



Verification Division
S/239/2001
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ENGLISH only

SUMMARY REPORT OF THE EIGHTH MEETING OF THE VALIDATION GROUP FOR THE UPDATING OF THE CENTRAL OPCW ANALYTICAL DATABASE 28 - 29 NOVEMBER 2000

1. The validation group met for the eighth time from 28 to 29 November 2000 to discuss the evaluation of new analytical data for possible inclusion in the Central OPCW Analytical Database, and also to consider matters related to this database. The meeting was chaired by Mr Eric Wils of the Netherlands.
2. The evaluators for the four analytical techniques evaluated the new data and sent their written reports to the appointed coordinators, as follows:

IR:	Mr Martin Söderström (Finland)
MS:	Mr Edward White (USA)
NMR:	Mr Luigi Abis (Italy)
GC(RI):	Mr Maciej Sliwakowski (Poland)

The coordinators provided an evaluation summary report to the group's Chairman for discussion at the meeting. The evaluators finalised the evaluation of the analytical data, and confirmed that the data approved are technically valid.

3. The validation group forwarded the validated analytical data to the Director-General for appropriate action.
4. The validation group was informed on the following issues:
 - (a) the new authentication and certification procedure was approved by the Executive Council (EC-XX/DEC.5, dated 24 July 2000) and is now in place. Certificates issued before July 2000 have been renamed authentication documents;
 - (b) the list of new validated data forwarded by the validation group at its seventh meeting was adopted by the Executive Council for inclusion in the Central OPCW Analytical Database (EC-XXI/DEC.3, dated 5 October 2000);
 - (c) the temporary working group on analytical procedures of the Scientific Advisory Board (SAB) discussed the possible inclusion of non-scheduled chemicals and riot control agents in the Central OPCW Analytical Database. The SAB will issue guidelines on this issue in early 2001; and

- (d) the Secretariat has produced a standard operating procedure (SOP) on the organisation of the Central OPCW Analytical Database, which is available to the members of the validation group upon request.
5. The Secretariat has announced the release of version 3 of the hard-copy version of the Central OPCW Analytical Database on CD-ROM (S/222/2000, dated 22 November 2000). Members of the validation group received a copy of the CD-ROM and were requested to examine the database, in particular the accessibility of the data, and to provide comments for discussion at the group's next meeting. In early 2001 the Secretariat will distribute a first version of the electronic version of the database containing 521 mass spectra. This data set has been already distributed to the MS evaluators of the validation group for testing, with positive results.
6. Upon request, the validation group reviewed the formats for analytical data submitted in electronic form. It was decided that the formats should remain basically the same, but that the Secretariat's identification code and the relevant schedule number should be added. Examples of these formats are shown in annex 1 hereto. The principle of one spectrum per file is to be maintained. Furthermore, the group decided that:
- from 1 January 2001 onwards, contributing laboratories should submit both hard-copy and electronic versions of spectra for evaluation;
 - in the case of an inconsistency between the electronic and hard-copy versions of a spectrum, the electronic version will be considered to be correct;
 - GC/MS data may be submitted as AMDIS¹ libraries;
 - the submission of complete data files (e.g. GC/MS runs) in addition to single spectra for evaluation will be encouraged; and
 - the above-mentioned SOP containing the procedures and criteria for the evaluation process must be adopted once these decisions are common practice.
7. The rules for naming chemicals, which were adopted during the seventh meeting of the validation group, were again discussed. It was felt that the examples giving the systematic and the trivial names in one field were confusing, and that the trivial names should appear in a separate field for synonyms. The amended naming rules are attached as annex 2.
8. The group discussed a number of issues relating to the GC(RI) data.
- 8.1 It was decided to apply retrospectively the alphabetic letter extensions (a, b, c, etc.) to diastereoisomers. This would lead to changes in the numbering of the GC(RI) data, as shown in the following table:

Old numbering	New numbering	Chemical name	Retention index	Schedule number
05-4-0012	05-4-0012:a	sec-Butyl methylphosphonofluoridate	915	1.A.01
05-4-0013	05-4-0012:b	sec-Butyl methylphosphonofluoridate	918	1.A.01

¹ AMDIS: Automated Mass Spectrometric Deconvolution and Identification Software.

