OPCW

Scientific Advisory Board

Twelfth Session
24 – 26 November 2008

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REPORT OF THE TWELFTH SESSION OF THE SCIENTIFIC ADVISORY BOARD

1. AGENDA ITEM ONE – Opening of the session

The Scientific Advisory Board (SAB) met for its Twelfth Session from 24 to 26 November 2008 at the OPCW headquarters in The Hague, the Netherlands. The session was opened by the Chairperson of the SAB, Philip Coleman of South Africa. Mahdi Balali-Mood of the Islamic Republic of Iran served as Vice-Chairperson. A list of participants appears as Annex 1 to this report.

2. AGENDA ITEM TWO – Adoption of the agenda

The SAB adopted the following agenda for its Twelfth Session:

1. Opening of the session
2. Adoption of the agenda
3. Welcome address by the Director-General, Ambassador Rogelio Pfirter
4. Overview of developments at the OPCW since the last SAB session
5. Presentation of the report of the third meeting of the temporary working group (TWG) on sampling and analysis:
   (a) Discussion of the report and its recommendations; and
   (b) SAB recommendation on sampling and analysis.
6. Education and outreach in relation to the Convention
7. Introduction to discussion on advances in science and technology and their potential impact on the implementation of the Convention:
   (a) Presentation on the role of microreactor technology in chemical-process intensification;

(b) Discussion; and

(c) SAB recommendation on microreactor technology in chemical-process intensification and its impact on the implementation of the Convention.

8. Initial discussion on the review of operational requirements and technical specifications for inspection equipment

9. Future work of the SAB:

(a) Meeting with governmental experts;

(b) Interaction between the SAB and States Parties, and between the SAB and the policy-making organs; and

(c) Agenda for the Thirteenth Session of the SAB.

10. Any other business:

(a) Presentation on a proposal for a seminar on other chemical production facilities (OCPFs);

(b) SAB Port@l; and

(c) Continuity of the work of the SAB.

11. Adoption of the report

12. Closure of the session

3. AGENDA ITEM THREE – Welcome address by the Director-General, Ambassador Rogelio Pfirter

3.1 The Director-General recalled that the Second Special Session of the Conference of the States Parties to Review the Operation of the Chemical Weapons Convention (hereinafter "the Second Review Conference") underlined the importance for the OPCW to keep abreast of the developments in science and technology in order to achieve the object and purpose of the Convention (RC-2/4, dated 18 April 2008). It recognised that the SAB continues to play a valuable role in enabling the Director-General to render specialised advice to the policy-making organs and the States Parties in areas of science and technology relevant to the Convention. It also noted the impact of scientific and technological progress on the effective implementation of the Convention and the importance for the OPCW and its policy-making organs of taking due account of such developments. In that context, it stressed that the SAB should continue to play an objective and balanced role in advising the Director-General.

3.2 The Director-General commended the work done by the SAB in preparation for the Second Review Conference. He indicated that the report of the SAB to the Second Review Conference, entitled "Report of the Scientific Advisory Board on

Developments in Science and Technology" (RC-2/DG.1, dated 28 February 2008 and Corr.1, dated 5 March 2008), will be considered during a meeting with governmental experts in 2009. He also expressed the hope that the SAB will contribute efficiently to this meeting under the leadership of its Chairperson and assist the Executive Council (hereinafter "the Council") in making sound recommendations based on the SAB report.

3.3 The Director-General welcomed the consideration by the SAB of the implications of microreactor technology in chemical-process intensification and encouraged the SAB to continue working on other subjects related to advances in science and technology and their potential impact on the implementation of the Convention. He also welcomed the work related to the initial review of the inspection equipment as a follow-up to the Second Review Conference.

3.4 The Director-General expressed his gratitude to the 10 States Parties that have so far contributed to the SAB trust fund. These contributions have enabled the SAB to maintain a high frequency of sessions. This higher frequency is important for the SAB to continue working on the various topics related to advances in science and technology and their potential impact on the implementation of the Convention.

3.5 The Director-General recalled that the Second Review Conference had encouraged States Parties to continue to make voluntary contributions to fund the work of the SAB. He also expressed his hope that the SAB members will promote their work among their respective National Authorities, as some members have already successfully done.

4. AGENDA ITEM FOUR – Overview of developments at the OPCW since the last SAB session

4.1 The Secretary gave a presentation to the SAB on developments at the OPCW since the SAB's Eleventh Session (from 11 to 13 February 2008).

4.2 The members of the SAB were briefed on the status of the destruction of Category 1 chemical weapons as of 31 October 2008, and on the latest situation regarding old and abandoned chemical weapons.

