CHEMICAL WEAPONS CONVENTION CHEMICALS ANALYSIS

Sample Collection, Preparation and Analytical Methods

Edited by

Markku Mesilaakso

Finnish Institute for Verification of the Chemical Weapons Convention (VERIFIN), University of Helsinki, Finland

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Dedication

To my wife, children, and parents.

About the Editor

Markku Mesilaakso was born in 1957 in Tornio, Finland. His main area of study was structural chemistry and he achieved the highest grade in organic chemistry at the University of Oulu. In 1985, he joined Professor Erkki Rahkamaa's research group and began work using NMR spectroscopy in toxin analysis. In 1992 in Helsinki, he began work in the CW Project of the Ministry for Foreign Affairs, and with Professor Marjatta Rautio he further developed the methods for NMR analysis of CW agents and related chemicals. He received his Ph.D. on this topic. Since the mid 1980s Dr. Mesilaakso has coauthored books in the series *Methodology and Instrumentation for Sampling and Analysis in the Verification of Chemical Disarmament* (the so-called Finnish Blue Books). His scientific publications deal with the analysis of NMR spectral parameters of CW agents and toxins, and the use of NMR spectroscopy as a complementary method for analysis of Chemical Weapons Convention (CWC) chemicals from the environment. His interests encompass verification and implementation of the CWC, environmental analysis, quality assurance, and training.

Dr. Mesilaakso is currently Acting Director of the Finnish Institute for Verification of the Chemical Weapons Convention (VERIFIN; former CW Project) at the University of Helsinki. Before this, he worked at VERIFIN as a Research Scientist, Quality Manager and Research Director. He is a member of the Finnish National Authority of the CWC, a member of the Scientific Advisory Board for the Defense of Finland, and a member of the OPCW Validation Group for data evaluation to the OPCW Central Analytical Database.

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A colour plate appears between pages 440 and 441.

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Preface

The States Parties to the Chemical Weapons Convention (CWC) have established the Organization for the Prohibition of Chemical Weapons (OPCW) in order to achieve the object and purpose of the Convention. It aims to ensure the implementation of the CWC's provisions, including those for international verification of compliance with it, and to provide a forum for consultation and cooperation among States Parties. The aim of this book is to give a comprehensive view of how to internationally verify compliance with the CWC, in principle, using analytical chemistry and related strategies and methods.

There are currently eighteen analytical laboratories that have established capability, and obtained recognized competence in, the analysis of samples for CWC-related chemicals; these laboratories are the OPCW designated laboratories. The majority of the chapters in this book discuss the analytical methods used in these off-site laboratories. The methods discussed are for the identification of target chemicals from environmental and human origin samples.

The procedures and strategies for on-site sampling and analysis are also discussed. In connection with the verification activities of the OPCW on-site, samples may be taken, for example, during a facility inspection for subsequent on-site analysis. The possibility to send the samples for analysis off-site also exists.

This book aims to serve different readers from the fields of environmental and analytical chemistry as well as researchers in civil and military laboratories. The National Authorities of the States Parties to the CWC may find the book useful as it covers developments in the area of CWC chemicals analysis. The approaches are practical, and the contributions are particularly useful for those people responsible for developing their own laboratory analysis methods and work instructions.

The book discusses a variety of topics. These include: the requirements of the CWC for verification; sampling; the mobile laboratory and its equipment and software; sample preparation from environmental samples; official inter-laboratory proficiency tests arranged by the OPCW; designated laboratories; the OPCW Central Analytical Database (OCAD); instrumentation, monitoring of hazardous chemicals, analysis strategies, quality assurance and screening; analytical methods including gas and liquid chromatography hyphenated with mass spectrometry or other detectors, nuclear magnetic resonance spectroscopy, infrared spectroscopy, capillary electrophoresis, and the detection of exposure to toxic scheduled chemicals.

I hope that this book will be of great benefit to people working in the field of the verification and implementation of the CWC. Without the professionals who have contributed to this book, it would not have been possible. I therefore express my sincerest thanks to all of the experts who have given their time and effort in publishing their precious knowledge in the field of CWC chemicals analysis.

Markku Mesilaakso

Helsinki, March 2005

CHAPTER 1
Introduction

Markku Mesilaakso
Finnish Institute for Verification of the Chemical Weapons Convention (VERIFIN), University of Helsinki, Finland

1 THE CHEMICAL WEAPONS CONVENTION (CWC)

The Convention on the prohibition of the development, production, stockpiling, and use of chemical weapons and of their destruction (the Chemical Weapons Convention, CWC) was signed on January 13, 1993, and entered into force on April 29, 1997. The CWC includes 24 Articles, the Annex on Chemicals, the Annex on Implementation and Verification (so-called Verification Annex), and the Confidentiality Annex. The Verification Annex, which by the length occupies the majority of the CWC, is written in 11 parts. Article I lists the general obligations of the CWC as shown in Figure 1.

2 DEFINITIONS

The terms used in the CWC need to be explained, and for this reason, the terms used in the Articles are defined in Article II, Definitions and Criteria. The terms used in the Verification Annex are defined in its first part. In general, a chemical weapon may be understood as munition filled with toxic chemical, but the definition of the CWC gives a larger perspective. Also, from the point of view of analytical chemistry, it is necessary to have an idea about what kind of chemicals we are aiming at. Also related to chemicals are a whole set of terms that need to be defined, that is, 'Chemical Weapons', 'Toxic Chemical', 'Precursor', and, in addition, 'key component'. The definitions are as follows.

'Chemical Weapons' means the following, together or separately: (a) toxic chemicals and their precursors, except where intended for purposes not prohibited under this Convention, as long as the types and quantities are consistent with such purposes; (b) munitions and devices, specifically designed to cause death or other harm through the toxic properties of those toxic chemicals specified in subparagraph (a), which would be released as

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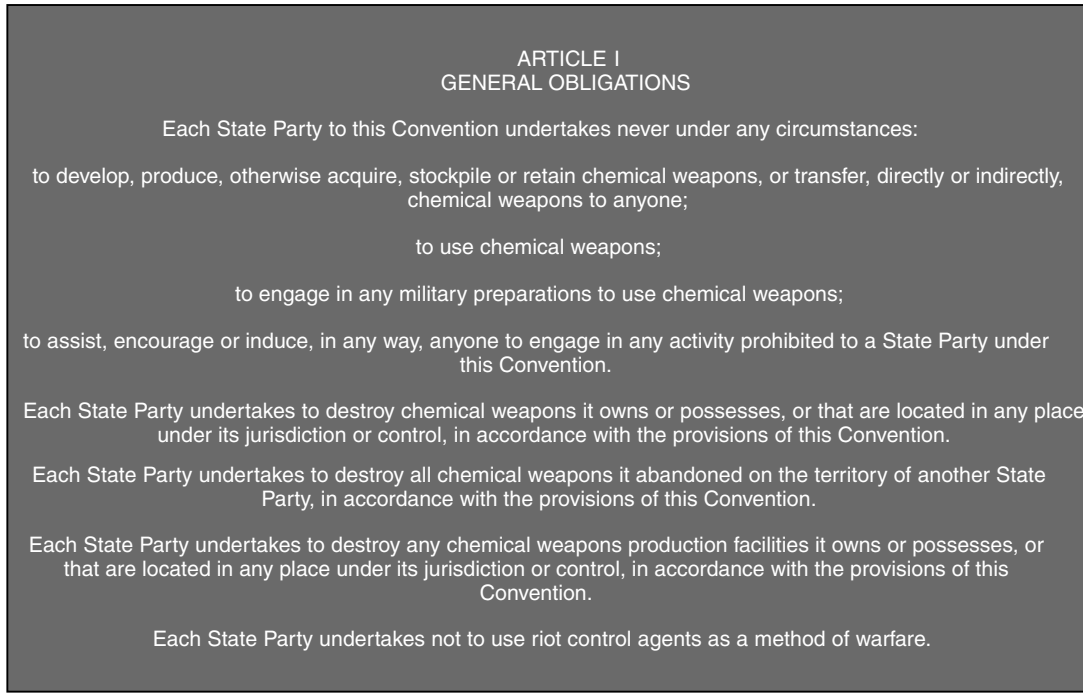

Figure 1. Article I of the Chemical Weapons Convention

a result of the employment of such munitions and devices; (c) any equipment specifically designed for use directly in connection with the employment of munitions and devices specified in (b).

