information greatly facilitated logistics control. This information was completed by daily production reports and the relevant book-keeping documentation concerning storage of raw materials and final products.

4.2 Documents examined

- Schema of formulae for the complete synthesis
- Diagram of mass-flow with the balance of all compounds at every process step
- List of compounds with conversion factors (kg E1, kg E2 -> kg of product, kg of by-product)
- Purchase documents of raw materials in storage (chemicals bought abroad)
- Computer printouts for daily movements respecting raw materials storage
- Acquisition documents of in-plant storage (chemicals produced by the firm)
- Computer printouts for in-plant storage (the inventory as of the present moment)
- Transfer documents for delivery of intermediate and final products to storage

- Continuous monitoring of both the programmed and the effective input and output, as well as of the inventory of compounds ex terminal (access to a special computer system elaborated by this company for specific operating calculations)

- The foreman's operations book, including a summary of chemicals received, provided in the form of a survey

- Production programme for the current year, with a supplement describing amendments

- Production records for the previous year

- Computer printout of instructions for the use of apparatus

4.3 Critical points for inspection

Comparison of yields of the most important declared chemicals according to the diagram of mass flow with the actual quantities produced and used, according to the book-keeping of the storage department:

- Random checks on process quantities, as well as storage input and output as entered daily in the computer system

- Inspection and random control of the storage areas

4.4 Results

4.4.1 Mass balance 1988

The declared quantity of the product which was required according to the annual programme was in fact produced and transformed in its entirety into the permitted end product B. The yield of B from A represented 102 % of the process. The deviation from 100 % is explained by the addition of re-processed substances taken from storage. It would not be possible for substantial quantities of A. to be used for other non-declared applications.

The yield of 100 % A pure, referring to educt E1 purchased from third parties, amounted to 80,5 % of the process - or 60 % of the theoretical possibility according to the stochiometric equation. Since E1 was used in excess, no quantitative transformation into the final product is possible. In addition, the batches inspected achieved a yield inferior to the normal.
The yield of 100 % of product A pure from the inplant starting material amounted to 100 % of the process and 77 % of theoretical possibility. This result lies within the permitted band of fluctuation.

There are still small quantities of intermediaries in storage which have resulted from crystallization and distillation. These will probably be used up in the course of 1989 and could improve the yields of both A and B by some few percentage points. Such quantities would not be sufficient for any non-declared use.

4.4.2 Capacity of the production plant

According to the forecast, demand for B in 1989 will double. In the programme at present in operation, the production plant which was inspected is reserved for the whole of the current year for the production of the first stage (E2), as well as process step 5 and 6 and the last step 7, which produces the final product B. It would therefore be theoretically possible to increase production of B by a maximum of 20 %, or indeed to manufacture other products for short periods of time.

It is nevertheless characteristic of batch processes of this type in multi-purpose production plants that orders may be changed within a period of months. If requirements are reduced, capacity would automatically become available for other, non-declared purposes.

Any company which possesses many similar multi-purpose production plants remains free at all times to shift the manufacture of either the first stages or the final product to other plant sections. As a result, the declared capacity for manufacture of product A might be increased several times over. However, any possible increase in production would require all the necessary chemicals to be stored in sufficient quantity to be used as required.

Consequently, all companies possessing many multi-purpose production plants should be subject to frequent inspections of actual production in the course of a year. The intervals to be laid down would depend on the time schedule of the production runs.

5. Team sub-division "facility"

5.1 Procedure followed and controls carried out

According to the inspection mandate and the check-lists contained in the instruction document.

5.2 Documents examined

- Schema of formulae concerning the entire synthesis process
- Diagram of mass-flow, including the balance of all compounds per step
- Technical installation plan, including the entire apparatus and pipework (including those not actual being used for the manufacturing process)
- Survey diagram of apparatus
- Standard operational procedure for the facility staff

- Continuous handwritten records of the manufacturing process with the foreman's stamp for each operation

- Facility operational procedures for detailed analysis

5.3 Critical points of control: Results

5.3.1 Reaction of E1 with H1

- Quantity of E1: the number of barrels kept in readiness and their labelling corresponds to standard operation procedure for this production plant.

- Identity of E1: a sample was taken under the supervision of the inspection team. No analysis was made, as this would have been possible only in the work's control department and would have been available only 4-5 days later. The analysis details for the substance used were examined and found to be in order.

- Process parameters: temperature and pressure values correspond to standard operational procedure. As expected, the reactor was one third full.

5.3.2 Reduction/distillation of H2

- Process parameters: temperature and pressure of the distillation correspond to those of previous batches.

5.3.3 Reaction of Z1 with E2

- Identity of H2: confirmed by its characteristic appearance.

- Quantity of E2: present as a solution from an earlier stage. The sample taken was analysed by the inspecting team in the production facility laboratory and the declared quantity was confirmed.

- Identity of A: the crude product in solution was analysed in the production facility laboratory according to the production facility operational procedure for analysis under the supervision of the inspecting team. The result was compared with a reference already provided, and its identity was confirmed.

- Quantity of A: a further thin-layer chromatogram would have made possible a semi-quantitative determination. This was not done for lack of time, the inspection being at an end at that point. For the same reason, precise determination of the quantity of A by weighing was omitted. This would have been possible only after some days.