4.3 As regards universality, and as of 24 November 2008, there were 184 States Parties to the Convention and efforts to achieve universality were and are ongoing. The SAB members were also briefed on a summary of the activities of the International Cooperation Branch, as well as on the outcome of the Second Review Conference.

4.4 The SAB was informed that the Second Review Conference requested the Council to consider, through a meeting with governmental experts, the suggestions and recommendations made by the SAB in its report to the Second Review Conference (RC-2/DG.1 and Corr.1) and to prepare a report on this subject[1].

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[1] "The Second Review Conference requested the Council, through a meeting of governmental experts open to all States Parties, to consider the report by the Scientific Advisory Board which the Director-General had forwarded to the Second Review Conference." (Paragraph 9.133 of RC-2/4).

5. AGENDA ITEM FIVE – Presentation of the report of the third meeting of the temporary working group on sampling and analysis

Subitem 5(a): Discussion of the report and its recommendations

5.1 The SAB received the third report of the TWG on sampling and analysis (Annex 2) presented by Robin Black, Chairperson of the TWG. The key findings and conclusions of the report are outlined below.

5.2 The TWG reaffirmed its previous recommendation that additional chemicals (non-scheduled degradation products and their derivatives, riot control agents, and chemicals from old and abandoned chemical weapons), as listed in the appendices to the two previous reports of the TWG, should be included in the OPCW Central Analytical Database (OCAD), with the selective use of data from the OCAD in accordance with the aims of inspection.

5.3 The TWG agreed that a reduction of analysis time (sample preparation and instrumental-analysis time) during on-site inspections should be a high priority for the Technical Secretariat (hereinafter "the Secretariat"). The following recommendations were made:

(a) The TWG recognised the difficulty of substantially reducing gas chromatography-mass spectrometry (GC-MS) run times by using fast GC, whilst minimising the variability in retention indices. It recommended that further study should be undertaken to assess the effects on retention indices with respect to on-site analysis.

(b) The predominant view of the TWG was that solid-phase microextraction (SPME) is not sufficiently robust with regard to 'dirty' matrices to be a complete substitute for current on-site sample preparation procedures. The TWG did acknowledge that SPME can provide a rapid and sensitive screening procedure.

(c) The TWG agreed that a reduction in on-site sample preparation time for polar analytes in aqueous samples should be a high priority for the Secretariat. Hollow-fibre-liquid phase microextraction in combination with in-fibre silylation was regarded as the most promising technique. The TWG encouraged collaboration between laboratories in this area.

5.4 The TWG members expressed different views on the importance of trace analysis of environmental-type samples in the context of investigations of alleged use. As a way forward, a correspondence group has been established within the TWG and will recommend criteria for a positive identification of trace components to the next meeting of the TWG.

5.5 The TWG noted and supported the intention of the OPCW Laboratory to hold a confidence-building exercise on biomedical sample analysis in 2009. This was a recommendation of the TWG on biomedical sampling and analysis.

5.6 The TWG evaluated the results of questionnaires returned by eight laboratories on methods that are preferred for toxin identification. A correspondence group, established at the second meeting of the TWG, will submit recommendations for identification requirements for toxins to the next meeting of the TWG.

Subitem 5(b): SAB recommendation on sampling and analysis

5.7 The SAB reviewed and endorsed the recommendations of the TWG on sampling and analysis. The SAB recommends that the study mentioned by the TWG under 5.3(a) include the views of the Validation Group.

6. AGENDA ITEM SIX – Education and outreach in relation to the Convention

6.1 As was proposed at the last SAB session, the International Cooperation and Assistance Division (ICA) has assumed the lead for future activities in this area. As a follow-up, the SAB heard a presentation from Kumaresh Misra, Head of the International Cooperation Branch, on the various programmes developed by his Branch.

6.2 The SAB also heard a presentation by Alberto Fratadocchi on a proposal for a book for educational purposes in relation to the Convention. He invited other members of the SAB, in their personal capacity, to contribute to the manuscript.

7. AGENDA ITEM SEVEN – Introduction to discussion on advances in science and technology and their potential impact on the implementation of the Convention

Subitem 7(a): Presentation on the role of microreactor technology in chemical-process intensification

7.1 The SAB enjoyed an excellent presentation by Professor Jaap Schouten from Eindhoven University of Technology on the role of microreactor technology in chemical-process intensification.

Subitem 7(b): Discussion

7.2 The SAB is unanimous in its opinion that microreactor technology is not sufficiently advanced to be employed in the production of chemical weapons, for the following reasons:

(a) Microreactor technology is still, for the most part, in a developmental stage and there are only a handful of companies capable of designing and building microreactors.

(b) Microreactors, at this point in time, are not generic reactors but are designed and custom-built for a specific chemical process.