'Toxic Chemical' means any chemical, which through its chemical action on life processes can cause death, temporary incapacitation, or permanent harm to humans or animals. This includes all such chemicals, regardless of their origin or of their method of production, and regardless of whether they are produced in facilities, in munitions, or elsewhere.

'Precursor' means any chemical reactant that takes part at any stage in the production, by whatever method, of a toxic chemical. This includes any key component of a binary or multicomponent chemical system.

'Key Component of Binary or Multicomponent Chemical Systems' (hereinafter referred to as 'key component') means any precursor, which plays the most important role in determining the toxic properties of the final product and reacts rapidly with other chemicals in the binary or multicomponent system.

3 SCHEDULES OF CHEMICALS

For the purpose of implementing the CWC, toxic chemicals and precursors, which have been identified for the application of verification measures, are listed in Schedules contained in the Annex on Chemicals (for the Schedules, *see* **Chapter 2**). Schedule 1 includes chemicals developed, produced, stockpiled, or used as a chemical weapon as defined above, and chemicals structurally close to them. Schedule 2 lists three toxic chemicals not included in Schedule 1 and the degradation products and precursors of these toxic chemicals as well as of those of Schedule 1. Schedule 3 lists four toxic chemicals and precursors not listed in the other Schedules. The Schedules contain mainly organic chemicals with different chemical and physical properties, being neutral chemicals, acids, bases, volatiles, and nonvolatiles, where phosphorus, fluorine, sulfur, chlorine, nitrogen, and oxygen occur frequently. Riot control agents are not included in the Schedules.

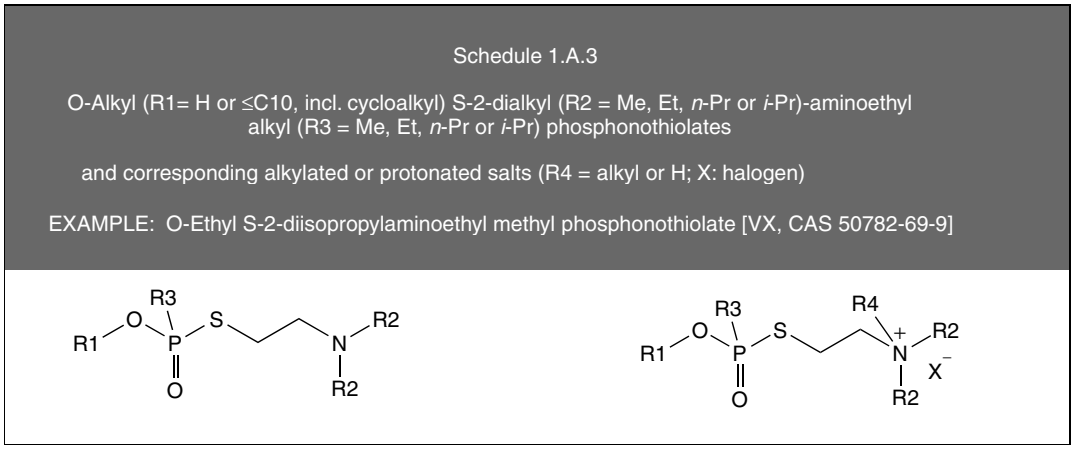

Figure 2. Definition and structures of Schedule 1.A.3 chemicals

The three Schedules contain altogether 57 list items from which 42 are individual chemicals and 15 are families of chemicals with a common structural backbone. Such families in the Schedules make the number of chemicals that are subject to verification very large. An idea of the number of chemicals in the Schedules may be obtained when considering, for example, the family of VX (Schedule 1.A.3), including its salts (Figure 2).

Another example is found in Schedule 2.B.4, which contains an even larger number of chemicals defined in the following way: 'Chemicals, except for those listed in Schedule 1, containing a phosphorus atom to which is bonded one methyl, ethyl or propyl (normal or iso) group but not further carbon atoms [exemption: Fonofos, CAS 944-22-9]'.

4 ORGANIZATION FOR THE PROHIBITION OF CHEMICAL WEAPONS (OPCW)

The States Parties to the CWC have established the Organization for the Prohibition of Chemical Weapons (OPCW; www.opcw.org) to achieve the object and purpose of the CWC, to ensure the implementation of its provisions, including those for international verification of compliance with it, and to provide a forum for consultation and cooperation among States Parties (SPs). All SPs to the CWC are members of the OPCW. The Organization shall conduct its verification activities provided under the CWC in the least intrusive manner possible, consistent with the timely and efficient accomplishment of their objectives. It shall request only the information and data necessary to fulfill its responsibilities under the CWC. It shall take every precaution to protect the confidentiality of information on civil and military activities and facilities coming to its knowledge in the implementation of the CWC and, in particular, shall abide by the provisions set forth in the Confidentiality Annex. In undertaking its verification activities, the OPCW shall consider measures to make use of advances in science and technology.

5 VERIFICATION

The OPCW performs verification activities on a regular basis and can conduct challenge inspections. The purpose is to verify that the SPs fulfill their obligations under the CWC. Regular verification includes assessment of the declarations made by the SPs by conducting on-site inspections of declared sites.

The general rules for verification (Verification Annex, Part II, paragraphs 52–54) describe sample taking (sampling, sample collection) and analysis. By way of example, sampling and analysis shall be undertaken to check for the absence of undeclared scheduled chemicals during inspections under

regime of Schedule 2 chemicals and facilities related to such chemicals. Also, sampling and on-site analysis may be undertaken to check for the absence of undeclared scheduled chemicals during inspections under regime of Schedule 3 chemicals and facilities related to such chemicals. In case of unresolved ambiguities, samples may be analyzed in a designated off-site laboratory, subject to the inspected SPs agreement. (For the summary on the sampling and analysis in the CWC, *see* Annex 1 in **Chapter 2**.)

For the on-site analysis, the inspectors bring with them mobile instrumentation capable of performing the analysis in the least intrusive manner where the chemicals are revealed only according to the purpose of the inspection and the information on nonscheduled chemicals will remain confidential (for so-called blinded analysis, *see* **Chapter 4**).

For the off-site analysis, the designated laboratories are used. These laboratories have instrumental capability, preparedness, and analytical methods to analyze the samples taken by the inspectors or by the inspected SP representatives. The samples sent (after the agreement of the inspected SP) to the off-site laboratory are coded, and therefore the laboratory receiving the samples will not know their origin. The laboratories are capable of confirming the presence or absence of CWC-related chemicals and other chemicals, but must report only data relevant to the purpose of the analysis as defined by the OPCW. The laboratory's work on the OPCW samples is confidential, which is a normal practice when regarding the work with laboratory's other collaborators and commercial business partners. The work is reported only to the OPCW.

6 THIS BOOK

Chemical Weapons Convention Chemicals Analysis discusses sample collection, sample preparation and analysis, and concentrates on verification that takes place on site, analyses off site, and methods and procedures used. In the first part of the book is discussed the mobile laboratory of the OPCW and instrumentation and software used therein, as well as other on-site analysis equipment, procedures, and strategies. The OPCW gas chromatograph–mass spectrometer for on-site analysis is described and an introduction to Automated Mass Spectrometry Deconvolution and Identification System (AMDIS) software is given. Various monitoring methods of hazardous substances are viewed. A comprehensive review to 10 OPCW proficiency tests has been done. The topics related to the OPCW Central Analytical Database (OCAD) are discussed.

The second part of the book begins with a discussion of the analysis strategy employed in an OPCW-designated laboratory and continues with a discussion on sample preparation methods in an off-site laboratory and concludes with discussion on the various analytical techniques used for analysis of CWC-related chemicals in (designated) laboratories worldwide. The analytical techniques are gas chromatography (GC), gas chromatography/mass spectrometry (GC/MS), liquid chromatography/mass spectrometry (LC/MS), nuclear magnetic resonance (NMR) spectroscopy, gas chromatography/Fourier transform infrared spectroscopy (GC/FTIR), and capillary electrophoresis (CE). The methods included in this part provide the best off-site performance for unambiguous identification of CWC-related chemicals. The success of these off-site analysis techniques has been unequivocally confirmed in the international proficiency tests. Other examples in the literature exist from excellent performance from 'real samples', for example, the detection of intact sarin by mass spectrometry from a painted metal fragment after four years of contamination.