5.3.4 Additional controls

Examination of a filter: dust from various past production runs had accumulated in the filter of the pneumatic feeding device for solids, and this permitted conclusions as to the use of the facility in the recent past. In this case, analysis by X-ray fluorescence detected in significant quantity only those elements which occur in substances whose fabrication and processing correspond to the declaration (with reference to the potential synthesis of Schedule (1) substances, however, the results obtained by analytical methods registering only parts of a chemical compound are of minor significance only).

5.4 Observations: Results

5.4.1 The production plant

Location. The production plant is located in a large production facility which also contains other multi-purpose production plants (faults in one plant could impair processes in other plants).

Utilization of the production plant. Parts of the production plant not in use: a large number of tubes and valves are not in use at any particular time (but can be used when required). This is typical of any multi-purpose production plant. Random checks showed that the production plant corresponded to the technical installation plans previously provided.

Construction materials. VA steel and steel-enamel, tubing partially glass (particularly suited for corrosive elements).

Chemicals kept ready for use. During a production run, the auxiliary compound H1 and the solvent are regenerated and stored in several containers for re-use. The quantity depends on the precise stage of the batch in question. In the present case, the quantity was comparatively large - which the production facility's representatives explained by problems in the regeneration process. The problems in question were caused by a defective drier, and this was verified by the team.

Filter/ventilation. The apparatus is run by inert gas, the pressure of which can be adjusted and controlled between 0 and 2,5 bars; ventilation streams are passed through a gas washer.

During the inspection, a smell of ammonia was detected in the surroundings of the facility. This was caused by a leak in a glass tube and could be traced back to the process. The defect was repaired immediately without interruption of the process.

Technical installations for measurement, adjustment and control. A process direction system exists with terminals both at the work-place and at the production facility office. Several important operations can be performed manually.

Separate cells and rooms. There exist specially isolated separated rooms equipped with all the necessary auxiliary installations for handling dangerous and toxic chemicals with maximum protection for both facility staff and the environment.

Warning equipment. A fixed warning device for inflammable gases has been installed. The sensitivity of this device could possibly be extended to toxic substances.

Protective equipment. In the manufacturing process which was inspected, the biologically active compound D1 is loaded on to the feeding device by protected staff. Such protection consists of disposable protective suits, gloves and masks, supplied with fresh air through a ventilation system, and the input parts of the installation are covered with temporary plastic foils. Specially trained staff are engaged in all work which necessitates the use of protective clothing involving a ventilation system.

Photographic documentation. For safety reasons (danger of explosion), photography is possible only in cases where a special permit is obtained. The issue of such a permit depends on the result of measurements of the solvent concentration in the air at the production facility.

Cleaning. No unusual measures were detected.

Safety and emergency tanks. There were no special installations of this kind in the immediate vicinity of the production facility.

Alarm organization and emergeny services. They correspond to the high standard required in large works in which highly toxic compounds may be handled. Such handling was not taking place at the time when the inspection took place.

First aid. There was a high standard of safety at the production facility, but no conspicuous accumulation of protective installations or arrangements was observed.

5.4.2 The process

Declaration. At the time of the inspection, the declared product A was being produced according to the declared process and in the declared quantity.

The production plant inspected was normally laid out for the production of the declared product, as well as for the production of a number of other commercial products and their preceding stages.

Interruption of the process. According to the manufacturing documentation provided, there is nothing which was surprising to the inspection team with regard to numbers of interruptions or the length of time such interruptions would involve. (The frequently expressed opinion that a modern production plant could be readjusted to another process within a period of 8 hours is thought to be unlikely.)

DECLARATION

Key Precursor Chemical(s)

(i) The chemical name (A), common or trade name (B) used by the production plant, structural formula (C), and Chemical Abstracts Service Registry Number

(ii) The total amount produced (X), consumed(Y), imported(Z) and exported(W) in the previous calendar year

(iii) The purpose(s) for which (A) is produced, consumed or processed:

(a) conversion on-site: yes

(b) sale or transfer to other domestic industry: yes

(c) export of a key precursor (specify which country): no

(d) other: no

Production plant

(i) The name of the production plant (E) and of the owner (F), company (F), or enterprise (F) operating the production plant

(ii) The exact location of the production plant: (G)

(iii) Whether the production plant is multi-purpose yes

(iv) The main orientation (purpose) of the production plant: (H)

(v) Whether the facility can readily be used to produce a Schedule chemical or another Schedule [2] chemical. Relevant information should be provided, when applicable: --

(vi) The production capacity for the declared Schedule [2] chemical(s): (Nameplate)

(vii) Relevant Activities

a) production yes

b) processing with conversion into another chemical: yes

c) processing without chemical conversion: no

d) other no

(viii) Whether at any time during the previous calendar year declared key precursors were stored on-site in quantities greater than 1 tonnes. no

ANNEX II

Instruction Document

MANDATE FOR INSPECTION TEAM B

ROUTINE INSPECTION of the production batch of the supposed Schedule-(2)-Substance A produced by "CHIMIA"-company

BASIS: Declaration of "Date"

TASKS:

- On site investigation as to whether the actual batch at the time of the inspection and the book-keeping correspond to the declaration.
- Preparation of an Inspection Report for team C.

