

NORWAY

Verification of Alleged Use of Chemical Weapons

1. INTRODUCTION

Norway's research programme on verification of alleged use of chemical weapons, which was initiated in 1981 by the Ministry of Foreign Affairs, is being carried out by the Division for Environmental Toxicology of the Norwegian Defence Research Establishment at Kjeller. The research findings have been presented to the Conference on Disarmament in annual reports and working papers. In 1987, Norway was in the position of elaborating general procedures for verification of alleged use of chemical weapons (document CD/762 of 24 June 1987). These research reports and documents are contained in the publication Contributions by Norway to the Conference on Disarmament 1982-1987, published by the Norwegian Ministry of Foreign Affairs in March 1988 (document CD/813 of 7 March 1988).

In addition, and in the context of the negotiations on Article IX of the Chemical Weapons Convention, Norway has together with Canada submitted a proposal for a text for an Annex to this Article on such procedures (document CD/766 of 2 July 1987).

The Norwegian research programme is proceeding with a view both to elaborating more detailed procedures which can be used by an international inspection team and to testing and, if necessary, improving the elaborated procedures in the light of inter alia scientific and technical developments in this field. In 1987-1988, the research has been focused on elaborating procedures and data pertinent to verification of chemical warfare agents under summer conditions and on improving the technical aspects of analytical methods.

2. DEVELOPMENT OF PROCEDURES FOR VERIFICATION OF ALLEGED USE OF CHEMICAL WEAPONS IN THE CONTEXT OF A CHEMICAL WEAPONS CONVENTION

2.1 Verification of chemical warfare agents under summer conditions

The possibility of obtaining a positive verification depends on the persistence of the agent which has been used. The research programme undertaken during the winters of 1981-1982, 1982-1983, 1983-1984 and 1984-1985 clarified the persistence of several possible chemical warfare agents under winter conditions. The research in 1987-1988 has been concentrated on field experiments to obtain information about the corresponding persistence of such agents under summer conditions.

During two field exercises in October 1987 and June 1988 small amounts (1 milligram) of the nerve agents sarin, soman, tabun and VX, the blister agent mustard gas, and the production impurity of sarin, diisopropyl methylphosphonate were added to small samples of water, soil, sand and grass. The samples were left in the field, exposed to prevailing weather conditions.

During the main exercise in June 1988 the temperature varied between 20°C and 34°C during the day and between 10°C and 20°C during the night over a period of 14 days. Samples were collected after 1, 2, 4, 7 and 14 days. The samples of soil, sand and grass were extracted with water and the aqueous solutions were added to a polymer adsorbent (C-18 cartridge) to adsorb the agent. The agents were then eluted from the cartridges with chloroform, and the eluates were analysed by a gas chromatograph equipped with a multiple ion detector.

Significant amounts of tabun for analytical purposes were found in all samples. The samples taken from grass, soil, sand and water contained between 300 and 700 nanograms of tabun. This is well above the detection limit which was 10-50 nanograms. For sarin, large amounts (20 micrograms) were recovered in the water sample after 14 days. The amount detected from sand and soil was about 100 nanograms, which is well above the detection limit (1-10 nanograms). Positive verification of sarin on grass after 14 days has not yet been obtained.

The findings for soman showed that both water and soil were excellent materials for sampling. Soman was also found in all samples from grass and sand. After 14 days the amount of soman in the samples varied between 40 and 700 nanograms, whereas the detection limit was 1-10 nanograms. VX was found in all samples after 14 days. More than 100 micrograms were left in the water sample and about 1 microgram in the other samples. The detection limit for VX in such samples was 10-50 nanograms. The recovered amount of the blister agent mustard gas after exposure of 1 milligram droplet was in the nanogram range after two days, and seemed to stabilize at this level. After 14 days the amount of mustard found in the samples was between 20 and 60 nanograms, whereas the detection level was 1-10 nanograms.

More than 500 micrograms of diisopropyl methylphosphonate were detected in the samples taken from water and soil and about 50 nanograms from grass and sand. This is far above the detection limit of diisopropyl methylphosphonate.

The findings of the exercise in October 1987, which took place under somewhat cooler weather conditions (10°C), were in accordance with the reported results of the main exercise. Additional studies showed that the hydrolysis product of soman and sarin could be detected even after 4 weeks of summer conditions (ref. 2.2.2.)

2.2 Improvement of analytical procedures

2.2.1 Adsorbents

The use of small cartridges filled with an adsorbent for preparation of different types of samples has become common in analytical work. Several adsorbents are therefore available in this form. Since most of the warfare agents contain a non-polar alkyl chain, the best adsorbent will be of a

non-polar type. Six different non-polar adsorbents were compared to select the best one. Aqueous solutions of the different agents were passed through the cartridges, and the eluates were analysed by gas chromatography.