In the third part, methods for retrospective detection of exposure to toxic scheduled chemicals using mass spectrometric and immunochemical analysis methods are discussed. The described methods are applied to human origin samples. These methods are essential when in cases of use, or allegations of use, previous presence or absence of toxic chemicals need to be confirmed. Identification of CWC-related chemicals provides key supporting evidence of noncompliance with the CWC.

ABBREVIATIONS AND ACRONYMS

AMDIS	Automated Mass Spectrometry Deconvolution and Identification System
CAS	Chemical Abstracts Service

CE	Capillary Electrophoresis
CWC	Chemical Weapons Convention
GC	Gas Chromatography
GC/FTIR	Gas Chromatography/Fourier Transform Infrared Spectroscopy
GC/MS	Gas Chromatography/Mass Spectrometry
LC/MS	Liquid Chromatography/Mass Spectrometry
NMR	Nuclear Magnetic Resonance
OCAD	OPCW Central Analytical Database
OPCW	Organization for the Prohibition of Chemical Weapons
SPs	States Parties
VX	*O*-Ethyl *S*-2-diisopropylaminoethyl methylphosphonothiolate

CHAPTER 2

Sampling and Analysis in the Chemical Weapons Convention and the OPCW Mobile Laboratory

Stefan Mogl
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1 INTRODUCTION

During the first session of the Preparatory Commission for the Organization for the Prohibition of Chemical Weapons (OPCW), which was held between 8 and 12 February 1993 in The Hague, Working Group B was established with the task of drafting procedures for 'verification and technical cooperation and assistance'. Up to the entry into force of the Chemical Weapons Convention (CWC) in April 1997, Working Group B developed procedures for the conduct of verification activities and specifications for equipment. An important part of the group's deliberations was the issue of sampling and analysis (S&A). On the basis of the recommendations of Working Group B, the First Conference of States Parties, held in The Hague in May 1997, adopted a list of approved

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equipment for inspection purposes and procedures relating to proficiency testing for the designation of laboratories for off-site analysis, thus setting the initial conditions for sampling and analysis. On the basis of this, the Technical Secretariat (Secretariat) has developed its on-site and off-site S&A capability. The following article describes the role of on-site analysis in the verification process and the OPCW mobile laboratory to be used by inspectors to perform such on-site analysis. Particular emphasis is given to the description of software tools that were developed to protect confidential information (commercial or military) and which limit the analysis capability to CWC-related chemicals.

2 ROLE OF ON-SITE ANALYSIS IN THE VERIFICATION PROCESS

The CWC [(1)] makes extensive reference to S&A in its 'Annex on Implementation and Verification (Verification Annex)'. First in part II, under General Rules of Verification, but also in later sections that describe verification activities for particular types of inspections or inspectable facilities as shown in Table 1. The full wording of the respective provisions is included in Annex 1.

S&A is one of several verification tools available to an inspection team (IT), and like any other, its purpose is to assist the IT in achieving the inspection mandate that was issued by the Director-General. From among all the tools available to the IT, S&A is special in the sense that it provides factual evidence for the presence of scheduled chemicals through detection and identification and/or supports a conclusion of absence of scheduled chemicals through analysis results. Considering the role that was given to S&A in the CWC and the potential of S&A to provide factual evidence on the presence or absence of chemicals, it can be followed that the Secretariat of the OPCW must establish and maintain the capability to perform chemical analysis.

Table 1. Provisions in the Verification Annex of the CWC for S&A. The full wording of the respective provisions is included in Annex 1. (Annotation: VA.II.52–58 reads as Verification Annex, part two, paragraph 52 to 58)

VA.II.52–58	General rules for S&A
VA.II.11(d),12	Inviolability of samples
VA.IV(A).49b,66,70	Storage, destruction facilities
VA.V.49(iii)	Production facilities
VA.VII.27	Schedule 2 facilities
VA.VIII.22	Schedule 3 facilities
VA.IX.19	Other chemical production facilities
VA.X.27c,36b,47,48	Challenge inspections
VA.XI.16–18	Investigations of alleged use
CA.16	Facility agreement: taking of samples and their analysis

The CWC provides for three principal ways to undertake chemical analysis:

(a) on-site analysis by the IT using approved inspection equipment [VA.II.53];
(b) on-site analysis conducted by the inspected State Party ((ISP)) in the presence of the IT, using equipment available at the inspection site [VA.II.53]; and
(c) off-site analysis at designated laboratories that have been certified by the Director-General for such analysis [VA.II.55].

The objective of S&A may depend on the type of inspection that is being conducted. However, the two standard inspection objectives of S&A are as follows:

- confirmation of the declaration (confirm identity of a declared chemical); and
- confirmation of absence of any undeclared scheduled chemicals, in particular, Schedule 1 chemicals.

While both objectives may pose particular challenges to S&A, the analysis for absence of any undeclared scheduled chemicals is significantly more demanding. The large number of chemicals that are theoretically possible to be synthesized based on the definitions in the Schedules of the CWC illustrate this in Table 2. A list of the Schedules of chemicals is included in Annex 2 [(2)].

Table 2 illustrates that the majority of *Schedule numbers* allow deriving only one or a few different possible chemicals. One *Schedule number* however, Schedule 2.B.4, describes millions of possibilities ('Chemicals, except for those listed in Schedule 1, containing a phosphorous atom to which is bonded one methyl, ethyl or propyl (normal or iso) group but no further carbon atoms'); and more importantly, the theoretical possibilities for the nerve agents

Table 2. Estimated number of possible chemicals that can be derived from the definitions for scheduled chemicals contained in the Annex on Chemicals of the CWC not counting corresponding protonated or alkylated salts, where this is applicable

Schedule number	(Estimated) number of chemicals	Schedule number	(Estimated) number of chemicals	Schedule number	Number of chemicals
1.A.1	>20 000[a]	2.A.1	1	3.A.1	1
1.A.2	>50 000[a]	2.A.2	1	3.A.2	1
1.A.3	>200 000[a]	2.A.3	1	3.A.3	1
1.A.4	9	2.B.4	Millions	3.A.4	1
1.A.5	3	2.B.5	20[b]	3.B.5	1
1.A.6	3	2.B.6	100	3.B.6	1
1.A.7	1	2.B.7	1	3.B.7	1
1.A.8	1	2.B.8	1	3.B.8	1
1.B.9	4	2.B.9	1	3.B.9	1
1.B.10	>200 000[a]	2.B.10	10	3.B.10	1
1.B.11	1	2.B.11	8	3.B.11	1
1.B.12	1	2.B.12	10	3.B.12	1
–	–	2.B.13	1	3.B.13	1
–	–	2.B.14	1	3.B.14	1
–	–	–	–	3.B.15	1
–	–	–	–	3.B.16	1
–	–	–	–	3.B.17	1

[a]Including branched chains and cyclo alkane chains, not including bicyclo alkane chains and stereoisomers and not including corresponding protonated and alkylated salts
[b]Only including dichlorides and difluorides

in Schedule 1.A.1 to 1.A.3 exceed two hundred seventy thousand. Schedule 1.B.10 chemicals are precursors to Schedule 1.A.3 and respectively represent the same number of possibilities.

While only few of all these theoretically possible Schedule 1 chemicals have ever been stockpiled as chemical weapons, all of them are scheduled chemicals. In order for the Secretariat to be able to check for absence of any undeclared scheduled chemicals, the methods used for analysis should be capable of detecting and identifying as many of them as possible.

The CWC provides for different types of inspections and as stated above, the actual role of S&A in a particular inspection depends on the type of inspection being conducted. Table 3 lists types of inspections provided for in the CWC and the particular objective of S&A for each one.