REALIZATION:

- In two team sub-divisions ("facility" and "logistics")
- According to ATTACHMENTS 1, 2, 3, ...

PROGRAMME:

- Briefings 27.2.89 and 1.3.89, afternoon
- Inspection 2.3.89, 0800 - 1800
- Report until ...

ORGANIZATIONAL:

- Composition of the Inspection Team
  - ..... (head of team)
  - .....
  - .....
  - .....
  - .....

- Contacts (company)

  .....

  .....

  .....

MISCELLANEOUS:

- Further details according to briefing.

For the inspectorate

Signature

Attachments mentioned

ATTACHMENT 1

TEAM SUB-DIVISION "LOGISTICS": GENERAL

Verification of available data regarding inventory transactions and mass balance as to their mutual consistence.

Comparison of these values with the declared amount of the supposed Schedule-(2)-Substance.

Procedure:

Examination of completed batch orders

Fixing of priorities for checking the parent compounds according to their significance in the batch

As far as possible, inclusion of quantitatively relevant by-products

If necessary, visual inspection of the storage houses, e.g. stock taking and control of inventories and sample taking

If necessary, inspection of the plant laboratories (records of analyses)

Documents for inspection (prepared by the company):

Material ordering sheet

Material delivery sheet

Further documents (possibly for random checks)

- Bill of material
- Operating book
- Production records
- Stock accounting book
- Inventory transaction reports
- Materials requirement reports
- Documents of raw materials storage
- Documents of in-plant storage
- Mass flow sheet

ATTACHMENT 2

"LOGISTICS": SUGGESTED ACTIVITIES

1) Examination of production records (see documents of company)

2) Identification of deficiencies in the production records

3) Proof of use (stock, conversion, sale)

4) Mass balance of production order

   operation records
   storage input/output
   yield
   by-products, waste
   recycling

5) Inquiry in case of divergences

6) General observations regarding stock-keeping

7) Comparison of the results with sub-division "facility"

8) Final discussions with representatives of the company

ATTACHMENT 3

"LOGISTICS": RECORD (suggestion) SUBSTANCE .....

	present yes/no	date	quantity	comments
Material ordering sheet				
Material delivery sheet				
Bill of material				
operating book				
production record				
Stock accountancy book				
documents stock of raw materials				
registration system of prod. data				
Massflow sheet				
. . .				

ATTACHMENT 4

SUB-TEAM "FACILITY": GENERAL CHECK-LIST

1) INQUIRY ON STATE OF PROCESS - ESTABLISHMENT OF TIME-TABLE FOR CHECKS

2) POSSIBLE CHECKS:

Conversion in the reactor:

Weight check G1 of parent compound E1
(operating book or on site)

Sample taking P1 during filling of E1
(or later at SOP point 1.8)

Estimation of filling level of the vessel

Check of operating conditions
(records, SOP, instruments ...)
according to checklist (ATTACHMENT 5)

General (visual) observations
(additional installations, anomalies)
according to checklist (ATTACHMENT 8)

Evaporation in the reactor

Sample taking of distillate P2 (parent compound E2)
(between SOP points 1.10 - 1.11)

Check of operation conditions
(records, SOP, instruments)
according to check-list (ATTACHMENT 5)

Conversion in the mixing vessel (coupling)

Weight check G2 of the "catalyst"
(operating book or on site)

Sample taking P3 after coupling
(SOP point 2.14)

Estimation of filling level

Operating conditions (ATTACHMENT 5)

General observations (ATTACHMENT 8)

ATTACHMENT 4/2

Conversion in the reactor (fragmentation)

Estimation of filling level

Operating conditions (see ATTACHMENT 5)

General observations (see ATTACHMENT 8)

Work-up in the reactor (fragmentation: washing, filtration, extraction)

not to be considered in the present case

Work-up in the mixing vessel (washing)

Operating conditions (see ATTACHMENT 5)

General observations (see ATTACHMENT 8)

Work-up in the reactor (evaporation)

Operating conditions (see ATTACHMENT 5)

General observations (see ATTACHMENT 8)

Transfer to mobile receiver

Weight determination G3 of reaction solution

Sample taking P4 (see SOP point 4.14)

Drying by batches

Weight determination G4, comparison with G1 and G3

Sample taking P5 (see SOP point 5.28)

ATTACHMENT 4/3

3) ESTIMATION OF CAPACITY:

Does the size of the batch correspond to the size of the facility?

What is the maximum annual production based on the actual batch size?

