# **Technical Secretariat**



Verification Division S/356/2003 24 March 2003 Original: ENGLISH

## **REPORT BY THE DIRECTOR-GENERAL**

### **OFF-SITE SAMPLE-HANDLING EXERCISE**

- 1. From 23 January to 21 February 2003, the Technical Secretariat (hereinafter "the Secretariat") conducted an exercise to test procedures for the transport and handling of samples sent for off-site analysis. The generous financial contribution of the United Kingdom of Great Britain and Northern Ireland made the exercise possible. The positive response by States Parties in providing assistance to, and participating in, the exercise encouraged the Secretariat to extend the number of laboratories to four from the three originally budgeted for.
- 2. The following laboratories took part in the exercise:
  - (a) Verification Laboratory, Centre for Chemical Defence, DSO National Laboratories, Singapore (CCD), for the preparation of the samples;
  - (b) Protechnik Laboratories (Pty) Ltd, South Africa (Protechnik), for the analysis of the samples;
  - (c) Defence Science and Technology Laboratory, the United Kingdom (Dstl), also for the analysis the samples; and
  - (d) Lawrence Livermore National Laboratory, the United States of America (LLNL), for the analysis of the samples.

#### **Exercise objectives**

- 3. The objectives of the exercise were to:
  - (a) test the procedures developed in order to comply with paragraph 56 of Part II of the Verification Annex to the Chemical Weapons Convention (hereinafter "the Verification Annex");
  - (b) test in particular the Secretariat's standard operating procedure for off-site sample analysis and related work instructions, both of which are based on the facilitator's draft on the ongoing informal consultations in relation to paragraph 56 of Part II of the Verification Annex;

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- (c) demonstrate that OPCW samples can be transferred in the following ways:
  - i. by air, through the invocation by the Director-General of special provision A106 of the International Air Transport Association (IATA) regulations;
  - ii. by road, through the invocation by the Director-General of special provision 250 of the Accord européen relatif au transport international des marchandises <u>d</u>angereuses par <u>r</u>oute (ADR, English translation, European Agreement for Transport by Road); and
  - iii. by sea, through the invocation by the Director-General of the International Convention for the Safety of Life at Sea (SOLAS) and the International Maritime Dangerous Goods Code (Amendment 30) under special provision 250.
- 4. Testing the transport by air of off-site samples to and from the OPCW Laboratory to the participating laboratories in accordance with international transport regulations was considered crucial. Air regulations allow airlines, and each pilot, to refuse to transport items such as OPCW samples, even if special provision A106 of the IATA regulations has been invoked.

# Movement of samples to the OPCW Laboratory and from the OPCW Laboratory to analysing laboratories

- 5. One of the major aims of the exercise was to assess the feasibility of transporting off-site samples via a combination of air, road, and sea. The Secretariat coordinated the transport of the sample, equipment, and personnel with the assistance of its subcontracted firm, Dangerous Goods Management (DGM).
- 6. A detailed chronology of events can be found in Annex 1 to this report.
- 7. First, the exercise team leader travelled to Singapore. There, the sample that had been prepared at the CCD was collected. A request to send the sample off-site was transmitted to the Director-General, who approved it. The authentic<sup>1</sup> sample was split, packed into an approved sample transport container, and transported off-site.
- 8. The container was moved from CCD to the international airport by car, and then flown out of Singapore on a cargo plane. Because of the unavailability of carriers flying to the Netherlands, it was flown from Singapore to Luxembourg. The same carrier, based in Luxembourg, handled the shipment by air of all sample transfers. The National Authority of Luxembourg was requested by the Secretariat to assist in clearing the samples through customs. Thanks to their efforts, the customs process could be expedited—a fact that serves to highlight the importance of close cooperation with States Parties in transporting these types of sample.

An authentic sample is collected as evidence during an OPCW inspection. It may be a liquid, solid, or gaseous substance, or an on-site extract of such a substance that will be subjected to an analysis of its components in order to meet the purpose of sampling and analysis as reflected in the relevant parts of the CWC. In this report the authentic sample is an organic liquid.

