Chemical Weapons Convention Bulletin

Department of Commerce
Bureau of Industry and Security

CHEMICAL WEAPONS CONVENTION: SAMPLING AND ANALYSIS

On February 13, 2006, the Director General of the Organization for the Prohibition of Chemical Weapons' (OPCW) Technical Secretariat announced that an eighteen-month trial period of sampling and analysis (S&A) during Schedule 2 inspections would commence on July 1, 2006. On April 27, 2006, the Bureau of Industry and Security (BIS) issued amendments to the Chemical Weapons Convention (CWC) Regulations (CWCR) (15 CFR 710 et seq.) that include new provisions related to the analysis of samples taken by the OPCW during inspections at U.S. facilities. The CWCR's inspection and analysis procedures are to be used in conjunction with the Department of State's (DOS) regulations on the requirements for the taking of samples (22 CFR 103 et seq.).

This outreach publication provides background and guidance on S&A activities that may take place during an initial or routine CWC inspection at a declared U.S. facility.

Background

The Verification Annex of the CWC specifies S&A as part of inspection activities. The conduct of S&A depends on the regime (i.e., Schedule 1, 2, or 3, or unscheduled discrete organic chemical (UDOC)) being inspected. The terms under which S&A will take place are as follows:

Schedule 1. Although not specifically designated as part of inspection activities, the Verification Annex does not preclude S&A activities.

Schedule 2. "Sampling and analysis **shall** be undertaken to check for the absence of undeclared scheduled chemicals."

Schedule 3 and UDOC. "Sampling and analysis **may** be undertaken to check for the absence of undeclared scheduled chemicals."

While the CWC states that the OPCW shall undertake S&A during Schedule 2 inspections, such activities have only occurred a few times to date at the State Party's request. No sampling has occurred at Schedule 3 or UDOC plant sites to date.

The CWC also stipulates that facility representatives shall take the sample at the request of the Inspection Team (IT) and, where possible, the analysis of samples shall be performed on-site. Off-site analysis will generally be restricted to addressing any unresolved ambiguities (case specific). If off-site analysis is necessary, the CWC requires the sample to be sent to at least two OPCW-designated laboratories for analysis.

DOS's regulation on sample taking states that the taking of the sample must be consistent with the inspection aims and the Confidentiality Annex (see 22 CFR 103.3(d)(1)) of the CWC. The CWCR limit analysis of any sample taken to verifying the absence of undeclared scheduled chemicals, unless otherwise agreed after consultation with the facility representative (see 15 CFR 716.7).

Process for Sampling and Analysis

General. Upon request by the IT to conduct S&A, the BIS Host Team (HT) will inform the facility and assess whether such a request satisfies the conditions listed in §103.3(d) of DOS's regulations. The facility representative may communicate to the Host Team Leader (HTL) any concerns with the taking of the sample.

In making a determination on consistency with DOS's regulations, the HTL will consult with site representative(s) to determine whether the taking of a sample unnecessarily hampers or delays the operation of the facility or affects its safety, and is consistent with safety regulations established at the premises. The taking of a sample will be carried out with the least possible inconvenience and disturbance to the facility. Methods of analysis and the specific scheduled chemicals being analyzed for will also be negotiated between the HT and the IT prior to granting such a request.

Sampling Points. Samples will be taken at processing points specified by the inspected facility in its facility agreement. In the absence of a facility agreement, consideration will be given to existing sampling points used by the inspected facility.

Sample Taking, Preparation, and Splitting. An authorized facility representative will determine whether the sample will be taken by the facility, IT, or HT. When possible, personnel from the inspected facility should take the sample. Such action will take place in the presence of the inspectors and host team.

Any IT or HT activity, as agreed upon by the inspected facility, should be restricted to sampling that does not involve the operation of site equipment (e.g., soil, water or wipe samples).

The IT is responsible for preparing and splitting samples, and all paperwork associated with the transport and handling of the sample and splits. Each sample will have 8 splits that are distributed as follows:

- One retained by inspected State Party (i.e., Host Team);
- One retained in the joint custody of the IT and HT;
- Four reserved for off-site analysis, if needed; and
- Two for use in on-site analysis.

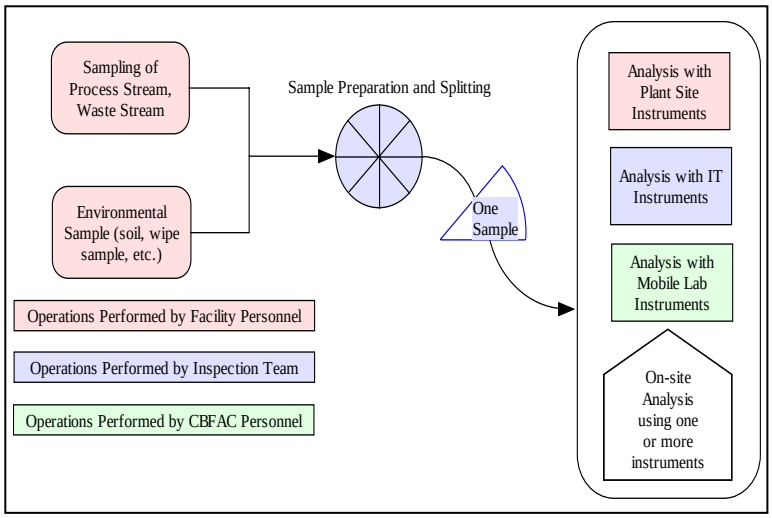
All unused samples or portions of samples will be returned to the inspected facility, if requested.