4) FURTHER INVESTIGATIONS:

Comparison of results with sub-division "logistics"

If necessary interview with representatives of the company

SOP : Standard operation procedure, Sept. 88, MP 220, 122, 123

ATTACHMENT 5

CHECK OF OPERATING CONDITIONS: CHECK-LIST

Comparison of:

- Standard Operation Procedure (SOP)
- process records
- operating book

with each other and with

- measuring instruments

regarding

- temperature
- pressure
- filling level (rough)

Inspection of calibration records (balances)

ATTACHMENT 6

ANALYTICS: CHECK-LIST

Point	
P1	Verify parent compound E1 (identity) with special order to facility laboratory Method TLC/HPLC
P2	Check distillate for excess E2 (identity) with special order to facility laboratory Method GC (corresponding to entry analysis)
P3	Check of coupling solution (identity) order to facility laboratory according to SOP point 2.14 Method TLC (max. 1 % by-products)
P4	Check of product (solution: identity, concentration) order to facility laboratory according to SOP point 4.14 Method LC
P5	Check of product (identity) order to facility laboratory according to SOP point 5.28 Method LC

OPERATING CONDITIONS AND OTHER CHECKED DATA - RECORDS							
Value to measure	Dimension	Time	S O P No.	S O P theoretical value	Instrument	Operating book	Comments
e.g. Temperature	°C	1040	2.4	100	100	100	---

ATTACHMENT 8

GENERAL OBSERVATIONS: CHECK-LIST

Facility

- Location/surroundings
- parts of facility not in use (technical installation plan)
- Changes in installation compared to documents
- Design of parts of the facility (e.g. filling devices)
- Construction materials
- Chemicals ready for use
- .
- .
- .

Infrastructure

- Filters/ventilation
- Cleaning and decontamination apparatus (e.g. high-pressure cleaning)
- Separate cells
- .
- .
- .

Safety measures

- Warning equipment
- Protection material (e.g. suits, ventilated suits, masks)
- First-aid measures (emergency, antidotes)
- Monitoring equipment
- Dimensions of emergency tanks
- .
- .
- .

ATTACHMENT 8/2

Facility records

Number and duration of breakdowns (operating book)

Accidents, fires, explosions

Frequency of revision, cleaning (operating book, cleaning records)

Frequency of replacing parts

.

.

.

ATTACHMENT 9

GENERAL OBSERVATIONS - RECORDS		
Findings / observations		
	Explanation / information	
	Open questions / assessment	

ATTACHMENT 10

LIST OF DOCUMENTS

From organization:

O-1 .....

O-2 .....

O-3 .....

O-4 .....

.
.
.

From company:

C-1 .....

C-2 .....

C-3 .....

C-4 .....

.
.
.

AGREEMENT RELATING TO PRODUCTIONS PLANTS AND FACILITIES PRODUCING, PROCESSING OR CONSUMING CHEMICALS LISTED IN SCHEDULE II

1. Identification of the site and the production plant

i) Site identification code, geographic coordinates

ii) Name of the works

iii) Owner(s) of the works on which the production plant is located

iv) Name of the company/enterprise operating the production plant

v) Exact location of the production plant

2) Exact Location (including the geographic coordinates, specific building and structure number) of the production plant

3) Location(s) of relevant building(s)/ structure(s) comprising the production plant within the works

i) Plan and layout of the works and the production plant

ii) Technical description of the production plant

iii) Layout of the production plant

4) Location of relevant plant sections

i) Tankfarm
   a) Raw Material
   b) Intermediates
   c) Final products

ii) Effluent/waste handling/treatment plant sections

iii) Recycling

iv) Other sections

5) Analytical laboratories

i) Instruments available at the works

ii) Instruments available at the production plant

iii) Analytical laboratories at the production plant

6) Computer system

7) Access to computer-based data and records

8) Safety Briefing

9) First aid

10) Areas to which inspectors have access

i) Unrestricted access

ii) Access on request

11) Technical information on the production plant

i) Reaction scheme

ii) Chemicals

iii) Type of process: e.g. continuous or batch;

iv) Technology employed; process engineering particulars

v) Data on processing and/or production batch

a) Quantity of end product
b) Duration of production/processing batch
c) Loading
d) Yield
e) Purity of final product

f) Other main products

vi) Data on product storage (type and capacity of storage)

a) Raw material

b) Intermediates

c) End product

vii) Data on waste/effluent treatment

viii) Data on maintenance and overhauls

ix) Sampling points at the facility.

x) Records and other documentation

a) Accounting records

b) Operating records

c) Calibration records as appropriate.

d) Other documentation

12) Confidentiality

GDR	CD/CW/WP.310	Report on a trial challenge inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1020 26 Jul. 90
		* * *	
Czecho-slovakia	CD/CW/WP.311	Report on a trial challenge inspection at a chemical facility NOT REPRODUCED (see WP volume)	Also issued as CD/1021 26 Jul. 90
		* * *	
Czecho-slovakia	CD/CW/WP.312	Report on a trial challenge inspection at a military facility NOT REPRODUCED (see WP volume)	Also issued as CD/1022 26 Jul. 90
		* * *	
Peru	CD/CW/WP.313	New article of a convention on chemical weapons relating to the environment NOT REPRODUCED (see WP volume)	Also issued as CD/1024 31 Jul. 90
		* * *	
Peru	CD/CW/WP.314	Proposal for the inclusion in the Chemical Weapons Convention of an Article on "Duration" NOT REPRODUCED (see WP volume)	Also issued as CD/1025 31 Jul. 90
		* * *	
FRG	CD/CW/WP.315	Chemical Weapons Verification Workshop, Munster, 14-15 June 1990 NOT REPRODUCED (see WP volume)	Also issued as CD/1026 3 Aug. 90

CONFERENCE ON DISARMAMENT

CD/CW/WP.316
6 August 1990

Original: ENGLISH

Ad hoc Committee on Chemical Weapons

CHAIRMAN'S SUMMARY OF THE 1990 OPEN-ENDED CONSULTATIONS ON ARTICLE IX

During the 1990 session the Chairman of the Ad hoc Committee carried out private and open-ended consultations on Article IX as a whole. At the end of the first part of the 1990 session the Chairman presented, during open-ended consultations, a draft suggestion for his consultations on Article IX.