The role of S&A at a Chemical Weapons Destruction Facility (CWDF) is different than in other types of inspections. At a CWDF, the main objective of S&A is to confirm the identity of a particular chemical that has been declared, that is, the declared chemical agent that is being destroyed. Further,

Table 3. Different types of inspections and the respective purpose of S&A

Type of Inspection	Main purpose of S&A
Inspection of Schedule 1 facility (other facility)	Confirm absence of nondeclared activity
Inspection of Schedule 2 facility Inspection of Schedule 3 facility Other chemical production facilities	Confirm absence of nondeclared chemicals, in particular, Schedule 1 chemicals
Chemical weapons storage facilities	Tagging of items for later S&A at CWDF
Chemical weapons destruction facilities (CWDF)	Confirm identity of chemical agent being destroyed, confirm nondiversion of agent and end point of destruction of chemical agent
Challenge inspections	Confirm absence of any nondeclared scheduled chemical(s)
Investigation of alleged use	Confirm absence of chemical weapons and riot control agents

S&A's objective is also to confirm that this chemical is not contained in the effluent streams above a certain concentration. This difference in the objective of S&A compared to that of confirming absence of nondeclared scheduled chemicals has influenced the approach taken by the Secretariat for the conduct of on-site analysis.

The next section explains how the two approaches, analysis of a declared chemical and analysis for undeclared scheduled chemicals, have influenced on-site analysis activities and how on-site laboratories are or may be used for such analysis.

It must be stated however, that the CWC would also allow for a different interpretation of the purpose of S&A at a CWDF. Paragraph 66 (c) of part IV (A) states that 'the specific type and quantity of chemical weapons being destroyed' should be part of systematic on-site verification measures. This would also allow analyzing for any undeclared scheduled chemicals being destroyed in addition to the declared chemical.

3 LABORATORIES FOR ON-SITE ANALYSIS

Sample collection will generally be conducted by the inspected State Party (during investigations of alleged use (IAU), the inspection team may collect the samples itself), however, the IT may collect the sample(s) if agreed in advance between the inspected State Party and the IT [VA.II.52]. The IT has the right to conduct sample analysis using its own approved equipment **or** it may witness analysis performed by the inspected State Party [VA.II.53]. This leads to *two different concepts* for on-site analysis as laid out below.

It is important to understand that OPCW inspectors must be able to demonstrate to all States Parties that all analysis results have been obtained on independent and verifiable bases, whilst remaining flexible to specific site conditions and requirements.

3.1 On-site Analysis Conducted by the Inspected State Party and Witnessed by the Inspection Team (IT)

If the analysis is conducted by the inspected State Party, OPCW inspectors are in the role of witnessing the analysis performed by the site personnel. This has various implications because analytical technique, sample preparation and analysis procedures, and quality assurance and quality control measures may be different to the ones described in OPCW procedures.

From a practical point of view, it may be rather difficult to verify the proper operation of computer programs or the identity of chemicals used by site personnel, and, as a consequence, the correctness of analysis results. This, in particular, if the analysis is checking for undeclared scheduled chemicals and not aiming to confirm the presence of a declared scheduled chemical. In order for an analysis for absence of undeclared scheduled chemicals to be credible for verification purposes, it must be conducted in accordance with OPCW procedures, fulfilling OPCW QA/QC (quality assurance/quality control) criteria and using the OPCW Central Analytical Database (OCAD) as reference [3]. [The OCAD contains peer-reviewed validated analytical data of Scheduled chemicals (mass spectra, gas chromatography retention indices, infrared spectra, nuclear magnetic resonance spectra) that has been approved for inclusion into the database by the policy making organs of the OPCW.]

Therefore, on-site analysis conducted by the inspected State Party based on its own procedures is used routinely only in CWDF. The main focus of analysis at CWDF is to confirm the identity of the agent being destroyed and its absence in the effluent streams as described above. *Agent identity* is confirmed by GC/MS (gas chromatograph/mass spectrometer) analysis of an agent sample; mass spectrum and retention index are compared to the OCAD. Alternatively, in place of retention index comparison, the retention time of the agent peak is compared to the retention time of an agent reference standard provided by the inspected State Party. *Absence of agent in effluent streams* is frequently proven by GC analysis using absence of a peak in the retention time window of the agent. The retention time window has been established by analyzing the standard described above. If the analysis requires sample preparation, such as solvent extraction, an aliquot of the sample matrix is spiked with the reference standard within the detection range of the method and analyzed in parallel for QA/QC purposes.

3.2 On-site Analysis Conducted by the IT

If the IT is conducting on-site analysis using its own equipment, the view taken has been that the team should be independent and carry the entire equipment necessary. This led to the design of the OPCW mobile laboratory, which is described in Section 4.

The preparation of the samples for analysis and the analyses itself will be performed by the IT using its own equipment approved for this purpose. Sample preparation and analysis is conducted in accordance with OPCW standard operating procedures (SOP) and work instructions (WI) applying OPCW QA/QC criteria.

Within this approach lays the possibility of various levels of assistance by the inspected State Party offering equipment, such as the use of inspected site laboratory facilities, fumed hoods, and the like. Such offers are laid down in facility agreements concluded between the inspected site and the OPCW, following an initial inspection. However, fundamental to this approach is that sample preparation and analysis are conducted by OPCW inspectors according to OPCW-approved procedures with equipment that meets OPCW QA/QC criteria. All activities may be witnessed by inspected State Party representatives.

The following section describes the design and capabilities of the OPCW mobile laboratory.

4 THE OPCW MOBILE LABORATORY

The OPCW mobile laboratory is designed for use in all types of inspections. It is able to function self-contained, if necessary in a tent powered by electricity generators. It contains sufficient equipment to allow the IT the collection and preparation of various types of sample matrices and the GC/MS analysis of their extracts. All items of equipment are packaged in *flight cases* in such a way that two persons can move each transport container by hand.

4.1 Equipment Requirements and Specifications

Any item of equipment that is carried by an OPCW IT must have been approved for this purpose. All equipment that is currently used or allowed for use in inspections is contained on a *list of approved equipment* that was adopted by the Conference of States Parties at its first sessions in May 1997, in decision 71 (C-I/DEC.71) [(4)]. This list of approved equipment was the result of extensive negotiations held during the period of the preparatory commission of the OPCW in the expert groups of Working Group B. For each item of equipment 'general and specific operational requirements, common evaluation criteria and technical specifications' were defined in Annexes to C-I/DEC.71. In C-I/DEC.71, individual items are listed together according to their intended purpose in particular equipment kits. An excerpt of C-I/DEC.71, the GC/MS sample preparation kit, is presented as an example in Annex 3.

Following is a description of the purpose and design of the OPCW mobile laboratory and its respective equipment kits. While the list of approved equipment in C-I/DEC.71 contains other items for S&A (i.e. FTIR, Alleged Use Sample Collection Kit), the items described in this article are the ones that have been actually procured and are actively used by OPCW ITs at the time of this publication. A more detailed description of S&A equipment kits can be found in **Chapter 3**.

4.2 Sample Collection Kit

The sample collection kit has been designed to allow the IT to collect bulk solid, soil, water, liquid and wipe samples. It contains enough items to collect eight samples of each matrix. All items intended to come into contact with the sample are packed individually for one time use. The items are arranged in transport boxes (flight cases) in individual drawers (Picture 1). Empty 'pelli cases' are included to allow the inspectors to assemble and carry forward specific items to the sampling location suitable for the particular sampling activity. A reduced version of the sample collection kit packaged in a briefcase is available for inspections where this will suffice.

The Secretariat developed sample collection procedures in accordance with its quality system that describe the collection of different types of samples (see Table 4).

Picture 1. Some items of sample collection kit

Picture 2. Sample preparation kit

Table 4. Secretariat quality documents describing the collection of samples during on-site inspection. The documents listed in the table are part of the Secretariat's quality management system and undergo regular revision. Thus, their names and codes may change in future

Work instruction for:	Secretariat reference
Collecting and splitting of toxic samples under hazardous conditions on-site	QDOC/LAB/WI/SC1
Collection and splitting of samples under non-hazardous conditions	QDOC/LAB/WI/SC2

4.3 Sample Preparation Kit

The sample preparation kit (Picture 2) holds all equipment necessary to prepare the samples for GC/MS analysis on-site in four transport boxes (flight cases). It contains everything from the pH paper to the laboratory coat including the chemicals to conduct the sample preparation procedures. A reduced version of the sample preparation kit packaged in two pelli cases is available for inspections where this will suffice.

The objective of all sample preparation procedures is to extract the analytes of interest from the different sample matrices and then to bring them into a form that they can pass through a GC column, using derivatization if required. One of the key items of the kit and next to the fume hood the most bulky one is the centrifugal evaporator that allows a gentle evaporation of water in order to isolate polar analytes that remain in the water fraction after solvent extraction. The Secretariat drafted generic sample preparation procedures as shown in Table 5. These procedures use liquid/liquid and solid/liquid extraction and are with slight modifications based on the VERIFIN Blue Books [5]. A detailed description of the sample preparation kit and methods can be found in **Chapter 3**.