(c) Microreactors do not provide a simple “off-the-shelf” solution to chemical production. The development of a viable process requires a considerable investment in expertise and research.

Subitem 7(c): SAB recommendation on microreactor technology in chemical-process intensification and its impact on the implementation of the Convention

7.3 The SAB recommends that it review this matter prior to the next Review Conference.

8. AGENDA ITEM EIGHT – Initial discussion on the review of operational requirements and technical specifications for inspection equipment

8.1 Irvine Swahn (Team Leader, Inspectorate Division) provided the SAB with a briefing on the review of the operational requirements and technical specifications for inspection equipment currently being undertaken by the Secretariat.

8.2 The SAB welcomed the opportunity to assist in the review of equipment specifications, and suggested that the specifications should be more generic, so as to enable greater flexibility in the acquisition of equipment items.

8.3 The SAB requested that the proposed changes to the specifications (together with a brief explanation of the rationale for each proposed change) be provided to SAB members not less than six weeks in advance of the next SAB session, to afford them sufficient time to prepare the discussion of these proposals during the session.

9. AGENDA ITEM NINE – Future work of the SAB

Subitem 9(a): Meeting with governmental experts

9.1 The Secretary briefed the SAB on the preparations for the convening of a meeting with governmental experts[2], as requested by the Second Review Conference, to consider the SAB's report to the Second Review Conference submitted by the Director-General (RC-2/DG.1 and Corr.1). The objective of the meeting would be to review the suggestions and recommendations made by the SAB and to prepare a report to the Council. The meeting is currently planned for 11 to 13 February 2009 in The Hague. A provisional agenda has been prepared by the Secretariat, in coordination with the SAB.

Subitem 9(b): Interaction between the SAB and States Parties, and between the SAB and the policy-making organs

9.2 The Secretary informed the SAB that the Second Review Conference "invited the Director-General to provide considered advice to the Council on how to enhance the interaction between the SAB and States Parties as well as the policy-making organs, making best use of governmental experts" (RC-2/4, paragraph 9.134). The SAB members were requested to make proposals to the Secretary during the intersessional period.

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2 A similar meeting was convened in January 2004 following a request by the First Review Conference (see report: EC-36/2, dated 16 February 2004).

Subitem 9(c): Agenda for the Thirteenth Session of the SAB

9.3 The SAB discussed the agenda for its Thirteenth Session, currently planned for 30 March to 1 April 2009, and decided to address, inter alia, the questions of advances in nanoscience relevant to chemical defence purposes, to consider the report of the meeting with governmental experts and the list of scheduled chemicals (including saxitoxin), and to continue discussing the review of operational equipment with a view to making recommendations to the Director-General.

10. AGENDA ITEM TEN – Any other business

Subitem 10(a): Presentation on a proposal for a seminar on other chemical production facilities (OCPFs)

10.1 Robert Mathews presented a proposal for an 'OCPF seminar', based on a presentation that he gave, in his individual capacity, at the Open Forum held during the Second Review Conference in April 2008. He indicated that since April 2008 he has received considerable informal encouragement from a number of Member States to pursue the proposed seminar, as a means of providing participants with a better understanding of the technical features of OCPFs. He also emphasised the increasing importance of the OCPF regime in the overall scheme of routine industry verification, in light of the advances in science and technology, and of developments in industry.

10.2 One possible approach suggested was that such an OCPF seminar could be held back-to-back with a future SAB session as a means of facilitating involvement by the SAB members (including giving relevant scientific and technical presentations). In general, there was support from the SAB members regarding the value of such an OCPF seminar being conducted.

Subitem 10(b): SAB Port@l

10.3 The SAB expressed great appreciation for the installation of the SAB Port@l initiated by the Secretariat. The Port@l has already proved itself to be an important tool for SAB members in the preparation of SAB sessions and work to be conducted in the intersessional period.

Subitem 10(c): Continuity of the work of the SAB

10.4 The SAB noted that over the next two years, terms of office will expire for 22 of the 25 SAB members. This situation is detrimental to the conduct of business and the SAB recommends that the Director-General consider implementing a succession plan that would reduce the impact of the replacement of a large number of SAB members at once.

11. AGENDA ITEM ELEVEN – Adoption of the report

The SAB considered and adopted the report of its Twelfth Session.

12. AGENDA ITEM TWELVE – Closure of the session

The Chairperson closed the session at 17:40 on 26 November 2008.