This document was based on texts contained in CD/961, i.a. existing provisions on Article IX, the Protocol on Inspection Procedures and the outcome of the open-ended consultations on Article IX, Part 2, undertaken by the Chairman of the 1989 session. In addition, it contained new material in order to continue the work on Article IX by taking a comprehensive approach to the issue and to commence actual drafting, in particular, of provisions for inspections on request.

The document provided a redrafting of the first part of Article IX in two sections: A for consultations and B for clarification. It further contained a new draft for a third section, C, addressing inspections on request, including investigations of alleged use of chemical weapons. To this suggested structure of Article IX were added procedures, essentially based on existing provisions in the first part of Article IX and the Protocol on Inspection Procedures, Part III.

The Chairman stated that the purpose of the approach was to provide a qualitatively new start of the deliberations by addressing the issues at hand comprehensively and in treaty language. It was further stated that the paper was intended to provide for a less confrontational approach, whereby demonstration of compliance by the inspected State Party was emphasized and the role of the Organization was enhanced. In preliminary comments to the paper delegations considered this treaty-oriented approach a useful and necessary step, which could serve as a basis for substantive deliberations during the second half of the 1990 session.

During the second half of the session the Chairman's draft suggestion was the subject of a series of intensive private and open-ended consultations. These consultations dealt with the suggested Article itself, whereas, due to lack of time, the procedures could not be addressed. Three consecutive revisions of the draft Article reflected the development of the discussions. Agreement was reached i.a. to use the proposed term "inspection on request" and to deal with investigations of alleged use under Article IX. Towards the end of the session one delegation submitted formal amendment proposals. These proposals addressed the issue of which site could be requested for inspection, introduced the view that the Organization should be given the power to decide whether a requested inspection should be launched, and dealt with the consideration by the Executive Council of an inspection report.

The lack of time did not allow for these proposals to be given due consideration in substance. However, during the last open-ended consultations delegations discussed how these proposals could be reflected in a further revision of the draft suggestion for Article IX. Agreement could not be reached on this issue.

The Chairman therefore concluded that a further revised version of the draft suggestion for Article IX could not be elaborated at this stage.

As a basis for further work on this issue the suggested text of Article IX as well as the above-mentioned amendments proposed to that text are included separately. The suggested text of Article IX is followed by suggested consequential amendments to Article VIII and procedures for clarification.

ARTICLE IX
(Text under consultation)

CONSULTATIONS, CLARIFICATION AND INSPECTIONS ON REQUEST

A. Consultations

1. States Parties to the Convention shall make every possible effort to clarify and resolve, through exchange of information and consultations, any matter regarding compliance with or implementation of this Convention.

2. States Parties shall consult and co-operate directly among themselves or through the Organization or other appropriate international procedures, including procedures within the framework of the United Nations and in accordance with its Charter, on any matter which may be raised by States Parties in relation to the objectives or the implementation of the provisions of this Convention.

3. Nothing in this Convention affects the right of any two or more States Parties to arrange by mutual consent for inspections or any other procedures among themselves to assist in the implementation of this Convention or to clarify and resolve any matter regarding compliance with or implementation of it. Such arrangements shall not affect the rights and obligations of any State Party under any provision of this Convention.

4. The Technical Secretariat, if so requested, shall provide all possible assistance to facilitate consultations among States Parties.

B. Clarification

1. A State Party shall have the right to request the Organization 1/ to assist it in clarifying any situation regarding its compliance with the Convention. The Executive Council or the Technical Secretariat shall respond by providing such assistance as appropriate.

2. A State Party shall have the right to request the Organization 1/ to assist in clarifying any situation regarding compliance with the Convention which involves another State Party. The Technical Secretariat shall provide information and data in its possession relevant to clarifying the matter consistent with the procedures for the handling of confidential information contained in the Annex on the Protection of Confidential Information.

3. A State Party shall have the right to request the Organization 1/ to obtain clarification from another State Party on any situation or activity regarding compliance with or implementation of the Convention. 2/

4. The Director-General of the Technical Secretariat shall inform the Executive Council about activities carried out under this section.

5. States Parties named in a clarification process may request a meeting of the Executive Council in which they shall have the right to take part. 3/ In such a meeting, the Executive Council shall consider the matter and may recommend any measure it deems appropriate to the situation.

C. Inspections on request

1. Each State Party has the right to request an on-site inspection in another State Party in order to clarify any [doubts or concerns] [matter] regarding compliance with the Convention, and to have this inspection conducted anywhere without delay by an inspection team designated by the Director-General of the Technical Secretariat. A requesting State Party is under the obligation to keep the request within the scope of the Convention.