- 9. The container was transported from Luxembourg to the OPCW by road, escorted by a staff member of the Secretariat. On arrival, it was handed over to the Head of the OPCW Laboratory in accordance with the relevant procedures.
- 10. Once the repacking of samples with the control sample and the matrix blank was completed at the OPCW Laboratory (paragraphs 15 to 19 below), three containers were ready to be escorted to the three analysing laboratories, that had been selected and which had been notified by the Director-General that the samples would soon be delivered.
- 11. The first analysing laboratory to receive the samples was Dstl. A Secretariat staff member escorted the container to the port of Rotterdam and thence on a cargo ferry to Harwich in the United Kingdom. After an unanticipated overnight stay in Harwich because of bad weather, the container—still with its Secretariat escort—was taken to Dstl by a dangerous-goods transport operator contracted by the UK National Authority.
- 12. The second laboratory to receive the samples for analysis was Protechnik in South Africa. The container was transported by car to Luxembourg and flown on a cargo plane to Johannesburg, from where it was transported to Protechnik by car.
- 13. The third analysing laboratory to receive the samples was LLNL in the United States. The container was transported by car to Luxembourg, and then by cargo plane to Los Angeles, and, finally, by car to LLNL. The flight from Luxembourg was delayed for three days because an airport clearance did not reach the carrier in time. This carrier operates only two flights per week to Los Angeles, and that also contributed to the delay in the shipment. The escort and equipment travelling to the United States was requested to stop over in Washington for point of entry (POE) procedures before travelling to LLNL.
- 14. The Secretariat and the dangerous-goods contractor went to great lengths to coordinate the transport of the samples by air, and to find air carriers willing to carry the samples on very short notice. In some cases additional complications arose in obtaining airport clearances for receipt of the samples and receiving customs clearance for their release.

#### Repacking of samples at the OPCW Laboratory

15. The OPCW Laboratory was notified on 24 January 2003 of the preparations to send samples for off-site analysis. Secure communication was established with the exercise team leader on-site in order to allow the exchange of information on the type of sample matrix and on the appearance of the authentic samples, in order in turn to

enable preparation of control samples  $^{2}$  and matrix  $\mathsf{blanks}^{3}$  to begin at the OPCW Laboratory.

- 16. While the sample transport container was in transit from Singapore to the Secretariat, control samples and matrix blanks were prepared and analysed through the use of three techniques (gas chromatography with an element-selective detector, gas chromatography/mass spectrometry, and liquid chromatography/mass spectrometry).
- 17. The sample transport container arrived at the OPCW Laboratory after midnight and was stored in the secure sample archive of the laboratory. The following morning, five authentic sample vials were unpacked, and seals and sample weights were checked. Because only three authentic sample vials were required for repacking, the remaining two were stored in the secure sample archive at the OPCW Laboratory.
- 18. Control samples and matrix blanks had been prepared in such a way that they would be indistinguishable from the authentic sample; one vial of each was packed together into one sample transport container. Three of these containers were packed in this way, one for each analysing laboratory. The three sample transport containers were picked up from the OPCW Laboratory the following day.
- 19. Unpacking and repacking were observed by the facilitator on the informal consultations on the "Procedures for Inclusion in the Inspection Manual Concerning the Security, Integrity and Preservation of Samples and for Ensuring the Protection of the Confidentiality of Samples Transferred for off-Site Analysis". One Confidentiality Officer from the Office of Confidentiality and Security was present at the OPCW Laboratory during the unpacking and repacking of samples.
- 20. A control sample and a matrix blank were re-analysed as described in paragraph 16 five days after the containers had been dispatched.

#### **Reporting of results from analysing laboratories**

- 21. The three analysing laboratories submitted their analysis reports to the Director-General in the requested format within the allotted timeframe of fourteen days from receipt of the samples. The laboratories had been provided with analysis instructions from the Director-General and a template for reporting their results, which was based on the format for reporting the results of OPCW proficiency tests.
- 22. The OPCW Laboratory conducted a technical evaluation and compared the results for control samples, matrix blanks, and authentic samples from all three laboratories. It reviewed the methodologies used for the analysis of the samples, performed a technical evaluation of the analysis results based on the criteria for OPCW proficiency testing, and summarised its findings in a report.

<sup>&</sup>lt;sup>2</sup> Control samples are used to assess the performance of the equipment, the methodology, and the personnel at the designated laboratory. To make a control sample, scheduled chemicals or their degradation products are spiked at a known concentration into a characterised matrix; preferably a matrix of a composition similar to that of the authentic samples.