4.4 Sample Transport Kits

The list of approved equipment contains two sample transport containers designed for the transport of samples for off-site analysis. The two containers are labelled as 'large- and small sample transport kits' (Pictures 3 and 4) and both are designed to fulfill the requirements for air transport [IATA (International Air Transport Association) provision A106] [6] and international standards for road, railway, and sea transport. The large container has been designed and tested by the United States and the small container by the United Kingdom. Therefore, the two containers are frequently referred to as US and UK containers.

Table 5. Secretariat quality documents describing the preparation of samples during on site inspection for GC/MS analysis, the destruction of sample material and the chain of custody documentation. The documents listed in the table are part of the Secretariat's quality management system and undergo regular revision. Thus, their names and codes may change in future

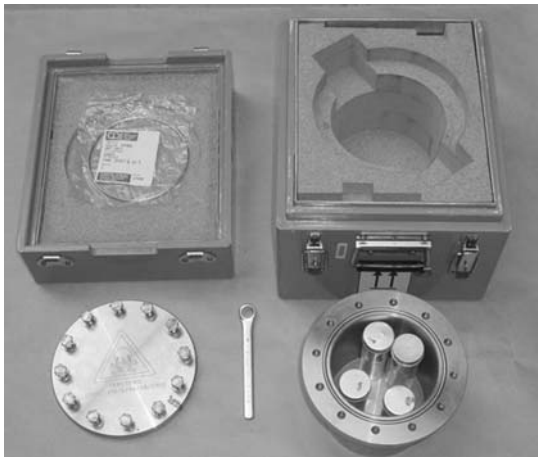
Work instruction for:	Reference
Preparation of samples on-site for GC/MS analysis	QDOC/LAB/WI/SP2
The chain of custody and documentation for OPCW samples on site	QDOC/LAB/WI/OSA3
The destruction of sample material	QDOC/LAB/WI/SP016

Picture 3. Large sample transport kit

The small container is approved for the transport of 10 g of pure material or 400 g of diluted material and the large container is approved for 350 ml of any material.

4.5 GC/MS

The OPCW mobile laboratory includes a portable GC/MS. The system shown in Picture 5 including printer and helium connection kit is packed in five transport boxes. Because of the modular design of the instrument, it is shipped with two GC-ovens and two GC-injectors in order to allow the inspector on-site to change the inlet and separation part should problems occur. At the time of writing, the Secretariat was in the process of replacing the GC/MS shown in Picture 5 with a standard bench top instrument adapted for OPCW purposes. Before any GC/MS is packed and dispatched from the Secretariat it is tested at the OPCW Laboratory in accordance with the Secretariat's quality system. A detailed description of the testing procedure and the instrument QA/QC criteria can be found in **Chapter 4**. For each GC/MS to be dispatched a certificate of the office of the internal oversight



Picture 4. Small sample transport kit

Picture 5. Bench top GC/MS instrument

(OIO) of the Secretariat is issued to confirm that the system meets all relevant performance criteria. Both organizational units, the OPCW Laboratory and the OIO, are accredited by the Dutch Accreditation Council (RvA) for this activity.

Two types of portable analytical equipment were approved by the Conference of State Parties for on-site analysis, that is, GC/MS and Fourier transform infrared (FTIR). Initially, it was anticipated that FTIR might be used in storage, destruction and Schedule 1 facilities for screening purposes to confirm the presence of declared chemicals. While FTIR analysis is suitable to identify pure chemicals or certain chemicals in mixtures at varying detection limits, FTIR cannot be used to analyze for absence of undeclared scheduled chemicals, for which the IT would have to carry in addition to the FTIR a GC/MS. For GC/MS analysis samples are analyzed at very low concentration contrary to FTIR analysis, which minimizes the risk of contamination in the on-site laboratory and reduces the risk of exposure of OPCW inspectors and on-site personnel. Considering the limitations of FTIR and the fact that GC/MS can cover the field of application of the FTIR for OPCW purposes, a decision was taken in 2000 to focus on the use of GC/MS in the OPCW mobile laboratory subject to future developments.

The Secretariat developed a set of quality documents for the preparation and handling of the GC/MS system (see Table 6).

Table 6. Secretariat quality documents describing the preparation and handling of the GC/MS system. The documents listed in the table are part of the Secretariat's quality management system and undergo regular revision. Thus, their names and codes may change in future

Work instruction for:	Reference
Bruker EM 640S portable GC/MS on-site analysis	QDOC/LAB/WI/GCMS001
Bruker EM 640S GC/MS testing and preparation of instruments for on-site analysis	QDOC/LAB/WI/GCMS002
Bruker EM 640S portable GC/MS packing procedures	QDOC/LAB/WI/GCMS003
Bruker EM 640S portable GC/MS installation and handling of software	QDOC/LAB/WI/GCMS004

5 BLINDING OF THE GC/MS INSTRUMENT

Confidentiality concerns, in particular, in relation to inspections of industry facilities and in relation to activities not prohibited by the Convention have led to several measures aimed at protecting sensitive information. In order to allow the inspected State Party to retain all data produced by the GC/MS, the equipment is operated from a removable hard disk, which may be retained on site at the end of the inspection. Another measure has been the design of dual mode software for the GC/MS by modifying the operating software of the instrument to offer operation in so-called open or blinded mode as explained in the following.

Open mode provides standard functionality of the GC/MS; blinded mode limits the information that is revealed by the instrument to the operator. If operated in blinded mode, the instrument will only display or reveal information on those chemicals contained in the sample that are relevant to assess presence or absence of scheduled chemicals. In order to achieve this the data postprocessing software can only search a specific mass spectral library that is an extract of the OCAD. This OCAD only contains chemicals that have been approved for inclusion into the database by the policy making organs of the OPCW. For a full description of the OCAD, please refer to **Chapter 7**. Two independent software modules were developed, which used independently or together limit the information that is displayed by the instrument. One is the *blinding feature* of the instrument operating system and the other the *negotiation module* of the data postprocessing software AMDIS (Automated Mass Spectral Deconvolution and Identification System developed by the United States Department of Commerce, National Institute of Standards and Technology (NIST)), which provides for different so-called *security levels*.

During the set-up process of the GC/MS instrument that is shown in Picture 5, the negotiation module is used to install the *on-site target library* and to select the *security level filter* of AMDIS (this approach may be modified for the new GC/MS system). The *security level filter*s of AMDIS progressively restrict the accessibility to and the content of the data that AMDIS provides after postprocessing of the spectral data. In order to protect

the selections made in the negotiation module, the IT and the inspected State Party enter a password. Any changes require both passwords to be entered. After installation of the library and selection of the security level filters, the decision must be made whether the instrument is to be operated in blinded mode or in open mode; in the first case, the blinding feature is activated; in the latter it is not.

Figure 1 shows schematically the relationship between *on-site target library, blinding*, and *AMDIS security level filter*.

The center of Figure 1 (not shaded) shows the GC/MS instrument *640S GCMS* allowing to *monitor method parameters* and producing *on-line data*; at the end of the analysis, *triggering* data processing with *AMDIS on-site version* and allowing for *postrun data* processing.

On the left side, the window labelled *available in open mode only*, displays features available with a standard GC/MS. This includes a *file manager* to handle files, a *word processor* to edit files, *display of online data* during analysis (total ion chromatogram (TIC) and mass spectrum) and after processing of *postrun data* using one of the two installed data analysis software: *Bruker DA* or *AMDIS open version*.

On the right side the window labelled *AMDIS on-site version* displays 5 security level filters of AMDIS gradually limiting the information displayed to the operator as described in Table 7.

(a) A *source library* on 3.5 in. floppy disk or CD-ROM is prepared at the OPCW Laboratory from the OCAD and issued with a certificate of authenticity from the OIO together with the GC/MS instrument to the IT. This source library contains mass spectra and retention indices data from the OCAD.