2. Throughout the inspection, the inspected State Party has the right and is under the obligation to demonstrate its compliance with the Convention.

3. Each State Party has the right to request an investigation on its own territory and/or on the territory of another State Party in order to clarify an allegation of use of chemical weapons. The Director-General shall on the basis of such a request or if need arises during the investigation request access to the territory of any State Party for the purpose of the investigation. 4/

4. The Director-General of the Technical Secretariat shall issue a mandate for the conduct of the inspection which shall comprise the request put into operational terms.

5. A State Party shall be under the obligation to admit the inspection team [and observer[s] appointed by the requesting State Party] to any site, area or location on its territory [or under its jurisdiction or control] as specified by the requesting State Party and to assist the inspection team and facilitate its task. 5/

6. The inspection shall be conducted in accordance with Part III, or, in 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.

7. [In accordance with para. A.3 of this Article States Parties may arrange by mutual consent for the presence of observers during an inspection.]

[The requesting State Party shall have the right to observe the conduct of the inspection.]

8. The Director-General of the Technical Secretariat shall promptly transmit the final report of the inspection team to the Executive Council and to all States Parties. He shall further transmit promptly to the Executive Council the [assessment] [views] of the requesting State Party, the views of the inspected State Party and the views of other States Parties which may be conveyed to him for that purpose, and then provide them to all States Parties.

9. After the submission of the final report the Executive Council shall [meet to] 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. The Executive Council shall inform all States Parties of the outcome of its meeting. 6/7/*/

10. At such a meeting the requesting State Party and the inspected State Party shall have the right to participate. 8/

Notes

*/ It is understood that this text must be seen in connection with the enclosed proposed additions to Article VIII.

1/ The issue of which organ of the Organization should be requested to take action needs further consideration.

2/ The procedures for the handling of a request for clarification need further elaboration.

3/ It is understood that the right of other States Parties to request and to participate in a meeting of the Executive Council will be dealt with under section C.(a) of Article VIII.

4/ 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.

5/ The definition of site, area and location needs further elaboration.

6/ A view was expressed that in the light of the review of Article VIII this paragraph was not needed.

7/ The view was expressed that the question of what further action the Executive Council might recommend, including possible sanctions after any on-site inspection, needs further consideration and discussion.

8/ It is understood that the rights of other involved States Parties including the possibility for any State Party to participate in the meeting shall be dealt with under section C.(a) of Article VIII.

Consequential amendments to Article VIII, section C, subparagraph (b).2.(h)

(Text under consultation)

Insert new ticks as follows

(ii)	- meet for special sessions by its own decision or with 48 hours when requested by any State Party
[(iii)	- meet for special sessions within 48 hours after the submission of an inspection report from an inspection on request.]

renumber old ticks

C.(b).2.(h) (ii) ... (iv) to C.(b).2.(h) (iv) ... (vi)

PROCEDURES FOR CLARIFICATION UNDER ARTICLE IX, PART B

(Suggested text)

1. A request for clarification according to paragraph B.3 in Article IX shall contain as precise information as possible concerning the matters to be clarified.

2. The Director-General of the Technical Secretariat shall forward a request for clarification to the State Party concerned within 24 hours of its receipt.

3. The requested State Party shall provide the necessary information to clarify the situation within seven days of the receipt of the request by it.

4. The Director-General of the Technical Secretariat shall forward the clarification to the requesting State Party within 24 hours of its receipt.

5. In the event that the requesting State Party deems the clarification to be insufficient, it may request further clarification.

Proposed Amendments to Section C

1. In subparagraph 1 on page 2, substitute the words "and to have this inspection conducted anywhere" with "which shall be conducted in any facility, location or installation relevant to the compliance with or implementation of the Convention".

2. Substitute subparagraph 4 on page 3 with the following:

"The Organization of the Convention shall examine the request and decide whether an inspection as requested should be launched. In case of an affirmative decision, it shall elaborate a mandate for the conduct of the inspection."

3. Revise subparagraph 9 on page 4 as follows (the inserted words are underlined):

"After the submission of the final report the Executive Council shall meet, without delay, to consider the report and conclude whether any non-compliance with the Convention or any abuse of the right for requesting such an inspection have occurred, and to consider and take appropriate further actions, including sanctions necessary, to redress the situation and to ensure the compliance with the Convention, including specific proposals to the Conference of the States Parties. The Executive Council shall inform all States Parties of the outcome of its meeting".

	CD/CW/WP.317	Draft report of the Ad hoc Committee on Chemical Weapons to the Conference on Disarmament NOT REPRODUCED	6.8.90
		* * *	
France	CD/CW/WP.318	Report on a trial challenge inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1029 8 Aug. 90
		* * *	
Canada	CD/CW/WP.319/ Rev.1	Report on a national trial inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1030/ Rev.1 8 Aug. 90
		* * *	
PRC	CD//CW/WP.320	Fundamental position and propositions on challenge inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1031 10 Aug. 90
		* * *	
Iran	CD/CW/WP.321	National trial inspection NOT REPRODUCED (see WP volume)	Also issued as CD/1040 31 Aug. 90

CONFERENCE ON DISARMAMENT

CD/1042
CD/CW/WP.322
3 December 1990

ENGLISH
Original: SPANISH

CHILE

Multilateral exchange of data relevant to the Chemical Weapons Convention

In document CD/828 of April 1988, the delegation of the Federal Republic of Germany submitted an outline to the Conference on Disarmament for the multilateral exchange of basic data on chemical weapons with a view to confidence-building at the time a new chemical weapons convention is being negotiated.