On-site Analysis. There are three ways in which analysis may be carried-out on-site (Figure 1). The facility, HT, and IT have the right to be present during all on-site analysis activities.

1. Using approved equipment brought by the IT.

The IT has the right to perform on-site analysis of samples using OPCW-approved equipment. The site representative(s) may also check and verify that the equipment meets local safety requirements. The OPCW-approved equipment includes a portable Gas Chromatograph/Mass Spectrometer (GC/MS) that is capable of detecting scheduled chemicals as low as 1 – 10 parts per million (ppm). The equipment is designed for qualitative analysis only (i.e., it cannot determine concentration or quantity, only presence above detection limits).

Figure 1. On-site Analysis



Chemicals to be Used On-Site. The following chemicals (Table 1) are used by the IT to conduct analysis. The chemicals can be brought on-site by the IT, purchased by BIS, or provided by the facility.

Table 1. Chemicals to be used by IT

Chemical	Qty.	Chemical	Qty.
Nitrogen	1 bottle	0.1 M Ammonium hydroxide	50 ml
Helium	1 bottle	Methanol	500 ml
Water	100 ml	Dichloromethane	500 ml
Sodium Sulfate	50 g	Triethylamine	50 ml
Tetrahydrofuran	100 ml	Methanol	500 ml
Hexane	500 ml	Acetonitrile	500 ml
0.1 M Hydrochloric acid	50 ml	N,O-bis-(trimethylsilyl) trifluoroacetamide (BSTFA)	10 ml
2.0 M Hydrochloric acid	50 ml		
*3,4-Dimecaptotoulene, solution 5 mg/mL in acetone	10 ml	*OPCW GC/MS Mixture Hexachlorobenzene/Dichloromethane	2 ml
*OPCW GC/MS test mixture	2x2 ml	*Methanol-Triethylamine (1%, v/v)	200 ml

** Provided by IT*

Note: The OPCW GC/MS test mixture contains: trimethyl phosphate, 2,6-dimethylphenol,5-chloro-2-methylaniline, tributylphosphate, dibenzothiphene, malathion, methylstearate and n-alkanes C8 to C24, even numbers - octane, decane, dodecane, tetradecane, hexadecane, octadecane, eicosane, docosane ,tetracosane - in dichloromethane.

If the facility chooses to provide these chemicals:

(i) Any chemicals provided must be originally sealed, labeled and unopened;
(ii) The purity of all solvents, chemicals and gases must be certified by the producer; and
(iii) The expiration date must fall at least six months after the date on which the chemicals are provided to the IT.

2. Using the inspected facility's equipment.

If the facility has the technical capability, and upon agreement between a company representative, HTL and IT, samples can be analyzed on-site using the equipment of the inspected facility. The facility's equipment may need to be configured to meet IT-established criteria. In such a case, the inspection team may observe this activity.

As a general rule, upon agreement to taking a sample, BIS will propose that the facility conduct an analysis of a sample taken from the same sampling point as soon as possible in the presence of the HT. The results from the analysis will be used to (1) anticipate and address any issues from the analysis conducted by the facility in the presence of the IT or (2) serve as an independent confirmation of the analytical result produced by the IT's equipment.

3. Using a mobile laboratory contracted by BIS.

BIS has established a contract with the Department of the Army's Chemical and Biological Forensic Analytical Center (CBFAC) at Edgewood, MD to provide on-site analysis capabilities with equipment similar to the OPCW's through the use of its mobile laboratory, if necessary. Deployment of the mobile laboratory is a special case scenario that may result in prolonging the inspection. For example, in the absence of IT analytical equipment and in the event the facility does not have the capability to conduct the analysis, the mobile laboratory may be utilized. It may also be used to confirm the analytical result of the IT or resolve an ambiguity.

It should be noted that if IT equipment and/or the CBFAC mobile laboratory is used, if possible, the facility will be requested to provide designated and secure laboratory space with certain capabilities and equipment, including: climate control, power supply, fume-hood, carrier gases, waste handling, and storage necessary for the operation.

Off-site Analysis. Section 304(f)(1) of the CWC Implementation Act prohibits any sample obtained from a U.S. facility from transfer for analysis to any laboratory outside the territory of the United States. Currently, there are two OPCW designated laboratories in the United States: CBFAC and the Lawrence Livermore National Laboratory – Forensic Science Center in Livermore, CA. In general, off-site analysis will be used in the event that on-site analysis produces inconclusive results or an ambiguity cannot be resolved by other means.