Annexes:

Annex 1: List of participants in the Twelfth Session of the Scientific Advisory Board

Annex 2: (English only, unedited): Report of the Third Meeting of the Temporary Working Group on Sampling and Analysis, The Hague, the Netherlands, 20 – 21 November 2008

Annex 1

LIST OF PARTICIPANTS IN THE TWELFTH SESSION OF THE SCIENTIFIC ADVISORY BOARD

	Participant	State Party
1.	Rolando A. Spanevello	Argentina
2.	Robert Mathews	Australia
3.	Herbert de Bisschop	Belgium
4.	Zhiqiang Xia	China
5.	Danko Škare	Croatia
6.	Jean-Claude Tabet	France
7.	Detlef Maennig	Germany
8.	László Halász	Hungary
9.	R. Vijayaraghavan	India
10.	Mahdi Balali-Mood	Iran (Islamic Republic of)
11.	Alberto Breccia Fratadocchi	Italy
12.	Shuzo Fujiwara	Japan
13.	Abdool Kader Jackaria	Mauritius
14.	José González Chávez	Mexico
15.	Godwin Ogbadu	Nigeria
16	Bjørn-Arne Johnsen	Norway
17.	Titos Quibuyen	Philippines
18.	Igor V. Rybalchenko	Russian Federation
19.	Philip Coleman	South Africa
20.	Miguel A. Sierra	Spain
21.	Stefan Mogl	Switzerland
22.	Valery Kukhar	Ukraine
23.	Robin Black	United Kingdom of Great Britain and Northern Ireland
24.	James Robert Gibson	United States of America

Annex 2

REPORT OF THE THIRD MEETING OF THE TEMPORARY WORKING GROUP ON SAMPLING AND ANALYSIS

THE HAGUE, THE NETHERLANDS

20 – 21 NOVEMBER 2008

1. INTRODUCTION

1.1 The SAB Temporary Working Group (TWG) on Sampling and Analysis (S&A) held its third meeting on 20th and 21st November 2008 at the OPCW in The Hague.

1.2 The meeting was chaired by Robin Black on behalf of the SAB.

1.3 The list of participants in the meeting is given in Appendix 1.

1.4 The chairman welcomed the members of the TWG, and Mr Maciej Sliwakowski, Senior Analytical Chemist from the OPCW Laboratory. He thanked Patrice Palanque, Secretary to the SAB, and his staff for organising the meeting.

1.5 The following agenda was adopted.

(a) Welcome by the Chairman of the S&A TWG.

(b) Tour de table for introduction of the S&A TWG members.

(c) Adoption of the Agenda (Chairman of the S&A TWG).

(d) Matters on S&A arising from the Second Review Conference.

(e) Matters on S&A arising from Schedule 2 facility inspections.

(f) Non-scheduled degradation products, RCAs and old/abandoned CW agents as possible additions to OCAD.

(g) New/additional techniques for on-site analysis:

(i) Fast GC.

(ii) Solid phase microextraction (SPME).

(iii) Aqueous sample preparation.

(h) Off-site analysis:

(i) Trace analysis in investigations of alleged use.

(ii) Update on biomedical sample analysis.

(i) Toxin analysis (ricin & saxitoxin), off-site and on-site.

(j) Any other business.

(k) Summary of conclusions and recommendations.

(l) Date of next meeting.

(m) Closure of the meeting.

2. MATTERS ON S&A ARISING FROM THE SECOND REVIEW CONFERENCE

The Chairman highlighted those paragraphs of the Report of the Second Review Conference that refer to S&A. These are provided in the SAB Port@l. Most of these comments refer positively to S&A in general terms rather than to specific areas.

3. MATTERS ON S&A ARISING FROM SCHEDULE 2 FACILITY INSPECTIONS

3.1 The chairman thanked Mr Irvine Swahn, Team Leader, Inspectorate Division, for advising the meeting on issues arising from inspections of Schedule 2 facilities. The Convention requires these inspections to undertake S&A to check for the absence of undeclared scheduled chemicals (Verification Annex, Part VII paragraph 27). Since September 2006 a total 19 Schedule 2 inspections involving S&A had been completed; OPCW Report S/719/2008 refers.

3.2 The main issue for S&A was the time required to complete analyses. In most of the inspections only two samples were analysed. Possible approaches to reducing sampling time are addressed in section 5 below.

3.3 The OPCW is working on several projects aimed at facilitating S&A in Schedule 2 inspections. A database is to be constructed on Schedule 2 process chemistry so that low level process impurities can be more readily predicted and identified. The OPCW laboratory is developing procedures for quantification of such impurities. The Secretariat is also developing software that would give greater flexibility when the AMDIS software is operated in ‘blinded mode’. It would allow access to a commercial mass spectral library (with the concurrence of the State Party) so that apparent matches with scheduled compounds in the OPCW Central Analytical Database (OCAD) could be resolved more easily. Spectra of additional scheduled compounds that have commercial applications are being submitted to the Validation Group for addition to OCAD.