05-4-0025	05-4-0025:a	Pinacolyl methylphosphonofluoridate	1044	1.A.01
05-4-0027	05-4-0025:b	Pinacolyl methylphosphonofluoridate	1048	1.A.01
05-4-0063	05-4-0063:a	2-Methylcyclohexyl methylphosphonofluoridate	1260	1.A.01
05-4-0064	05-4-0063:b	2-Methylcyclohexyl methylphosphonofluoridate	1262	1.A.01

8.2 The different values of the retention indices (RIs) of these diastereoisomers will be stored in the Central OPCW Analytical Database. However, the Secretariat will select just one average value for inclusion in the on-site databases using AMDIS software.

8.3 The difference in RI values for the same chemical measured on the same stationary phase purchased from different suppliers was discussed. Examples are presented in the following table:

Chemical	RI library	MDN, DB5	AT-5MS	CP-SIL8 PEST	HP5MS SPB5	ΔRI_{max}
Trimethyl phosphate	938	928	930	932	936	8
2,6-Dimethylphenol	1112	1111	1112	1108	1112	4
5-Chloroaniline	1308	1307	1308	1300	1308	8
Tributyl phosphate	1655	1642	1644	1649	1655	13
Hexachlorobenzene	1734	1716	1728	1725	1734	18
Dibenzothiophene	1774	1776	1780	1764	1774	16
Malathion	1986	1965	1970	1975	1986	21
Methyl stearate	2130	2127	2128	2127	2130	3

In order to gain a better insight into these variations it was proposed that newly submitted data should be accompanied by RI measurements of the OPCW GC/MS check mixture (see chemicals in the above table).

9. In the GC(RI) part of version 3 of the Central OPCW Analytical Database a few errors have been identified, which need to be corrected. For example, the name of the chemical with the Secretariat's identification code 17-4-0010 was wrongly reproduced as isopropyl trimethylsilyl propylphosphonate in various tables (EC-XII/DG.2, dated 4 September 1998). The data as originally submitted bears the correct name, propyl trimethylsilyl propylphosphonate.

The names and structural formulas of the compounds with the codes 16-4-0032, 16-4-0034, 16-4-0037 and 16-4-0039 are incorrect, and must be revised as follows:

16-4-0032: O-Isopropyl O-trimethylsilyl isopropylphosphonothionate
 16-4-0034: O-Isopropyl O-trimethylsilyl propylphosphonothionate

16-4-0037: O-Propyl O-trimethylsilyl isopropylphosphonothionate
16-4-0039: O-Propyl O-trimethylsilyl propylphosphonothionate

The contributing laboratory has already submitted new forms to the Secretariat.

10. The IR evaluators discussed the inclusion of both condensed phase and vapour phase spectra in the same database. These two sets of data are not directly comparable, and therefore a search of the combined database may give misleading results. The evaluators recommended that the OPCW identification codes for the vapour phase data be modified to distinguish them from those of the condensed phase. This could be achieved by adding the suffix “: v” to the OPCW identification codes for the vapour phase spectra. The IR coordinator will provide the Secretariat with a list of the spectra in question.
11. A number of mass spectra recorded on an ion trap (IT) mass spectrometer was submitted. This type of spectrometer is known to produce somewhat different data (in particular the relative intensities of the mass fragments) from those obtained using quadrupole (Q) and magnetic sector (M) instruments. The validation group decided to postpone accepting a number of these IT-type spectra until better insight is gained into the difference between Q- and M-type spectra. A test will be carried out by Mr Gary Mallard (USA), and the outcome will be discussed at the group’s next meeting . The Secretariat should be aware that the database contains mainly Q- and M-type mass spectra, and this fact should be taken into account in the selection of new equipment.
12. The following new analytical data were provided for evaluation:
 - IR: from 04-1-0047 to 04-1-0070
 - MS: 04-2-0205:r, 04-2-0228:r, 04-2-0229:r, 04-2-0230:r
from 04-2-0238 to 04-2-0261
from 05-2-0149 to 05-2-0198;
07-2-0348:r, 07-2-0488:r, 07-2-0517:r, and
from 07-2-0520 to 07-2-0615
 - NMR: 05-3-0083:r, 05-3-0092:r, 05-3-0093:r, 05-3-0094:r, 05-3-0098:r, 05-3-0099:r, 05-3-0110:r, 05-3-0113:r, 05-3-0114:r, 05-3-0116:r, 05-3-0119:r, 05-3-0120:r, 05-3-0137:r, 05-3-0144:r, 05-3-0147:r, 05-3-0148:r, 05-3-0151:r, 05-3-0158:r, 05-3-0162:r, 05-3-0165:r, 05-3-0166:r, 05-3-0167:r, 05-3-0169:r, 05-3-0173:r
 - GC(RI): from 04-4-0047 to 04-4-0070,
from 05-4-0158 to 05-4 0207, and
from 07-4-0286 to 07-4-0381

The Secretariat distributed the new data to the evaluators present at the meeting, and will send the new data to the other evaluators as soon as possible.

