

unexpectedly 1-10  $\mu$ l sample injections directly on the analytical column were possible without any peak shape disturbances (for further discussion, see Part II). In this instance, next to DMP, MPA was found, as is shown in Figure 10. The peak at a retention time of *ca.* 17 min was also detected as an impurity in DMP standard solutions (ppm levels); separate LC-TSP-MS experiments using a conventional-size PRP-X100 column under the above eluent conditions proved it to be monomethyl phosphoric acid (MMP). The micro-LC chromatogram clearly shows the separation power of PRP-X100 for the closely related MPA, MMP and DMP.

From the above data it was concluded that the presence of the Schedule 2 compounds MPA, IMPA and OMPA indicates probable production of a Schedule 1 chemical. Other workers participating in the Round Robin test procedure unambiguously identified this chemical by means of GC-MS to be the hydrolysis product of a sarin analogue, *viz.* octyl methylphosphonofluoridate (OMPF).

*Evaluation of Round Robin Test II.* The samples were prepared by Australia with the objective to simulate a realistic inspection of a chemical industrial facility for the presence of any scheduled chemicals. The supposed industrial facility produced the commercial organophosphorus insecticide dichlorvos starting from the Schedule 3 compound trimethyl phosphite. At the time of the inspection the facility was producing dichlorvos; however, it was supposed to have been recently producing OMPF. The alkaline aqueous sample was supposed to be obtained from a waste storage tank for neutralization of non-volatile waste material. It had been spiked with dichlorvos and OMPF - a chemical not known to be a chemical warfare agent - at concentration levels of 8 mg/ml and 2 mg/ml, respectively. Sodium salts of hydrolysis products and by-products of both compounds were formed by refluxing the above sample.

LC is most suitable for handling aqueous samples and for separating polar alkyl methylphosphonic acids. However, the lack of sensitive universal and selective detectors is a major drawback. This may well be the reason that LC was only used by six out of the fifteen participating laboratories; UV, enzymatic, conductometric and, in our case, FPD and MS detection were used. As discussed in the final report on the test, most of the interesting phosphorus-containing compounds could be detected only with MS or FPD