In this connection, Chile has the honour to draw the attention of the other delegations to its replies concerning the outline for the multilateral exchange of basic data:

	INFORMATION REQUESTED	REPLIES
1.	Presence of CW on own territory;	No
	Possession of CW on territory of another State	No
2.	Aggregate number of facilities for the production and storage of CW, and for production, processing and consumption of chemicals on Schedules (1), (2), (3)	None
3.	Types and names of CW agents produced	Chile does not produce or possess chemical weapons
	Types of CW munitions stored; CW agents in bulk	None
	Names of chemicals on Schedules (1), (2) and (3) produced in the chemical industry	Magnesium phosphide, aluminium phosphide (used as pesticides), sodium cyanide and methanol (used in copper mining)
4.	Plans and methods for the destruction of CW, including the number of facilities and the anticipated length of their operation during the 10-year destruction period	Chile does not produce or possess chemical weapons

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CONFERENCE ON DISARMAMENT

CD/CW/WP.323
10 January 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

Editing of the Draft Convention

During the resumed session of the Ad Hoc Committee (8-18 January 1991), the Committee adopted some editing changes in the Articles of the Draft Convention.

For further reference, the guidelines used for the editing, as well as a list of some outstanding editing issues, are enclosed.

SOME GUIDELINES FOR THE EDITING OF THE CHEMICAL WEAPONS CONVENTION

Nomenclature

As far as possible, for each concept one single term should be used throughout the Convention. A starting point for the elaboration of a list of recommended terms is contained in Enclosure 1. A number of words and expressions to be avoided are listed in Enclosure 2.

Numbering, etc.

The headline to each Article should be as in this example:

ARTICLE VIII

THE ORGANIZATION

In the Articles, three levels of numbering for the paragraphs should be used:

1. Arabic numerals

(a) small letters

(i) small Roman numerals

Provisionally, in Article II a fourth numbering level is used in one paragraph:

(1) Arabic numerals

Tirets should not be used.

If desirable, unnumbered headlines can be inserted to make the text more readable. The paragraphs are numbered consecutively in each Article without any attention to headlines.

Before any decision on the format of headlines is taken, various possibilities should be presented for consideration, using Article VIII as a test case.

Time and timeframes

Dates are written thus:

(day in numerals) (month in letters) (year in numerals)

15 August 1990

Time periods are expressed in:

hours,

days, or

years.

Also the words "annual" and "annually" can be used.

Months are not used. Months already present in the Rolling Text are converted to days on the basis of a 30-days "standard month".

The expressions "calendar year" and "destruction year" do not occur in the Articles. They will have to be discussed once the editing of the Annexes begins.

Future time-limits are designated by the use of the expression "not later than". The word "within" should not be used for this purpose.

The expression "at any time since" is used for designating past time-limits when it is necessary to make it clear that even an occasional occurrence of something during the time period is sufficient for it to be covered. Generally, such time-limits are designated by the use of the word "since". It must be evaluated on a case-by-case basis if "since" could be interpreted as a requirement for a continuous occurrence in a context where that was not intended.

Numbers

If the number is less than or equal to twelve, it is spelled out in letters. If the number is greater than twelve, it is written in numerals.

Numbers referring to paragraphs are always written in numerals.

Capital letters

Words to be written with (a) capital first letter(s) are listed in Enclosure 3. When a term is defined, it should be written thus:

"Abra Cadabra Hocus Pocus" means any language that ...,

otherwise with (a) small first letter(s) unless listed.

All numbered paragraphs and subparagraphs should begin with a capital letter, even if being part of an enumeration and not a complete sentence in itself.

Punctuation

Standard United Nations editing rules should be applied.

SOME OUTSTANDING EDITING ISSUES IN RELATION TO THE ARTICLES

A standardized nomenclature for the verification system and its various parts has to be adopted. In this context attention has to be paid to what extent the expression "on-site" is to be repeated as well as to what extent words like "international" have to be included.

The use of words like "immediately" and "promptly" has to be reviewed. If possible, only one single word should be used for this purpose. (Cf. e.g. Art. IV, para. 3; Art. V, paras. 2 and 5; Art. XIII, paras. 2 and 5; and Art. XVIII, para. 1.).

It has been suggested that Working Group B discuss the question of "destruction" or "destruction process" in Article IV, paragraph 6 (c) and Article. V, paragraph 8 (c), since a substantive matter might, in the view of some delegations, be involved. Likewise, it has been suggested that Working Group B discuss Article VIII, paragraph 23 (e) (also numbered D.2.(e)).

The use of adjectives such as "general", "detailed" and "precise" should be reviewed.

The use of the expression "subsidiary agreements" in Article VIII, paragraph 23 (b) (also numbered D.2.(b)) should be reconsidered.