(b) In the *negotiation module*, the chemicals from this *source library* that should be copied into the *on-site target library* can be selected on the basis of a chemical name or a schedule number; by default, the content of the *on-site target library* includes all chemicals of the *source library*. In order to install the mass

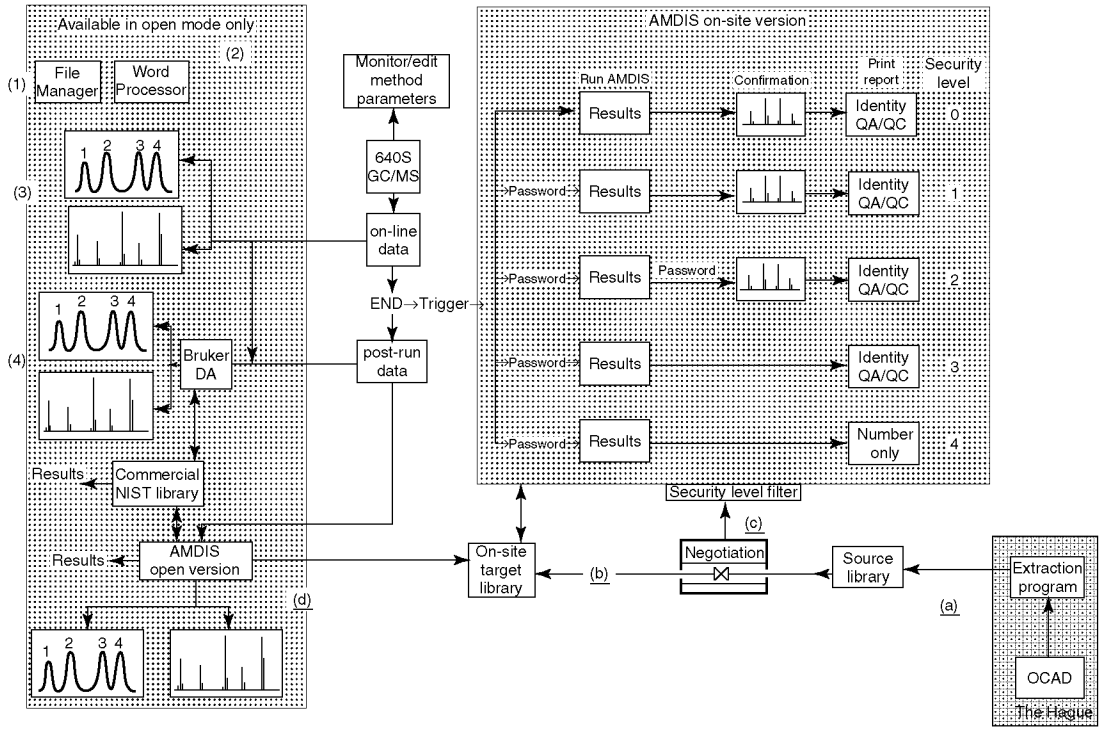


Figure 1. Relationship between *on-site target library, blinding*, and *AMDIS security level filter*

Table 7. The effects of the different AMDIS security level filters

Security level 0	*AMDIS on-site version* data analysis software is triggered automatically after each GC/MS run. The output includes names, net match factor, retention times and retention indices, and a set of quality assurance parameters, i.e. peak width, peak tailing, signal to noise ratio of each identified chemical and the internal standard. In addition, general gas chromatography QA/QC parameters such as background, solvent tailing and bleeding are reported. The software allows the opening of the *AMDIS confirmation window,* which displays the TIC of identified peaks, mass spectra at peak maximum and extracted spectrum after deconvolution; and the corresponding mass spectrum of a library hit in the *on-site target library*.
Security level 1	Includes the same features as security level 0. Further, entering of both passwords is requested to start *AMDIS on-site version* data analysis software following a GC/MS analysis run. Because of this, data analysis can only be performed in the presence of both IT and ISP.
Security level 2	In addition to security level 1, the opening of the *AMDIS confirmation window,* which displays the TIC of identified peaks and their mass spectra requires the passwords of the ISP and IT. Because of this, again, data analysis can only be performed in the presence of both IT and ISP.
Security level 3	In this security level, the *AMDIS confirmation window* is no longer available. As a result, the IT can no longer compare mass spectra of an analyte identified by AMDIS to the respective library spectra.
Security level 4	The restrictions of security level 3 apply. In addition, AMDIS only reports how many chemicals it identified. No further information is provided such as chemical names, retention time/indices and QA/QC parameters. An internal standard, which is injected with each analysis should always be identified by AMDIS. However, because AMDIS will only report i.e. 'one chemical identified' it remains unknown if the internal standard or any other chemical was in fact detected; thus, this security level produces results of very limited value.

Table 8. Effects of operating instrument in 'blinded' mode as indicated in Figure 1

Effects of blinding	Effects on analysis
(1) File manager deleted	No copying of files possible
(2) Word processor deleted	No editing of files
(3) No display of real-time data	No display of TIC or mass spectra on PC screen during data acquisition
(4) Bruker mass spectral data analysis software and AMDIS open version deleted	The instrument is issued by the OPCW Laboratory with three data postprocessing software tools: • Bruker mass spectral data analysis software from the instrument provider; • *AMDIS open version*, a full version of AMDIS mass spectral data analysis software; and • *AMDIS on-site version*, mass spectral data analysis software, which is triggered at the end of each analysis run. This reduced on-site version of AMDIS only displays TICs and mass spectra of analytes that match a chemical in the on-site target library. As a result only peak(s) and mass spectra of analytes identified by AMDIS in the on-site target library are displayed
(5) No search of libraries other than on-site target library.	The library search function is altered. AMDIS on-site version can only search in the on-site target library.

spectra library onto the GC/MS computer hard disk, the passwords entered in the negotiation module are required (inspected State Party and IT). As a result, it is not possible to change the content of the *on-site target library* without both parties entering their respective passwords.

(c) The *security level filter* of *AMDIS on-site version* is selected with the effects as indicated in Table 7. The *security level filters* may be changed at any time by entering the required passwords. Data of a previous analysis may be reanalyzed at a different security level.

(d) If the software switch for *blinded mode* is selected, the effects will be as indicated in Table 8. Contrary to the *AMDIS security level filters*, blinding is irreversible on a particular PC hard disk. Returning the instrument to open mode requires formatting of the hard disk and reinstallation of all operating software. In order to facilitate the return of the system into open mode during an inspection, the instrument is dispatched with two removable hard disks. This allows the IT to replace a blinded mode hard disk and to reanalyze the samples in open mode if necessary.

At the time of writing, a new blinded mode software was being developed. This new version is based on different GC/MS operating software and will include additional security features such as unique installation numbers and check sum. Also, the need of AMDIS security level filters is being reviewed.

6 LIMITATIONS OF THE OPCW MOBILE LABORATORY

The OPCW mobile laboratory equipment has been selected to allow operation of a GC/MS laboratory on site, aiming at a performance similar to the one of a stationary laboratory using a standard bench top instrument. However, restrictions in the selection and operation of the equipment (i.e. protection of confidential information) and issues related to logistics/transport of the laboratory equipment cause certain limitations, which are described in the paragraphs below.

The capabilities of the mobile laboratory depend to a large extend on the equipment and procedures that are approved for the use on site. Limitations are described best by discussing them separately for sample collection, sample preparation, GC/MS equipment, and analysis procedures.

6.1 Sample Collection Equipment

The equipment approved by the first Conference of States Parties in C-I/DEC.71 for collecting samples for on-site analysis is sufficient and adequate to collect samples of various types of matrices, environmental, and bulk. The sample collection kit as it is designed now allows for the collection of a limited number of samples of each type, about eight, but could be extended if there was a need. The key limitation in relation to sample collection at this point in time is that the kit does not contain equipment for the collection of air samples and that there are no respective procedures in place. A more general limitation is the very detailed specifications in the list of approved equipment given for each equipment item, rendering any change or development impossible without approval by the policy-making organs.

6.2 Sample Preparation Equipment

This has particular limiting effects for sample preparation because of the detailed specifications given for equipment and chemicals approved for the sample preparation kit. The items of the sample preparation kit and not their purpose have been specified in detail. It turned out that some items, originally approved on the list, where not available on the market, except if custom produced for the OPCW. Because of these detailed specifications, developments in the area of sample preparations cannot easily be applied to on-site analysis. As a result, any change to sample preparation procedures that require an additional item of equipment, or more so an additional chemical, such as a different solvent (i.e. for sample clean up), cannot be implemented until the list of approved equipment is amended.