<sup>&</sup>lt;sup>3</sup> A matrix blank consists of a characterised matrix, preferably a matrix of a composition similar to that of the authentic samples. This blank is analysed in advance for the absence of scheduled chemicals. A matrix blank is used to assess the performance of equipment, methodology and personnel at the designated laboratory.

23. All three laboratories had correctly identified the chemical added to the control sample, and none had reported any chemicals for the matrix blank. The results for the authentic sample were consistent: All laboratories reported the same scheduled chemical. One laboratory reported one additional scheduled chemical in the authentic sample, which was detected after significantly concentrating the sample. For the purpose of the exercise the reportable concentrations were set at a level similar to those for OPCW proficiency testing, with a lower limit of 1 to 10 ppm.

#### **Confidentiality and security**

- 24. Paragraph 1.15 of Part V of the OPCW Policy on Confidentiality states that, "unless specified otherwise in accordance with lesser sensitivity...samples taken from inspected sites and returned samples from designated laboratories, and results from analysis of samples might be classified as OPCW HIGHLY PROTECTED when they are acquired or generated by any means by the Organisation". In order to facilitate the dissemination of results during this exercise, the classification applied to materials was "UNCLASSIFIED" (marked as "OPCW Exercise"); however, the procedures for handling and protecting confidential information were followed as closely as possible, in order to reflect real sampling situations. In this respect, the off-site sampling exercise proved invaluable for identifying key security and confidentiality matters.
- 25. Only laboratory-related activities such as the receipt of authentic samples, storage, unpacking, and repacking with control samples and matrix blanks were carried out at the OPCW Laboratory during the exercise. All activities related to the technical evaluation of analysis results from the analysing laboratories were carried out in the Secure Critical Area at OPCW headquarters, and the report of the results was drafted on the Secure Critical Network. In general, the confidentiality and security measures taken for the exercise were satisfactory. However, the security procedures at the Rijswijk facility are currently under review.
- 26. The exercise highlighted some deficiencies in physical security at the Rijswijk facility. A general review of security there had been started prior to the exercise, in January 2003. While this review has not yet been completed, it has become apparent that, in a real sampling situation, security guards would need to be assigned to the Rijswijk facility to maintain a 24-hour-a-day presence until authentic samples had been destroyed. This would provide the necessary level of security whenever the OPCW Laboratory was storing authentic samples.
- 27 Regarding external participants in the exercise, certain measures were necessary in order to bring the analysing laboratories and couriers into compliance with the OPCW confidentiality regime. For example, it will be necessary to have designated laboratories sign secrecy agreements with the OPCW. The procedure for transmitting confidential documents from the designated laboratories to the Secretariat also requires further delineation. Because samples are packed in containers with tamper-evident seals, and because an OPCW escort will take custody of all documents related to the samples, couriers will not have to be cleared for access to the sample container. The container with sample materials is always protected with fibre-optical seals when in transit. Nevertheless, during the exercise, the DGM firm that had been contracted by the Secretariat was requested to sign the standard secrecy agreement used for all OPCW consultants.

#### Lessons learned

- 28. The successful conduct of the exercise demonstrated that the Secretariat is ready and able to carry out the kinds of demanding task the exercise required. However, it should be emphasised that these tasks and procedures are complex and depend to a large extent on smooth and effective cooperation and communication among the Secretariat, States Parties, their National Authorities, and the commercial air companies involved.
- 29. The National Authorities of States Parties involved can greatly contribute to the timely transport of samples, because of their knowledge of national regulations and their relationship to the local customs offices.
- 30. Air transportation with several stopovers can complicate matters, because the regulations of the relevant State Party have to be applied at each stopover.
- 31. Analysis reports, which will generally be classified, "OPCW Highly Protected", should be hand delivered as soon as possible to the Director-General, either by a representative of the designated laboratory or by a representative from the Secretariat.
- 32. It should be noted that security upgrades to the Rijswijk facility are under evaluation.
- 33. Secrecy agreements for designated laboratories need to be concluded.
- 34. Designated laboratories and their National Authorities play an important role not only in the analysis of samples but also in transporting them. Each designated laboratory should notify the Secretariat of the point of entry for samples from which it will take over the transport of samples to its laboratory, and any other transport requirements that may be relevant. Designated laboratories may be required to develop their own procedures in order to implement existing provisions in the facilitator's draft and the Secretariat SOP.
- 35. The conduct of OPCW proficiency tests should be adjusted to reflect the sample setup for actual off-site analysis. Therefore, samples for proficiency testing should contain in future none-indicated vials representing authentic samples, control samples, and matrix blanks
- 36. The costs of the exercise were kept to a minimum, but time constraints and limits on the availability of flights might increase them substantially. Also, during this exercise the DGM was used extensively to transport the samples by road; but in future, it would be cheaper to have the escort transport them alone in an OPCW vehicle.
- 37. The Secretariat encourages the States Parties involved to give the benefit of their views and their experiences with the exercise.

