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Analysis of Chemical Warfare Agents at the NC Laboratory Spiez

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Abstract. The group for chemical analysis and verification at the NC Laboratory Spiez engages in the analysis of chemical warfare agents and related compounds in the frame of international chemical disarmament. Its area of work and instrumentation are described together with the analysis of a sample taken in Iraq after the Gulf War.

1. The NC Laboratory Spiez

The NC Laboratory Spiez is a division of the Swiss Defence Procurement Agency in the Federal Military Department. It is concerned with the effects of and the protection from nuclear (N) and chemical (C) threats and risks.

After the use of poison gas in the first world war, the so-called 'Gas Laboratory' was established as a technical authority to design for the first time protective measures for the troops against this new weapon. With the development and subsequent use of the atomic bomb, the former Gas Laboratory was converted into a technical facility not only for the military but also for the new civil protection organization. This institute, now called 'NC Laboratory', developed the technical foundations without which Switzerland would not have been able to provide suitable protective measures against N and C weapons.

The progress in biotechnology, which made possible new forms of threat, called for a thorough study of defensive and protective measures. As a consequence, a corresponding group concerned with biological (B) weapons was integrated into the NC Laboratory.

One part of a comprehensive protection against N, B, and C weapons is to support all efforts to restrict or totally eliminate all means of mass destruction. In this way, the NC Laboratory supports the Swiss Delegation in disarmament negotiations with technical knowledge, advises the Federal Government in questions of export control, and contributes to the prevention of proliferation of chemical and biological mass destruction agents. In 1993, Switzerland signed the Chemical Weapons Convention (CWC), and, with the help of the NC Laboratory, has since implemented this agreement. Furthermore, our experts support the United Nations' worldwide activities in chemical disarmament with their expertise in detection, identification, and destruction of chemical weapons. In that context, members of the NC Laboratory gave their support, *e.g.*, during the Iran-Iraq War (1980–1988) and the Gulf War.

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2. The Group for Chemical Analysis and Verification

2.1. *Area of Work*

Integrated into the section of chemistry and biology, this group deals with the instrumental analysis of chemical warfare agents and related compounds in all kinds of matrices. These might include samples of soil, water, air, activated charcoal, and concrete.

Most of the requests for analyses of samples come from The United Nations Organization. Many of these samples have been taken in places around the world, *e.g.*, during inspections of industrial facilities, or at sites where an alleged use of chemical warfare agents had to be investigated. The tasks range from the easiest case of confirming the identity of a suspected chemical warfare agent to searching all kinds of matrices for any chemicals related to the CWC at trace levels. The challenge may culminate in tasks in which a structural elucidation of an unknown compound is needed. Due to possibly significant consequences of the results of these analyses, they have to include at least two independent spectroscopic methods.

Many analyses are performed for structure confirmation of reference compounds synthesized at the NC Laboratory. Reference compounds are needed, because the identity of an unknown compound must be confirmed by comparing its spectra to those of the authentic reference. Again, these data must be acquired by two independent spectroscopic methods.

Spectra of reference compounds are also submitted to the Organisation for the Prohibition of Chemical Weapons (OPCW) in the Hague (NL). The OPCW, which is responsible for the effective international implementation of the CWC, establishes, among other things, a spectral data base for chemical warfare agents and related compounds, which will later on be distributed to laboratories performing analyses to verify compliance with the CWC.

In order to perform analyses with the most suitable methods, the NC Laboratory is equipped with a variety of analytical instruments. Most of them are based on chromatographic techniques. A survey of the most important instruments is given in *Table 1*.

2.2. *Example of a Sample Analysis*

During an UNSCOM-Mission after the Gulf War, several samples of neat chemical warfare agent were taken from pieces of ammunition in order to confirm the declarations given by Iraqi representatives. One such sample was a turbid brownish liquid which had been declared as a mixture of GB (Sarin) and GF chemical nerve agents. 11 mg of the sample were dissolved in 800 µl of $CDCl_3$ for NMR measurements. For GC/MS measurements, *ca.* 10 mg of the sample were dissolved in 10 ml of dichloromethane. To a part of this solution, diazomethane was added in order to methylate the phosphonic acids which were expected as degradation products of the declared chemical agents. Moreover, the head space of the sample was analyzed by GC/MS.