Current sample preparation procedures for water samples use low temperature vacuum evaporation of water with a centrifugal evaporator followed by derivatization. They are time consuming and

require bulky equipment. Any sample preparation procedure that would allow obtaining/derivatization of analytes without slow elimination of water would greatly speed up the sample preparation process and reduce the amount of equipment that has to be carried.

The list of chemicals approved to be brought by the IT and to be used in the OPCW mobile laboratory on site contains no scheduled chemicals in order to prevent any contamination of on-site samples. Therefore, the IT cannot compare analytes detected by GC/MS analysis to reference standards or synthesize these standards on site.

6.3 GC/MS instrument

The specifications approved for the GC/MS instrument allow the selection of an instrument with performance criteria of a standard GC/MS bench top system. The requirements specified in relation to blinding and transportability are substantial and require significant adaptations to any system currently available on the market. However, they do not limit analysis performance in terms of sensitivity or selectivity. The current system specifications do not include chemical ionization and limit analysis to electron impact spectra.

The system is dispatched with a commercial spectral database and a copy of the OCAD, which are both available if analysis is conducted in open mode. If the instrument is operated in blinded mode and AMDIS security level filters are applied, the limitations as outlined in Section 5 above apply.

The content of the OCAD is currently limited to scheduled chemicals, their degradation products and some derivatives, and depends on the data contributed to the OPCW by Member States. The current version of the OCAD (June 2004) is sufficient for on-site analysis; it contains the mass spectra of about 2000 chemicals. A detailed description of the OCAD can be found in **Chapter 7**.

7 THE OPCW S&A NETWORK

While the OPCW mobile laboratory is an important tool in the verification process, it is only one element of the OPCW S&A network. In Section 1, it was explained that the CWC provides for three principal ways to undertake chemical analysis, one of them being off-site analysis at designated laboratories. The CWC provides for off-site analysis for all types of inspections but off-site analysis is subject to agreement of the inspected State Party in the case of Schedule 3 inspections and inspections of other chemical production facilities (OCPF). In the event of off-site analysis, the Director-General must select a minimum of two laboratories from a list of laboratories that have been designated by him.

7.1 OPCW Proficiency Testing

The OPCW Secretariat conducts official OPCW proficiency tests that lead to designation by the Director-General; under normal circumstances, two tests per year. Until July 2004, 15 such tests have been conducted. All Member States are invited to nominate laboratories to participate in these proficiency tests. For laboratories to become designated, they must participate successfully three times consecutively and they must obtain an accreditation for the analysis of chemical weapons–related chemicals by their national accreditation bodies. It is the aim of the OPCW to have designated laboratories from a geographical distribution as wide as possible. Currently, there are 18 laboratories designated in four regional groups. A comprehensive description of the OPCW proficiency testing can be found in **Chapter 6**.

7.2 The Role of the OPCW Laboratory in Rijswijk during Off-site Analysis

In the event of off-site analysis, authentic samples will pass from the inspection site through the OPCW Laboratory to designated laboratories. At the OPCW Laboratory, the samples will be unpacked from their transport container(s) to confirm identity by checking seal numbers and sample weight. Before the samples are repackaged and dispatched to designated laboratories, preanalyzed spiked control samples and matrix blanks from similar matrix (water, organic solvent, soil) are prepared at the OPCW laboratory for distribution together with the authentic samples. A designated laboratory receives for each sample, three nonindicated vials containing sample, spiked control, and blank. During the entire

process, the authentic samples remain sealed until their arrival at a designated laboratory. Off-site analysis results are assessed by the OPCW laboratory for correct analysis of control samples and blanks, for conforming to relevant reporting criteria and for consistency of results between designated laboratories.

ABBREVIATIONS AND ACRONYMS

AMDIS	Automated Mass Spectral Deconvolution and Identification System
BSTFA	Bis-(trimethylsilyl)trifluoroacetamide
CWC	Chemical Weapons Convention
CWDF	Chemical Weapons Destruction Facility
DMT	Dimercaptotoluene
FTIR	Fourier Transform Infrared
GC/MS	Gas Chromatograph/Mass Spectrometer
IATA	International Air Transport Association
IAU	Investigations of Alleged Use
ISP	Inspected State Party
IT	Inspection Team
NIST	National Institute of Standards and Technology
OCAD	OPCW Central Analytical Database
OCPF	Other Chemical Production Facilities
OIO	Office of the Internal Oversight
PFIB	1,1,3,3,3-Pentafluoro-2-trifluoromethyl)-1-propene
QA/QC	Quality Assurance/Quality Control
QL	O-Ethyl O-2-diisopropylaminoethyl methylphosphonite
SOP	Standard Operating Procedures
THF	Tetrahydrofuran
TIC	Total Ion Chromatogram
VX	O-Ethyl S-2-diisopropylaminoethyl methyl phosphonothiolate
WI	Work Instructions

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ANNEX 1

Provisions for sampling and analysis in the Verification Annex of the CWC

PART II: General Rules of Verification

11. To exercise their functions effectively, inspectors and inspection assistants shall be accorded privileges and immunities as set forth in subparagraphs (a) to (i). . .

    (d) Samples and approved equipment carried by members of the inspection team shall be inviolable subject to provisions contained in this Convention and exempt from all customs duties. Hazardous samples shall be transported in accordance with relevant regulations.

12. When transiting the territory of non-inspected States Parties, the members of the inspection team shall be accorded the privileges and immunities enjoyed by diplomatic agents pursuant to Article 40, paragraph 1, of the Vienna Convention on Diplomatic Relations. Papers and correspondence, including records, and samples and approved equipment, carried by them, shall be accorded the privileges and immunities set forth in paragraph 11 (c) and (d).

Collection, handling and analysis of samples

52. Representatives of the inspected State Party or of the inspected facility shall take samples at the request of the inspection team in the presence of inspectors. If so agreed in advance with the representatives of the inspected State Party or of the inspected facility, the inspection team may take samples itself.
53. Where possible, the analysis of samples shall be performed on-site. The inspection team shall have the right to perform on-site analysis of samples using approved equipment brought by it. At the request of the inspection team, the inspected State Party shall, in accordance with agreed procedures, provide assistance for the analysis of samples on-site. Alternatively, the inspection team may request that appropriate analysis on-site be performed in its presence.
54. The inspected State Party has the right to retain portions of all samples taken or take duplicate samples and be present when samples are analysed on-site.
55. The inspection team shall, if it deems it necessary, transfer samples for analysis off-site at laboratories designated by the Organization.
56. The Director-General shall have the primary responsibility for the security, integrity and preservation of samples and for ensuring that the confidentiality of samples transferred for analysis off-site is protected. The Director-General shall do so in accordance with procedures, to be considered and approved by the Conference pursuant to Article VIII, paragraph

21(i), for inclusion in the inspection manual. He shall:

(a) Establish a stringent regime governing the collection, handling, transport and analysis of samples;
(b) Certify the laboratories designated to perform different types of analysis;
(c) Oversee the standardization of equipment and procedures at these designated laboratories, mobile analytical equipment and procedures, and monitor quality control and overall standards in relation to the certification of these laboratories, mobile equipment and procedures; and
(d) Select from among the designated laboratories those which shall perform analytical or other functions in relation to specific investigations.

57. When off-site analysis is to be performed, samples shall be analysed in at least two designated laboratories. The Technical Secretariat shall ensure the expeditious processing of the analysis. The samples shall be accounted for by the Technical Secretariat and any unused samples or portions thereof shall be returned to the Technical Secretariat.
58. The Technical Secretariat shall compile the results of the laboratory analysis of samples relevant to compliance with this Convention and include them in the final inspection report. The Technical Secretariat shall include in the report detailed information concerning the equipment and methodology employed by the designated laboratories.

PART IV (a): Destruction of Chemical Weapons and its Verification Pursuant to Article IV

Inspections and visits

49. Inspectors shall, in accordance with facility agreements:

(b) Have the right, during the first and any subsequent inspection of each chemical weapons storage facility, to designate munitions, devices, and containers from which samples are to be taken, and to affix to such munitions, devices, and containers a unique tag that will indicate an attempt to remove or alter the tag. A sample shall be taken from a tagged item at a chemical weapons storage facility or a chemical weapons destruction facility as soon as it is practically possible in accordance with the corresponding destruction programmes, and, in any case, not later than by the end of the destruction operations.

