

# Table Top Exercise, Poland 2010

## OPCW on-site and off-site analysis of samples

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## Introduction:

- The OPCW Laboratory is part of the Verification Division and the focal point for all issues related to Sampling and Analysis (S&A)
- S&A is one of the inspection tools available to the inspection team (IT)
- In accordance with the CWC samples can be analysed **on site** by the inspection team, or, transferred **off site** to laboratories that have been certified for such analysis by the Director-General
- The purpose of such analysis is to provide **factual evidence** for the **presence** of scheduled chemicals and/or support a conclusion of **absence** of scheduled chemicals
- Following is a description of the processes that have been developed for the conduct of such on-site and off-site analysis

## On site analysis:

Sample collection and splitting

Sample preparation for GC/MS analysis

GC/MS analysis and identification of chemicals

## Off site analysis:

**On site:**  
Packing of samples and transport to OPCW Laboratory

**At OPCW Laboratory:**  
Unpacking and checking of samples; repackaging with control samples

**At Designated Laboratory:**  
Off site analysis and reporting of results to Director-General

**At Technical Secretariat:**  
Assessment of validity of results; Integration of results into inspection report

## Key Objectives for OPCW Sampling and Analysis:

- **Sample Authenticity:** Place and time of collection, splitting, sealing, chain of custody...
- **Validity of Sample treatment:** validation of sample preparation procedures, detailed documentation, no cross contamination ...
- **Correctness of analysis results:** validation of method, instrument performance, no cross contamination, **NO FALSE POSITIVES**

## On-site analysis:

Sample collection and splitting

Sample preparation for GC/MS analysis

GC/MS analysis and identification of chemicals

- **Detailed WI** for collection and splitting of different types of sample matrices (liquid/water, soil, wipe, neat/ bulk, any type of solid...)
- **Certified clean sample collection equipment** for one-time use only
- **Splitting and sealing** into 8 sample aliquots;
  - 2 for on-site analysis
  - 1 Inspected State Party
  - 5 for off-site analysis
- **Information collected** in one document 'ON-SITE ANALYSIS BOOKLET'

## Sample collection

- Detection devices:  
AP2C, CAM-2, RAID-M, CALID3 etc.
- OPCW Sample Collection Kit
- Standard IPE, decontamination equipment  
etc.





**OPCW sample collection**



**Sample collection equipment (sealed)**

## On-site analysis:

Sample collection and  
splitting

Sample preparation for  
GC/MS analysis

GC/MS analysis and  
identification of  
chemicals

- **Detailed WI** for sample preparation **using validated procedures** for different types of sample matrices and for different groups of target chemicals
- **Certified clean sample preparation equipment** for one-time use only and **certified chemicals**
- **Preparation of blanks**
- **All steps documented in detail** in ‘ON-SITE ANALYSIS BOOKLET’
- **No target chemicals brought by the IT** (prevent contamination by IT); But no spiking for QC possible



## Equipment to conduct sample preparation:



Dangerous Goods  
(no Scheduled  
chemicals)



Sample preparation kit (part of)

# On-site analysis laboratory (IAU Exercise)







## On-site analysis:

Sample collection and splitting

Sample preparation for GC/MS analysis

GC/MS analysis and identification of chemicals

- **Instrument testing and certification** (accredited) before dispatch: performance, cleanliness, completeness
- Before dispatch, **testing and certification of OCAD reference library** (accredited) to be used during on-site analysis
- **Detailed WI** for instrument operation, method parameters, sample analysis and quality control requirements
- **Regular analysis of OPCW test mixture** checking GC and MS performance and calibration of GCRI
- **Co-injection of internal standard of quality (HCB)** for each analysis
- **Analysis of solvent blanks, sample preparation blanks** before any sample analysis
- **Detailed documentation** in 'ON-SITE ANALYSIS BOOKLET' and instrument logbook
- **Criteria for positive identification:** MS library match + GCRI match

**OPCW GC/MS equipment**  
**for on-site analysis:**



**Agilent 6850 GC/ 5973 or 5975 MSD**

OPCW GC/MS equipment  
for on-site analysis:

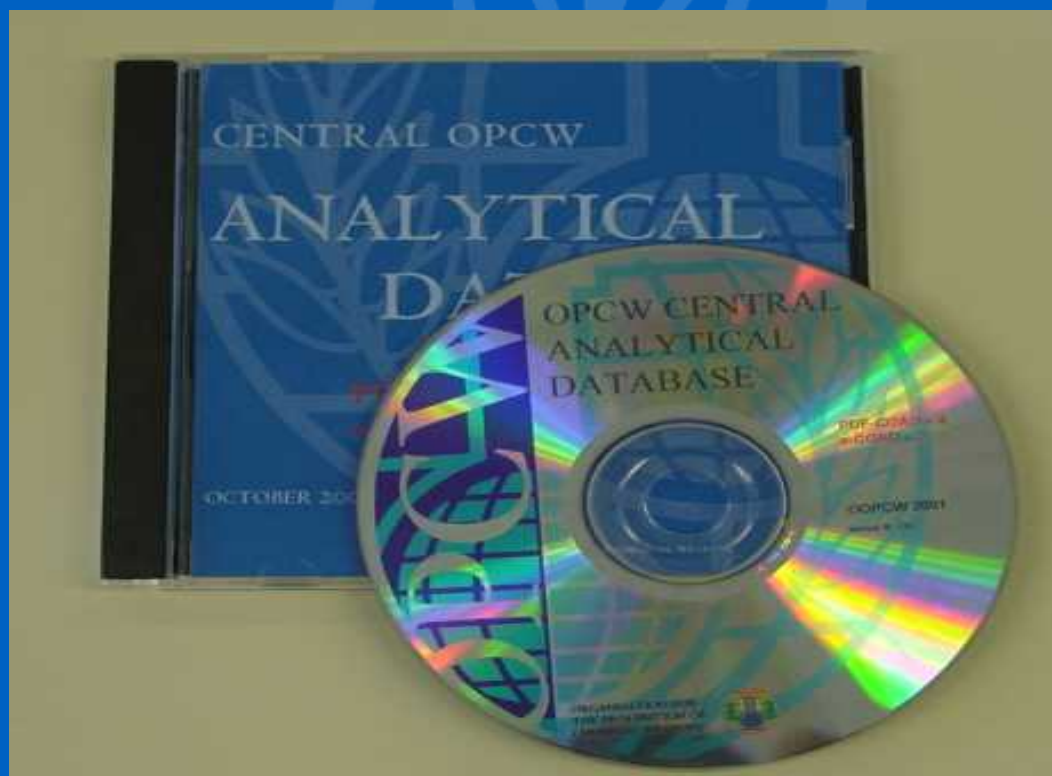


**MOBILE IR**



**Analytical reference library**  
**for on-site analysis:**

MS spectra:	4183 chemicals
GCRI data:	3494 chemicals
FTIR spectra:	1391 chemicals
NMR spectra:	936 chemicals



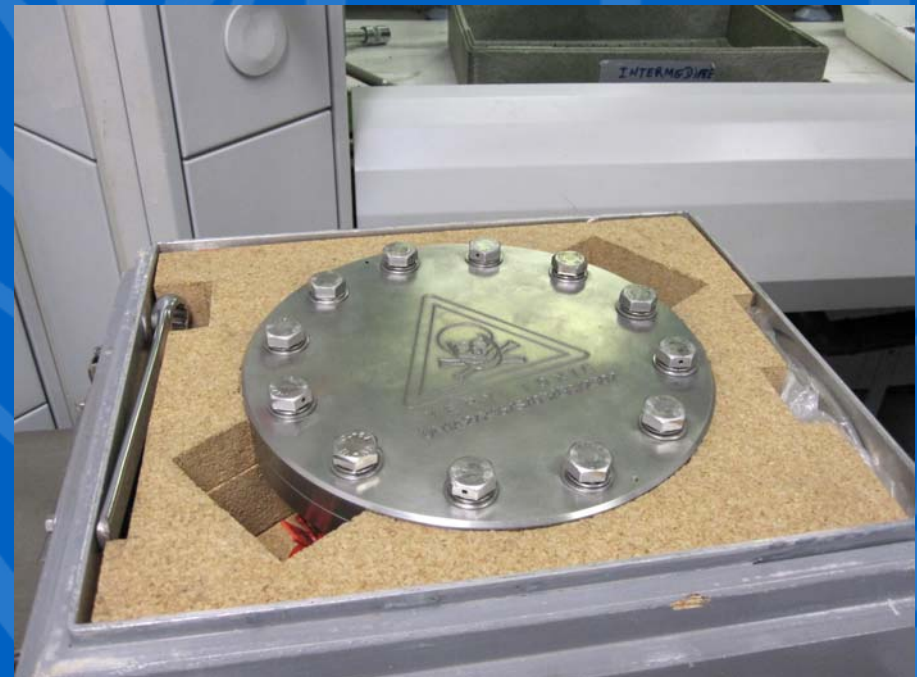
**OPCW Central Analytical Database**

## Quality Management System for off-site analysis:

Before a laboratory can be designated for off-site analysis:

- It must obtain **accreditation** under ISO 17025 for the analysis of chemical weapons related chemicals
- It must participate (at least) once per year in **OPCW Proficiency Testing**
- It must demonstrate **continuous excellent performance** reflected in a proficiency test scoring sequence of its last three most recent scores: either 3x A; or 2x A and 1x B

# Custody during transport



## Custody during transport





## Off-site analysis:

### On site:

Packing of samples and transport to OPCW Laboratory

### At OPCW Laboratory:

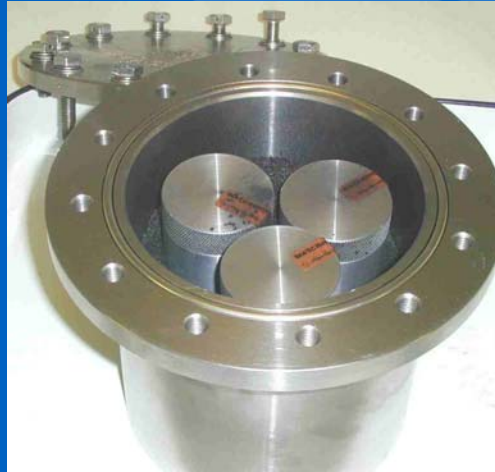
Unpacking and checking of samples; repackaging with control samples

### At Designated Laboratory:

Off site analysis and reporting of results to Director-General

### At Technical Secretariat:

Assessment of validity of results; Integration of results into inspection report



**Integrity of samples during transport controlled through seals and checking of weight**



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- Upon arrival **verify identity** of sample vials through seal numbers, sample weights
- Re-packaging with:
  - (a) one authentic sample from on-site
  - (b) **one control sample containing Scheduled chemicals**
  - (c) **one blank sample containing no Scheduled chemicals**
- Detailed **analysis of control and blank** samples before dispatch (homogeneity, impurities,...)
- Monitoring of **stability** after dispatch (ISO Guide 35)



## Off-site analysis:

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- Upon arrival **verify identity** of sample vial through seal numbers, sample weights
- All vials (**authentic sample and controls**) are analysed in accordance with instructions issued by OPCW Director-General
- The results are provided within a maximum of 14 days in the form of a stand alone report to the OPCW Director-General
- All **results must be supported by comprehensive analytical data providing proof for any identifications made with two different analytical techniques** (in accordance with the technical requirements for reporting in OPCW proficiency testing)
- All sample material must be **accounted** for and all waste must be collected

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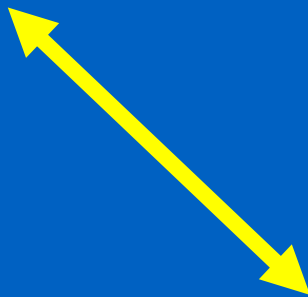
- Designated Laboratory results are **assessed** by OPCW for:
  - (1) Correct analysis of control sample and blank
  - (2) Compliance with relevant reporting criteria
  - (3) Consistency between Designated Laboratories
- Results become part of inspection report

## Reporting criteria for off-site analysis results:

- The identification of each test chemical must be based on at least two different analytical techniques giving consistent results; at least one of these techniques must be a spectrometric technique (e.g. GC/MS-EI, GC/MS-CI, LC/MS, NMR).
- The laboratory must indicate on which basis the test chemicals have been identified:
  - (a) By a comparison with data obtained from reference chemicals;
  - (b) By a comparison with reference data available in analytical databases; or
  - (c) By interpretation of spectra with comparison to closely related reference data that may have been obtained from a library or from analysis of a closely related chemical (applicable to mass spectrometric and NMR spectrometric techniques)
- The laboratory must describe in detail the sample preparation procedures and analytical methods it used.
- There must be an unbroken chain of evidence linking each test sample to each reported chemical.

## Summary:

- The methods and procedures for analysis have shown to allow detection and identification of scheduled chemicals and their degradation products at ppm level in a range of different sample matrices;
- Off site analysis requires the movement of samples and analysis results; this has significant implications for logistics;
- During off site analysis a number of different analysis techniques and methods will be employed by designated laboratories; 18 laboratories from 3 regions are currently designated;
- Off site analysis is technically superior to on site analysis and is particularly recommendable in cases where on site analysis has identified an unexpected scheduled chemical



**QUESTIONS ?**