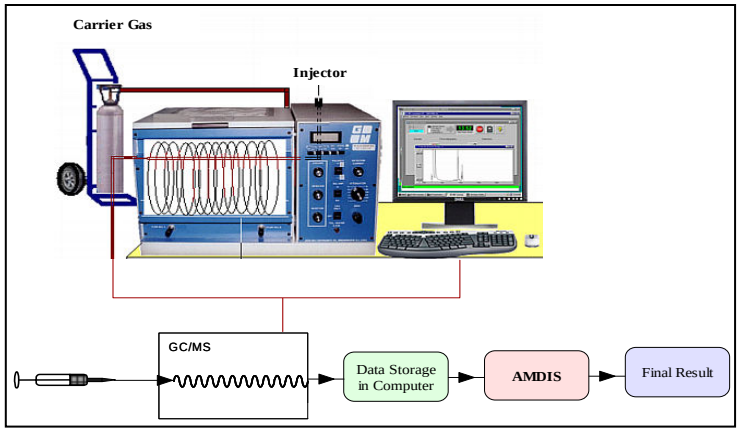
Analysis and AMDIS "Blinding" Software. The normal sequence for GC/MS analysis (Figure 2) of samples used by the IT is presented in the following steps. During S&A, the outcome of each step is evaluated to ensure that the results are satisfactory before proceeding to the next step.

(i) Set-up and start-up of instrument;
(ii) Tuning and mass calibration of the MS;
(iii) Retention index calibration and performance testing by injection of the OPCW GC/MS test mixture;
(iv) Installation of the OPCW database library -- the HT will ensure the following results (v – vii) are consistent with the spectral data in the OPCW database;
(v) Analysis of the solvent blanks;
(vi) Analysis of the prepared authentic blanks and samples; and
(vii) Analysis of the OPCW GC/MS test mixture when finishing the analyses for the day.

As noted above, analysis will be restricted to verifying the absence of (specified) undeclared scheduled chemicals, unless otherwise agreed after consultation with the facility representative.

To help prevent disclosure of information unrelated to the purpose of the analysis, the IT GC/MS employs the Automated Mass Spectral Deconvolution and Identification System (AMDIS) software program to analyze the data. AMDIS was developed by the Department of Commerce's National Institute of Standards and Technology with support from the Department of Defense. It extracts spectra for individual components in a GC/MS data file and identifies target compounds by matching these spectra against a reference library, in this case the OPCW analytical database. A compound is considered identified if the matching factor is greater than 80%. Only the compound being targeted is displayed in the final result.

Figure 2. GC/MS Analysis



If the site equipment is used to perform the analysis, the facility may choose to have AMDIS installed to help protect confidential business information (CBI) and data not relevant to the purpose of the inspection. The link to the software is available online at **www.cwc.gov**. This link also provides a complete overview and explanation of the program.

Waste Handling. In general, any waste produced from S&A activities should be disposed of on-site. Any waste generated by the use of the facility's equipment will be processed on-site. However, if IT equipment is used and the waste cannot be disposed of on-site, the HT will handle off-site waste disposal with the assistance of CBFAC. In such case, the facility should provide information regarding the safety and characterization of the samples necessary for proper labeling and shipment, as required by the Department of Transportation.

Advance Preparation for Sampling and Analysis

BIS will, upon request by a facility prior to or after notification, deploy an Advance Team (AT) to assist the site in preparing for the upcoming inspection. The AT will coordinate with the appropriate facility personnel (e.g., lab manager, chemist, health, safety and environmental engineer) to carry-out the specific steps in conducting S&A activities. These may include, but are not limited to, the following:

- Assessing the capability of the facility's equipment and analytical capability;
- Setting-up the facility's GC/MS using IT-approved parameters;
- Installing, reviewing, and testing AMDIS (blinding) software and OPCW Central Analytical Database on the facility's GC/MS, which includes comparing analysis of the current sample or past analysis results against the OPCW database;
- Identifying any site safety equipment to be used to carry-out S&A activities;
- Identifying possible sampling point(s), sampling container(s), sealing and packing material, and methods of transporting samples on-site;
- Reviewing procedures involving storage, splitting of samples, chain of custody, handling of waste, decontamination and associated documentation;
- If using CBFAC mobile laboratory support or IT equipment, setting-up work space with designated requirements; and
- Reviewing sample handling and transportation requirements for off-site analysis, if necessary.

Conclusion

BIS is committed to ensuring that S&A activities are performed in a manner that minimize safety hazards and financial or other burdens, and maximize the protection of CBI. BIS recognizes that S&A is an intrusive procedure and will negotiate with the OPCW on its appropriate use. When possible, the HT will suggest alternative means to satisfy inspection aims. Any preparatory steps taken by the facility will assist in expediting the S&A process in a more efficient manner.

Further Information

To learn more about the CWCR and CWC inspection activities, or to request assistance, including a Site Assistance Visit, please visit our website at **www.cwc.gov** or contact BIS's Treaty Compliance Division at (703) 605-4400 or fax (703) 605-4424.