3.4 Efforts are currently being directed at decreasing analysis time by improvements in instrumental software and the use of autosamplers for gas chromatography-mass spectrometry (GC-MS) analysis. Improved sample preparation procedures are a longer term aspiration.

3.5 A broader discussion was held, which included proposed S&A in future inspections of Schedule 3 facilities. The OPCW currently has a limited capability to analyse certain Schedule 3 chemicals, particularly those that are highly volatile or too reactive to pass through a gas chromatograph. The OPCW would like to address these difficult analytes in collaboration with Member States, although at this preliminary stage further discussion is required. VERIFIN and the Spiez Laboratory are already addressing some difficult Schedule 3 compounds; Paula Vanninen offered to present on this topic at the next TWG meeting.

3.6 The OPCW Laboratory is in the process of procuring two FTIR instruments for rapid screening of compounds that are not amenable to GC-MS analysis. Air sampling onto sorbent tubes is currently being addressed, including on-tube derivatisation of lewisite with butanethiol. It was noted that no validated procedure currently exists for perfluoroisobutene (PFIB). Two approaches are cryogenic GC or capture on an adsorbent tube with on-tube derivatisation, e.g. with dimercaptotoluene.

4. NON-SCHEDULED DEGRADATION PRODUCTS, RCAs AND OLD AND ABANDONED CW AGENTS AS POSSIBLE ADDITIONS TO OCAD

4.1 At its two previous meetings the TWG has discussed and made recommendations on non-scheduled chemicals of relevance to inspections, investigations of alleged use and old and abandoned chemical weapons.

4.2 The TWG reaffirmed its recommendation that the mass spectra of these chemicals should be added to the OCAD in order to facilitate the verification provisions of the Convention, with the selective use of data from OCAD in accordance with the aims of the inspection (routine, challenge, investigations of alleged use, old and abandoned chemical weapons).

4.3 A copy of a report on amiton degradation products has been provided on the TWG Port@l to assist the TWG in future consideration of the addition of such degradation products to the OCAD.

5. NEW/ADDITIONAL TECHNIQUES FOR ON-SITE ANALYSIS

Fast GC

5.1 At the second meeting of the TWG Paula Vanninen described the results of preliminary investigations of fast GC undertaken at VERIFIN.

5.2 Robin Black summarised results of work undertaken at the Defence Science and Technology Laboratory (Dstl) in the UK several years ago (see SAB Port@l).

5.3 Fast GC of a selection of CW agents, plus the OPCW test mixture used to check system performance, was investigated using 8 GC columns with varying dimensions and stationary phases. Up to a five-fold reduction in chromatographic run times was obtained whilst maintaining good chromatographic resolution. However, significant variability was observed with retention indices. Under the conditions investigated none of the columns was able to meet the current on-site quality control requirement (± 5 retention index units of reference values) for some compounds in the test mixture. A Varian FactorFour® VF-5MS column (10 m x 0.15 mm x 0.3 µm film thickness) gave retention index values within ± 20 units of reference values for all the chemicals evaluated (test mix and CW agents), which would be acceptable for off-site analysis. For on-site GC–MS analysis, there is no rigid criterion for retention indices, but outside ± 20 index units of the OCAD values the match factor is penalised with increasing retention index difference. The use of hydrogen as carrier gas reduced retention times compared to helium, but was not acceptable due to a distortion of some of the mass spectra, particularly for some nerve agents.

5.4 Marieke van Deursen informed the TWG that much work on fast GC of non-CW analytes had been reported from the Eindhoven University of Technology in the Netherlands, and that this work should be consulted. A thesis, if available, will be posted on the TWG Port@l.

5.5 It was noted that for on-site analysis retention indices may be important for distinguishing some V agents, where the EI mass spectra are dominated by abundant ions originating from the dialkylaminoethylthio substituent on phosphorus, with very weak high mass ions (for off-site analysis additional techniques are used, e.g. chemical ionisation MS for determination of the molecular mass). The TWG recommended that the application of fast GC–MS to on-site analysis is studied further. Any fast GC–MS method used on-site must maintain sensitivity, chromatographic resolution and robustness comparable to the current method.

5.6 The OPCW is currently looking at a more modest reduction (2 fold) in GC run time, which should allow on-site retention index requirements to be more readily achieved. There will be an internship working on fast GC at VERIFIN and the OPCW Laboratory in 2009, funded by the International Cooperation Branch of the OPCW.