38. The Secretariat would like to express its gratitude to all States Parties for their offers of assistance and cooperation. It would also like to thank the laboratories involved and the respective National Authorities for their outstanding contributions to the exercise. Finally, it would like to extend its thanks to the National Authority of Luxembourg for its tireless efforts in supporting this exercise.

Annex: Chronology of Events

#### Annex

# CHRONOLOGY OF EVENTS

## **Exercise activities at CCD in Singapore**

Date	Time (The Hague)	Activity	Comments
23-01-2003		Exercise team leader (ETL) departs for Singapore.	Start of exercise
24-01-2003		ETL arrives in Singapore.	
	08:45	ETL faxes request to Director-General to approve the dispatch of samples for off-site analysis.	Director-General approves request and invokes IATA provision A106.
	10:00	ETL transmits information about sample appearance and matrix to Head of OPCW Laboratory.	Via Secure phone communication through Operation Centre
25-01-2003		Authentic samples are split, sealed, and packed into a transport container.	Performed by ETL at CCD
28-01-2003		Container is transported to Singapore Airport.	
29-01-2003		Container leaves Singapore.	
29-01-2003	15:00	Container arrives in Luxembourg.	
30-01-2003	00:30	Container arrives at the OPCW Laboratory.	

## Exercise activities at the OPCW Laboratory

Date	Time (The Hague)	Activity	Comments
24-01-03	14:15	Initial laboratory meeting to select control sample spiking chemical	
27-01-03	12:00	Security upgrade of OPCW Laboratory completed	Access restrictions, tamper-evident seals on windows and emergency-exit doors, video recording with CCTV cameras, two-person access to sample archive
	14:00	Preparation of control sample/matrix blank	
	17:00	Analysis of control sample/matrix blank completed	Vials stored in secure sample archive
30-01-03	00:30	Sample container arrives at OPCW Laboratory.	Stored in sample archive over night
	08:30	Unpacking of sample container started	
	11:00	Re-packing of three containers completed	Containers and remaining authentic sample vials stored in sample archive
31-01-03		Pick-up of all three sample containers by DGM	
04-02-03		Re-analysis of control sample/matrix blank	

## Transport of Samples to Dstl, the United Kingdom

Date	Time (The Hague)	Activity	Comments
31-01-03	08:15	Container departs from OPCW Laboratory.	By car to Rotterdam and by ferry to Harwich
31-01-03	19:00	Container and OPCW escort arrive in Harwich.	Overnight stay because of bad weather
01-02-03		Transfer to Dstl	
	14:30	Unpacking and hand-over of samples at Dstl	
19-02-03		Analysis report delivered to Director-General	

# Transport of Samples to Protechnik, South Africa

Date	Time (The Hague)	Activity	Comments
31-01-03	16:00	Container departs from OPCW Laboratory.	Stored at DGM headquarters
02-02-03	05:00	Container leaves DGM headquarters for Luxembourg Airport.	
03-02-03		Container leaves Luxembourg for South Africa.	By cargo plane
		Container arrives in Johannesburg.	
		Container is transferred by car to Protechnik.	
04-02-03		Unpacking and hand-over of samples	
21-02-03		Analysis report delivered to Director-General	

# Transport of Samples to LLNL, the United States

Date	Time (The Hague)	Activity	Comments
31-01-03	16:00	Container departs from OPCW Laboratory.	Stored at DGM headquarters
02-02-03	05:00	Container leaves DGM headquarters for Luxembourg Airport.	
06-02-03		Container leaves Luxembourg for Los Angeles.	By cargo plane
		Container arrives in Los Angeles.	
		Container is transferred by car to LLNL.	
07-02-03	09:40	Unpacking and hand-over of samples	
18-02-03		Analysis report delivered to Director-General	