The use of "all", e.g. in Article V, paragraph 10 ("Each State Party shall provide access to all chemical weapons production facilities ...") and "any", e.g. in Article IV, paragraph 7 ("Each State Party shall provide access to any chemical weapons destruction facilities ...", should be discussed. It is possible that sometimes "its" or "all of its" or a similar expression would be more appropriate.

CONFERENCE ON DISARMAMENT

CD/CW/WP.324
10 January 1991

Original: ENGLISH

Ad Hoc Committee on Chemical Weapons

CHAIRMAN'S PAPER

Article X: Assistance and Protection against Chemical Weapons

1. For the purposes of this article, assistance means the co-ordination and delivery to States parties of protection against chemical weapons, that covers, inter alia, the following areas: detection equipment and alarm systems, protective equipment, decontamination equipment and decontaminants, medical antidotes and treatments and advice on any of these protective measures.

2. Nothing in this Convention shall be interpreted as impeding the right of any State party to it to conduct research into, develop, produce, acquire, transfer or use means of protection against chemical weapons, for purposes not prohibited by the Convention.

3. All States parties to the Convention undertake to facilitate, and shall have the right to participate in, the fullest possible exchange of equipment, material and scientific and technological information concerning means of protection against chemical weapons.

4. 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.

The Technical Secretariat shall also, within the resources available to it, and at the request of a State party, provide expert advice and assist it in identifying how its programmes for the development and improvement of a protective capacity against chemical weapons could be implemented.

5. Nothing in this Convention shall be interpreted as impeding the right of States parties to request from and provide assistance bilaterally and to conclude individual agreements with other States parties concerning the emergency procurement of assistance.

6. Each State party undertakes 1/ to provide assistance through the Organization and to this end to elect:

(i) to contribute to the voluntary fund for assistance; and/or

(ii) to conclude, if possible within six months after the entry into force of the Convention for it, agreements with the Organization concerning the procurement, upon demand, of assistance; and/or

(iii) to declare within six months after the entry into force of the Convention for it the kind of assistance it might provide in response to an appeal by the Organization.

7. Each State party has the right to request and, subject to the procedures set forth in paragraphs 8, 9 and 10 below, to receive assistance and protection against the use or threat of use of chemical weapons if it considers that:

- chemical weapons have been used against it;
- it faces actions or activities by any State which are prohibited for States parties by article I of this Convention.

8. The request, substantiated by relevant information, shall be made to the Director General of the Technical Secretariat, who shall immediately inform all States parties and the Executive Council about it.

The Director General of the Technical Secretariat shall initiate within 24 hours an investigation in order to provide foundation for action, complete it within 72 hours and forward a report to the Executive Council. If further time is required for completion of the investigation, an interim report shall be submitted within the same time frame. The investigation shall, as appropriate and in conformity with the request and the information accompanying it, establish relevant facts related to the request as well as the types and scope of assistance and protection needed. 2/ 3/

9. The Executive Council shall meet not later than 24 hours after receiving the investigation report to consider the situation and shall take a decision by simple majority in the following 24 hours on whether to instruct the Technical Secretariat to provide assistance. The Technical Secretariat shall immediately communicate to all States parties and relevant international organizations the investigation report and the decision taken by the Executive

Council. When so decided by the Executive Council, the Director General of the Technical Secretariat shall provide assistance. For this purpose, he may co-operate with the requesting State party, other States parties and relevant international organizations. The States parties shall make the fullest possible efforts to provide assistance.

10. In case the information available from the ongoing investigation or other reliable sources would give sufficient proof that there are victims of use of chemical weapons and immediate action is indispensable, the Director General of the Technical Secretariat shall inform all States parties and shall take 4/ emergency measures of assistance, using the resources the Conference of States Parties has placed at his disposal for such contingencies. The Director General shall keep the Executive Council informed of action he is taking in this respect.

SUGGESTED PARAGRAPHS TO BE ADDED TO:

ARTICLE VIII, C 2 (a) in fine.

... conclude agreements with States parties in connection with article X and establish a voluntary fund for the purposes of this article.

ARTICLE VIII, D 2. (i).

... in relation with article X, paragraph ..., administrate the voluntary fund, compile declarations made by States parties and register, when requested, bilateral agreements concluded between States parties or a State party and the Organization for the purposes of article X.

Notes

1/ Views were expressed that assistance should be purely voluntary. A view was expressed that it should be on topical response to specific instances only. Other views were expressed that assistance should be automatic and obligatory to prevent any political expediences which could prevent or delay the delivery of assistance.

2/ In elaborating the procedures, appropriate elements of the inspection procedures under article IX, including the time frames set forth therein, as well as the experience gained through investigations by the Secretary-General of the United Nations concerning the possible use of chemical weapons, shall be taken into account.

3/ The need for quick and timely reporting, including interim reporting if necessary, as well as for speedy conclusion of the investigation has to be further elaborated.

4/ The view was expressed that the Director General of the Technical Secretariat should only facilitate emergency measures of assistance.

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CD/CW/WP.325	Draft Report of the Ad Hoc Committee on Chemical Weapons to the Conference on Disarmament on its work during the period 8-18 January 1991.	17.1.91

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