Solid-Phase Microextraction (SPME)

5.7 Sample preparation takes up a major part of analysis time, and simplified procedures are desirable for on-site analysis. SPME is now a mature technique and is used routinely by a number of Member States in the context of homeland security. A discussion was held on the pros and cons of SPME for on-site analysis. SPME provides a rapid and semi-automated sampling procedure for analytes in air and in aqueous solution. For many analytes it also provides low limits of detection. Disadvantages are the cost of the fibres, the need to use them only once in order to avoid cross contamination, time taken to condition the fibres, and their susceptibility to interference by high levels of contaminants. The predominant view of the TWG was that SPME-GC-MS would provide a useful screening procedure in certain scenarios but would not be a complete substitute for current on-site sampling procedures. Its use for aqueous samples is discussed below.

Sample preparation of aqueous solutions

5.8 A major disadvantage of current on-site S&A is the time and additional equipment that is required to process aqueous samples. Current procedures for polar precursors or degradation products of Schedule 1 chemicals in aqueous solution require centrifugal evaporation to dryness followed by derivatisation with water-sensitive derivatising agents, a procedure that can take up to three hours. In recent years a number of new techniques have been developed for aqueous sample analysis in non-CW related spheres, and several of these techniques have been explored for CW analysis. One of the problems with regard to CW related analytes is the range of reactivities, polarities and pK_As of the various degradation products such as phosphonic acids, thiodiglycol, ethanolamines etc.

5.9 Techniques that have been reported in other fields include:

(a) solid-phase extraction (SPE) with on-SPE derivatisation;

(b) on-SPME derivatisation;

(c) hollow-fibre liquid phase extraction with in-fibre derivatisation;

(d) stir-bar sorptive extraction with derivatisation;

(e) microemulsion extractive derivatisation;

(f) two-phase extractive derivatisation;

(g) aqueous phase alkylation;

(h) aqueous phase derivatisation with chloroformates; and

(i) molecularly imprinted polymers.

5.10 Mui Tiang Sng described work undertaken at the DSO Verification Laboratory, Singapore (see SAB Port@l). SPME was shown to be effective in the extraction of CW agents from water, and selected degradation products with on-fibre derivatisation. More impressive results were obtained using the newer technique of hollow-fibre-liquid phase microextraction (HF-LPME), in which the organic solvent and derivatising agent, if required, are held within a small disposable hollow fibre at the tip of a syringe. This technique was found to be far superior to SPME for degradation products of CW agents. Both techniques are very sensitive, and both techniques have been successfully validated with proficiency test samples. A disadvantage of SPME is that it can be seriously affected by matrix materials whereas HF-LPME thus far appears to be more robust. HF-LPME has the advantages of ease of use, short extraction time and low cost. It also requires a very small volume of solvent for extraction (1 ml), compared to SPME (3 ml) and the currently used on-site ROP (10 ml). It requires further validation with a wider range of scheduled compounds and degradation products before it could be recommended as an operating procedure for on-site analysis.

5.11 R.Vijayaraghavan summarised investigations of several techniques for CW agents and their degradation products undertaken at the Vertox Laboratory, India (see SAB Port@l). Single-drop microextraction (SDME) and HF-LPME were investigated as liquid-phase microextraction techniques. The advantages described above were found for HF-LPME. Extraction conditions do have to be optimised for HF-LPME. Additional techniques investigated for polar degradation products were in situ derivatisation (propyl or pentyl esters of phosphonic acids) and extraction (INDEX), and anion or cation exchange SPE for acidic and basic analytes, followed by derivatisation. Further assessment is required for some of these techniques.

5.12 Paula Vanninen presented a review of the various techniques (see SAB Port@l), including variations in derivatising agents. Consistent with the studies reported above, HF-LPME was recognised as the most promising technique with the advantages:

(a) it is very sensitive;

(b) it is more robust than drop-based LPME;

(c) it is much cheaper than SPME and the hollow fibre can be disposed after each use to avoid cross contamination; and

(d) it has been successfully demonstrated for CW agent degradation products with derivatisation to tert-butyldimethylsilyl (TBDMS) derivatives.

5.13 As with SPME, HF-LPME is a non-exhaustive extraction method and it has yet to be shown if it is prone to interference by contaminants as is the case with SPME.

5.14 The TWG discussed the various techniques and agreed that HF-LPME was the most promising. It also expressed the view that other techniques should not yet be discounted. One potential method that has yet to be successfully demonstrated for CW related analytes is SPE with on-SPE derivatisation. The TWG agreed that a technique that was universally applicable to CW degradation products was preferable, as opposed to techniques that work only for selected analytes (e.g. phosphonic acids). Currently silylation is the only derivatisation method that fulfils this requirement. The merits of trimethylsilyl diazomethane as a safe methylating agent was recognised and it was suggested as a possible additional derivatising agent for phosphonic acids.

5.15 The TWG encourages laboratories to collaborate on further work in this area.

6. OFF-SITE ANALYSIS

Trace analysis in investigations of alleged use

6.1 The OPCW Laboratory in cooperation with Member States has established rigid criteria for the unequivocal identification of chemicals relevant to the CWC at levels (ppm +) where full scan spectral data can be acquired. These criteria are at least up to the standards required by other regulatory bodies.