Systematic on-site verification measures at chemical weapons destruction facilities

67. Inspectors shall have the right to tag, for sampling, munitions, devices, or containers located in the temporary holding areas at the chemical weapons destruction facilities.
70. Inspectors shall, in accordance with facility agreements:. . .

(b) Monitor the systematic on-site analysis of samples during the destruction process; and
(c) Receive, if necessary, samples taken at their request from any devices, bulk containers and other containers at the destruction facility or the storage facility there at.

PART V: Destruction of Chemical Weapons Production Facilities and its Verification Pursuant to Article V

Systematic verification of chemical weapon production facilities and cessation of their activities

49. The detailed facility agreement for each chemical weapons production facility shall specify:

(a) Detailed on-site inspection procedures, which may include: . . . .

(iii) Obtaining and analyzing samples;

PART VII: Activities not Prohibited Under this Convention in Accordance with Article VI

Regime for Schedule 2 Chemicals and Facilities Related to such Chemicals

Inspection procedures

27. Sampling and analysis shall be undertaken to check for the absence of undeclared scheduled chemicals.

PART VIII: Activities not Prohibited Under this Convention in Accordance with Article VI

Regime for Schedule 3 Chemicals and Facilities Related to such Chemicals

Inspection procedures

22. Sampling and on-site analysis may be undertaken to check for the absence of undeclared scheduled chemicals. In case of unresolved ambiguities, samples may be analyzed in a designated off-site laboratory, subject to the inspected State Party's agreement.

PART IX: Activities not Prohibited Under this Convention in Accordance with Article VI

Regime for other Chemical Production Facilities

Inspection procedures

19. Sampling and on-site analysis may be undertaken to check for the absence of undeclared scheduled chemicals. In cases of unresolved ambiguities, samples may be analyzed in a designated off-site laboratory, subject to the inspected State Party's agreement.

PART X: Challenge Inspections Pursuant to Article IX

Securing the site, exit monitoring

27. Additional procedures for exit monitoring activities as agreed upon by the inspection team and the inspected State Party may include, inter alia: . . .

    (c) Sample analysis.

Perimeter Activities

36. In conducting the perimeter activities, the inspection team shall have the right to: . . .

    (b) Take wipes, air, soil or effluent samples;

Managed Access

47. The inspected State Party shall designate the perimeter entry/exit points to be used for access. The inspection team and the inspected State Party shall negotiate: the extent of access to any particular place or places within the final and requested perimeters as provided in paragraph 48; the particular inspection activities, including sampling, to be conducted by the inspection team; the performance of particular activities by the inspected State Party; and the provision of particular information by the inspected State Party.
48. In conformity with the relevant provisions in the Confidentiality Annex the inspected State Party shall have the right to take measures to protect sensitive installations and prevent disclosure of confidential information and data not related to chemical weapons. Such measures may include, inter alia: . . .

    (e) Restriction of sample analysis to presence or absence of chemicals listed in Schedules 1, 2 and 3 or appropriate degradation products;

PART XI: Investigations in Cases of Alleged use of Chemical Weapons

Sampling

16. The inspection team shall have the right to collect samples of types, and in quantities it considers necessary. If the inspection team deems it necessary, and if so requested by it, the inspected State Party shall assist in the collection of samples under the supervision of inspectors or inspection assistants. The inspected State Party shall also permit and cooperate in the collection of appropriate control samples from areas neighboring the site of the alleged use and from other areas as requested by the inspection team.
17. Samples of importance in the investigation of alleged use include toxic chemicals, munitions and devices, remnants of munitions and devices, environmental samples (air, soil, vegetation, water, snow, etc.) and biomedical samples from human or animal sources (blood, urine, excreta, tissue etc.).
18. If duplicate samples cannot be taken and the analysis is performed at off-site laboratories, any remaining sample shall, if so requested, be returned to the inspected State Party after the completion of the analysis.

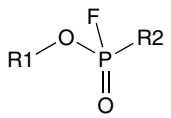
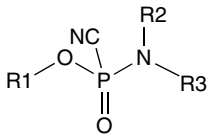
Confidentiality Annex

C. Measures to Protect Sensitive Installations and Prevent Disclosure of Confidential Data in the Course of On-site Verification Activities

16. In the elaboration of arrangements and facility agreements, due regard shall be paid to the requirement of protecting confidential information. Agreements on inspection procedures for individual facilities shall also include specific and detailed arrangements with regard to the determination of those areas of the facility to which inspectors are granted access, the storage of confidential information on-site, the scope of the inspection effort in agreed areas, the taking of samples and their analysis, the access to records and the use of instruments and continuous monitoring equipment.

ANNEX 2: SCHEDULES 1–3

Adapted from ref. (2), M. Mesilaakso, and M. Rautio, Verification of Chemicals related to the Chemical Weapons Convention, **Table 1**, Encyclopedia of Analytical Chemistry, Wiley, 2000

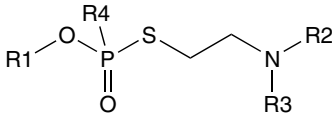
Schedule 1	
A. Toxic chemicals (CAS reg. No.)	Structure
(1) *O*-Alkyl (R_1: $\leq C_{10}$, incl. cycloalkyl) alkyl (R_2: Me, Et, *n*-Pr or *i*-Pr)-phosphonofluoridates e.g. Sarin: *O*-Isopropyl methylphosphonofluoridate (107-44-8); Soman: *O*-Pinacolyl methylphosphonofluoridate (96-64-0)	
(2) *O*-Alkyl (R_1: $\leq C_{10}$, incl. cycloalkyl) *N*,*N*-dialkyl ($R_{2,3}$: Me, Et, *n*-Pr or *i*-Pr) phosphoramidocyanidates e.g. Tabun: *O*-Ethyl *N*,*N*-dimethylphosphoramidocyanidate (77-81-6)	

(*continued overleaf*)

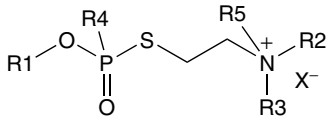
(*continued*)

Schedule 1

(3) *O*-Alkyl (R_1: H or $\leq C_{10}$, incl. cycloalkyl) *S*-2-dialkyl ($R_{2,3}$: Me, Et, *n*-Pr or *i*-Pr)-aminoethyl alkyl (R_4: Me, Et, *n*-Pr or *i*-Pr) phosphonothiolates and corresponding alkylated or protonated salts (R_5: alkyl or H; X: anion)

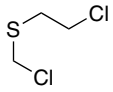


e.g. VX: *O*-Ethyl *S*-2-diisopropylaminoethyl methyl phosphonothiolate (50782-69-9)

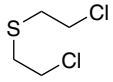


(4) Sulfur mustards:

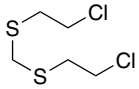
2-Chloroethylchloromethylsulfide (2625-76-5)



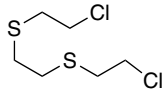
Mustard gas: Bis(2-chloroethyl)sulfide (505-60-2)



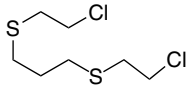
Bis(2-chloroethylthio)methane (63869-13-6)



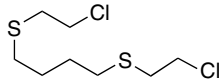
Sesquimustard: 1,2-Bis(2-chloroethylthio)ethane (3563-36-8)



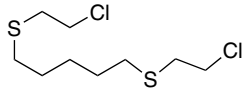
l,3-Bis(2-chloroethylthio)-*n*-propane (63905-10-2)



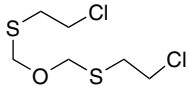
l,4-Bis(2-chloroethylthio)-*n*-butane (142868-93-7)



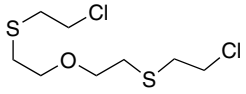
l,5-Bis(2-chloroethylthio)-*n*-pentane (142868-94-8)



Bis(2-chloroethylthiomethyl)ether (63918-90-1)

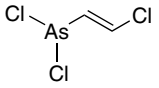


O-Mustard: Bis(2-chloroethylthioethyl)ether (63918-89-8)



(5) Lewisites:

Lewisite l: 2-Chlorovinyldichloroarsine (541-25-3)



Lewisite 2: Bis(2-chlorovinyl)chloroarsine (40334-69-8)