The results of the testing of the different adsorbents applied to adsorb chemical warfare agents from aqueous solutions, showed that the long alkyl-chained adsorbents C-8 and C-18 were the best general adsorbent for screening the nerve agents sarin, soman, tabun and VX, the blister agents mustard gas, and the production impurity of sarin, diisopropyl methylphosphonate. These cartridges, containing 100 milligrams adsorbents, were the most efficient in the overall procedure, i.e. both for the adsorption of the warfare agents and in the release of agents after elution with chloroform.

C-18 was preferred to C-8 since it gave the best results with a water sample of 100 millilitres. It should be remembered that C-8 gave slightly better results with small aqueous sample volumes (10 millilitres) of soman and tabun when eluted with 2-propanol instead of chloroform. This may be required for analysis by certain gas chromatographic detectors.

2.2.2 Analysis of hydrolysed nerve agents

Previous work has underlined the importance of using the degradation products of the nerve agents as a means of verification. These products are more stable and less volatile than the agents themselves and may therefore be easier to verify. An improved method for analysing hydrolysis products of sarin and soman has been developed.

The present method is based on the use of cartridges filled with an anion exchanger to trap the hydrolysed nerve agents. The cartridges are eluted with the methylating agent trimethyl phenyl ammonium hydroxide. The eluates are injected into the gas chromatograph where the methylation takes place in the injection port and detection can be carried out on the methylated compounds. The cartridge containing an anion exchanger may be combined with a C-18 cartridge. In this way the agents themselves are adsorbed to the first cartridge and hydrolysed nerve agents may be trapped on the second cartridge. This was tested in a field exercise in which samples contaminated with sarin and soman were exposed to prevailing weather conditions for one, two and four weeks before analysis. (Temperature between 10°C and 20°C.)

Three different anion exchangers were tested, and aminopropyl and diethylaminopropyl were found to be applicable in analysis of the hydrolysed nerve agents sarin and soman. The best results were obtained with aminopropyl, which was preferred in the further work. The recoveries varied with the sample volume and were 78 per cent for hydrolysed sarin and 59 per cent for hydrolysed soman from volume of 50 millilitres. The tests showed that cartridges of 100 milligrams are sufficient to deal with aqueous samples of up to 100 millilitres.

This analytical method was applied to samples of water, grass and sand, which were contaminated with 1 milligram of sarin and 1 milligram of soman, and exposed for one, two and four weeks. In the water samples, traces of sarin and soman were detected after four weeks, together with 1 microgram of the hydrolysis products. Hydrolysis products were also found in the samples taken from grass and sand after four weeks.

3. CONCLUSIONS

The research conducted in 1987-88 comprised the first full-fledged Norwegian exercises carried out under summer conditions. In the context of a Chemical Weapons Convention it is of interest that the nerve agents sarin, soman, tabun and VX, as well as the blister agent mustard gas, could be verified in contaminated samples from water, soil, sand or grass after two weeks' exposure to summer conditions with temperatures ranging from 10°C to 34°C. In general, water and soil were the best sampling materials from a verification point of view.

The present research is an extension of previous studies on the persistence of chemical warfare agents under winter conditions (1981-1985). It is noted with satisfaction that the procedures developed for winter conditions can be directly applied to work under quite different climatic conditions as described in this working paper. Even more interesting is the fact that the persistence of trace amounts of agents (a few nanograms) needed for analysis is similar to that previously described under winter conditions.

Under winter conditions, the droplets of agents dissolve small amounts of water from the snow. This leads to significant hydrolysis caused by acid catalysis even at low temperatures. In addition, droplets on snow surface are readily evaporated. Under summer conditions trace amounts of the agents are adsorbed in material such as soil and therefore protected from evaporation or degradation. In dilute water solutions the agents are hydrolysed slowly even under summer conditions.

The search for better adsorbents to be used in the field has continued. The best of six adsorbents was a cartridge of C-18 (100 milligrams), which could adsorb all the agents investigated from an aqueous sample of 100 millilitres. This cartridge could be used in combination with an anion exchanger, aminopropyl, to adsorb the hydrolysis products of nerve agents. The hydrolysis products could then be eluted with a methylating reagent and analysed as their methylated products. The whole set-up was successfully tested with samples extracted from sand, soil, grass and water contaminated with sarin or soman. This successful experiment underlines the need for a continuous improvement of the analytical procedures in the light of the rapid scientific and technical developments.