6.2 There are currently no OPCW criteria for environmental-type samples where identification requires compound-targeted trace analysis under non-scanning conditions. This is probably only relevant to the OPCW in the context of investigations of alleged use. Other regulatory bodies, including the European Commission (EC), US Food and Drug Administration (FDA) and the World Anti-Doping Agency (WADA), have developed criteria for unequivocal identification of banned substances in food, animal or human sources at trace levels. The International Laboratory Accreditation Cooperation Guide (ILAC-G7) states that the identification of a prohibited substance must result from a direct comparison with a reference material analysed in parallel or series with the test sample using a mass spectrometric technique, and there must be written laboratory criteria as to what constitutes a match.

6.3 The TWG members expressed a number of different views on the relevance of trace analysis. Some members were of the view that it has low relevance to verification activities. Other members of the TWG regarded trace analysis as being of high relevance to investigations of alleged use, based on historical experience. It was pointed out that the Finnish Blue Book, which provided the initial basis for OPCW ROPs, does contain proposed criteria for selected ion and multiple reaction monitoring MS, techniques that are usually used for trace analysis. All members of the TWG agreed that at the present time it was not appropriate to test the capabilities of laboratories with regard to trace analysis.

6.4 As a way forward it was agreed that a correspondence group should be established within the TWG and be asked to recommend criteria to the next meeting of the TWG. Paula Vanninen of VERIFIN agreed to coordinate this correspondence group (members: VERIFIN Finland, LLNL US, DSTO Australia, Dstl UK, CEB France, Vertox India, FOI Sweden).

6.5 The TWG emphasised that trace analysis should always be considered with other evidence and not in isolation.

Update on biomedical sample analysis

6.6 Robin Black summarised the current situation with regard to biomedical samples. Although these plans have yet to be confirmed, the OPCW is hoping to hold the first Confidence Building Exercise in mid-2009. Member States will be invited to participate. Subject to confirmation of funding, TNO Defence, Security and Safety, the Netherlands, has offered to prepare samples for this exercise, with Robin Black acting as the coordinator. The proposal is that urine (probably synthetic) would be used as the matrix to simplify handling and safety procedures, and would be spiked with nerve agent and sulphur mustard metabolites at high (50 or 100 ppb) and low (5 or 10 ppb) concentrations of each metabolite. The use of high and low concentrations will demonstrate the limitations of some techniques, and the low concentration samples will also stimulate discussions on criteria for a positive identification in trace analysis. Details of analytical methods (GC-MS and liquid chromatography-mass spectrometry, LC-MS) will be distributed well before commencement of the exercise. One issue that has not yet been resolved is the preparation and distribution of standards; the OPCW is seeking a laboratory to volunteer to prepare some metabolites of sulphur mustard. Ideally internal standards are also desirable but this may not be

possible with the resources currently available. It is emphasised that this will not be a test of proficiency and that experienced laboratories will offer help and advice on request during the exercise. The objective will be to build expertise in biomedical sample analysis in participating laboratories.

7. TOXIN ANALYSIS (RICIN & SAXITOXIN), OFF-SITE AND ON-SITE

7.1 Laboratories were invited to respond to 2 questionnaires on toxin analysis, one for saxitoxin, prepared by Spiez Laboratory (Martin Schär), and one for ricin (Sten-Åke Fredriksson) prepared by FOI CBRN. Laboratories were asked to rank combinations of two orthogonal analytical methods for unequivocal identification. Laboratories were encouraged to suggest additional analytical methods.

7.2 Approximately 20 questionnaires were sent to interested laboratories. Eight responses were received. Results were extracted from the returned questionnaires (see SAB Port@l) and the following conclusions were made on the basis of the rankings.

7.3 For saxitoxin the combination of LC-fluorescence detection with LC-MS/MS (multiple reaction monitoring) or LC-MS was favoured by several laboratories. Immunoassay, as is used routinely in the food industry, was an alternative to LC-fluorescence. Other methods proposed were 2D-nuclear magnetic resonance (2D-NMR), LC-nitrogen chemiluminescence detection plus other method combinations.

7.4 For ricin the preferred combination was a real-time polymerase chain reaction (PCR) method or immunological assay (ELISA) combined with MS and LC-MS/MS. Several additional methods (see SAB Port@l) were also suggested.

7.5 The outcome of the questionnaires, together with criteria for unequivocal identification already adopted by EC, WADA and others, will be used to formulate firm requirements for the OPCW.