The compounds contained in the sample were identified with GC/MS and NMR (H-1 and P-31). Quantitative estimates were obtained from GC- and NMR meas-

urements. *Table 2* shows the compounds identified together with the estimated percentages.

The analysis confirmed in essence the declarations given by the Iraqi representatives. The main undeclared compounds in the sample were most probably precursors and degradation products of the declared compounds GB and GF.

2.3. *Quality Management*

Several teams in the NC Laboratory run their activities under an EN 45000 series quality management (QM) system. The group for chemical analysis and verification has been accredited by the Swiss Federal Office of Metrology under the norm EN 45001 for the analysis of samples for chemical warfare agents and related compounds since early 1993. For some years, it was the only laboratory worldwide having obtained this kind of recognition in the field.

Obviously, the group is accredited for a very wide scope, which includes basically all chemical agents and related compounds in virtually any matrix. This would never be possible without the NC Laboratory's documented ability to develop and validate new methods by itself. Quite often, especially with new compounds and matrices, analysis methods have to be validated 'on the job', *i.e.*, during the actual analysis.

As requested, the group complies with the strict QM rules. Standard Operating Procedures (SOPs) govern analytical activities as well as the maintenance of analytical and other equipment. For traceability, special attention is given to all kinds of records. The proper function of the QM system is checked annually by both, internal and external auditors.

In the context of QM, the NC Laboratory participates on a regular basis in international interlaboratory proficiency tests. In these tests, one of the participating laboratories spikes samples with special compounds, and the remaining *ca.* 25 laboratories around the world try to identify the spiked chemicals. Initially, a research group at the University of Helsinki ran four of these tests, whose primary goal was to develop and validate analytical methods for chemical warfare agents and related compounds. The next three tests were run as comparison tests by the Preparatory Commission of the OPCW in order to give laboratories in the field a possibility to judge their performance by international standards. Since 1996, the OPCW organized three more real proficiency tests with the aim of assessing the performance of the various analytical laboratories all over the world. Good results in these three tests are a prerequisite for a later designation by the OPCW to do analyses for the verification of compliance with the CWC. Since the first test in 1989, our group has passed all tests without mistakes; the fact that in 1996, the NC Laboratory was given the opportunity to provide the test samples is a sure sign of confidence of the OPCW in our activities.

Table 1. *Instrumentation at the NC Laboratory Spiez*

Technique	Instrument	Options; principal use
GC/MS/MS	*Finnigan TSQ 7000*	EI, CI; identification, structural elucidation
GC/MS	*Fisons MD 800*	EI; identification
GC/MS	*Viking Spectratrak 672*	EI; identification
LC/MS/MS	*Finnigan TSQ 7000*	ESI, APCI; identification, structural elucidation
GC/FTIR	*BioRad Tracer F45A*	identification
GC/FID	*HP 5890*	estimated quantitation
GC/FPD	*HP 5890*	estimated quantitation
GC/AED	*HP 2350*	estimated quantitation
NMR	*Varian VXR-300*	identification, structural elucidation

Table 2. *Compounds Identified in Chemical Warfare Agent Sample*

GB (Sarin)	7%
GF (Cyclohexyl methylphosphonofluoridate)	3%
Methylphosphonic acid monofluoride	26%
Isopropyl methylphosphonate	22%
Cyclohexyl methylphosphonate	9%
Diisopropyl methylphosphonate	2%
Cyclohexyl isopropyl methylphosphonate	1%
Methylphosphonic acid	8%
Isopropanol	1%
Cyclohexanol	1%
Cyclohexene	5%
Triethylamine	3%
Dichloromethane	6%
Dicyclohexyl methylphosphonate	< 1%
1,2-Dichloroethane	< 1%
Propene	< 1%
Diisopropyl ether	< 1%
Cyclohexyl isopropyl ether	< 1%
1-Methylcyclohexene	< 1%
Chlorobenzene	< 1%
2-Propyl chloride	< 1%
Cyclohexyl chloride	< 1%
Tetrafluorosilane	< 1%

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