7.6 Francesco Pilo summarised an evaluation of a commercial biostrip test for rapid on-site screening of samples as an indicator of environmental safety with regard to ricin. Further evaluation of and possible improvements to such assays would be required before they could be recommended for OPCW on-site screening prior to additional analysis, if required, off-site.

7.7 As a way forward the TWG asked the Spiez and FOI Laboratories to make recommendations on methods and criteria for off-site and on-site identification of saxitoxin and ricin at the next meeting of the TWG. Martin Schär informed the TWG that during a recent ricin workshop of the Global Health Security Action Group several laboratories agreed to organise a round-robin exercise on ricin analysis, to be held in October 2009. The Spiez Laboratory will provide samples and coordinate the exercise. The results will be made available to the TWG and should assist the members in making firmer recommendations to the SAB.

7.8 A view was expressed that the OPCW designated laboratory network should include laboratories that have the capability of analysing other toxins (in addition to ricin and

saxitoxin) particularly in the context of possible investigations of alleged use involving other toxins.

8. ANY OTHER BUSINESS

8.1 The Chairman thanked Mr Maciej Sliwakowski for attending the meeting and advising the TWG on S&A procedures. The TWG welcomed an offer by Mr Sliwakowski to provide a detailed overview of OPCW on-site procedures to the next meeting of the TWG.

9. SUMMARY OF CONCLUSIONS and RECOMMENDATIONS

9.1 The TWG reaffirmed its previous recommendation that additional chemicals (non-scheduled degradation products and their derivatives, riot control agents, and old and abandoned chemical agents), as listed in Annexes to the two previous meetings of the TWG, should be added to the OCAD, with the selective use of data from OCAD in accordance with the aims of the inspection.

9.2 The TWG agreed that a reduction of analysis time (sample preparation and instrumental analysis time) during on-site inspections should be a high priority for the Secretariat. The following recommendations were agreed.

(a) The TWG recognised the difficulty of substantially reducing GC-MS run times using fast GC whilst minimising the variability in retention indices. It was recommended that further study should be undertaken to assess the effects on retention indices with respect to on-site analysis.

(b) The TWG was of the predominant view that SPME is not sufficiently robust with regard to ‘dirty’ matrices to be a complete substitute for current on-site sample preparation procedures. The TWG did acknowledge that SPME can provide a rapid and sensitive screening procedure.

(c) The TWG agreed that a reduction in on-site sample preparation time for polar analytes in aqueous samples should be a high priority for the Secretariat. Hollow-fibre-liquid phase microextraction in combination with in-fibre silylation was regarded as the most promising technique. The TWG encouraged collaboration between laboratories in this area.

9.3 The TWG members expressed different views on the importance of trace analysis of environmental-type samples in the context of investigations of alleged use. As a way forward a correspondence group has been established within the TWG and will recommend criteria for a positive identification to the next meeting of the TWG.

9.4 The TWG noted and supported the intention of the OPCW Laboratory to hold a confidence building exercise on biomedical sample analysis in 2009. This was a recommendation of the SAB TWG on Biomedical Sampling and Analysis.

9.5 The TWG evaluated the results of questionnaires returned by eight laboratories on methods that are preferred for toxin identification. A correspondence group, established at the second meeting of the TWG, will submit recommendations for identification requirements for toxins to the next meeting of the TWG.

10. DATE OF NEXT MEETING

10.1 To be arranged subject to funds being made available to the SAB Trust Fund.

11. CLOSURE OF THE MEETING

11.1 The meeting was closed at 17.15 on 21 November 2008.

Appendix 1

LIST OF PARTICIPANTS IN THE THIRD MEETING OF THE TEMPORARY WORKING GROUP ON SAMPLING AND ANALYSIS

	Participant	Member State
1.	Robert Mathews	Australia
2.	Jiří Matoušek	Czech Republic
3.	Jiří Cermak	Czech Republic
4.	Paula Vaninnen	Finland
5.	Jean-Claude Tabet	France
6.	Anne Bossée	France
7.	Ralf Trapp	Germany
8.	R. Vijayaraghavan	India
9.	Shigeyuki Hanaoka	Japan
10.	Francesco Pilo	Italy
11.	Jose Luz Gonzalez-Chavez	Mexico
12.	Marieke van Deursen	Netherlands
13.	Mui Tiang Sng	Singapore
14.	Philip Charles Coleman	South Africa
15.	Miguel A. Sierra[3]	Spain
16.	Roberto Martinez-Alvarez	Spain
17.	Sten Åke Fredriksson	Sweden
18.	Martin Schär	Switzerland
19.	Robin Black[4]	United Kingdom of Great Britain and Northern Ireland
20.	Armando Alcaraz	United States of America
21.	James Robert Gibson	United States of America

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3 Vice-Chairman of the TWG

4 Chairman of the TWG