13. Evaluators for the four analytical techniques were appointed (annex 3), and the new analytical data were transferred to them. The evaluators agreed to send their written evaluation reports to the appointed coordinators by 15 February 2001 at the latest.
14. The coordinators agreed to send an evaluation summary report to the group's Chairman by 1 March 2001 at the latest for discussion at the ninth meeting of the validation group, which is scheduled for 13 and 14 March 2001. The evaluators agreed to attend that meeting prepared to finalise the evaluation of the analytical data.

Annexes:

Annex 1: Formats for analytical data submitted in electronic form

Annex 2: Rules for naming chemicals to be included in the Central OPCW Analytical Database

Annex 3: List of evaluators for the four analytical techniques

Annex 1

FORMATS FOR ANALYTICAL DATA SUBMITTED IN ELECTRONIC FORM

Input data formats

(a) NIST ASCII format for MS:

Example:

Name: Cyclopropylmethyl S-2-dimethylaminoethyl ethylphosphonothiolate

Synon: 02-2-0030 (Secretariat ID number)

Synon: S1.A.03 (Schedule number of chemical)

Comments: S1.A.03; 02-2-0030

RI: 1234

Formula: C10H22NO2PS

MW: 251

CASNO: ID0

Num Peaks: 33

39 42; 40 5; 41 19; 42 124;
43 49; 44 49; 45 11; 47 15; 53 23;
54 17; 55 144; 56 84; 57 35; 58 999;
59 49; 60 5; 61 22; 63 6; 65 20;
70 45; 71 764; 72 115; 73 6; 77 5;
90 5; 93 15; 102 16; 104 16; 109 7;
147 6; 153 8; 168 5; 180 9.

(b) HP-JCAMP-DX format for MS

##TITLE = 1,2-Dimethylbutyl methylphosphonofluoridate

##JCAMP-DX=Revision 4.10

##DATATYPE=MASS SPECTRUM

##SAMPLE DESCRIPTION = any text string up to 128 characters. Full schedule number of the chemical plus additionally (optional) as many as possible synonyms. For example S1.A.01, Cyclohexylsarin,GF

##NAMES=02-2-0188 ! Secretariat ID code. Up to 10 chr\$

##CAS NAME=1,2-Dimethylbutyl methylphosphonofluoridate

##MOLFORM=C7H16FO2P ! Molecular formula. Alphabetic order of heteroatoms

##CAS REGISTRY NO= 000000-43-0

##MP= 6.4 ! Melting point of the chemical. Up to one decimal place.

##BP= 234.6 ! Boiling point of the chemical. Up to one decimal place.

##MW= 284 ! monoisotopic molecular weight

##\$RETENTION INDEX=2222.5 ! Up to two decimal places.

```
##$CONDENSED SPECTRUM=NO
##NPOINTS= 37
##XYDATA=(XY..XY)
 26  1
 27 12
 40  2
```

(c) JCAMP-DX format for IR

```
##TITLE=1-Methylpropyl methylphosphonofluoridate
##JCAMP-DX=4.24
##DATA TYPE=INFRARED SPECTRUM
##MINY=0
##MAXY=2.0
##CAS REGISTRY NO=352-52-3
##NAMES=sec-Butyl methylphosphonofluoridate
##MOLFORM= C5 H12 F O2 P
##MW=154.1
##DATE=12/30/99
##SPECTROMETER/DATAS=Bio-Rad
##XUNITS=1/CM
##YUNITS=ABSORBANCE
##FIRSTX=6.673687652E+02
##LASTX=3.996497340E+03
##NPOINTS=1727
##FIRSTY=1.4953820000E-02
##LASTY=6.0425640000E-03
##DELTA X=1.928811457E+00
##XFACTOR=1.0
##YFACTOR=0.000061036
##ORIGIN= OPCW Code 02-1-006
##OWNER=Laboratory No. 02
##SAMPLING PROCEDURE=Tracer
##RESOLUTION=4.0
##CROSS REFERENCE= Schedule 1A1
##XYDATA=(X++(Y..Y))
667.37 245 233 199 234 284 268
678.94 257 269 257 251 251 265....
```

Annex 2

RULES FOR NAMING CHEMICALS TO BE INCLUDED IN THE CENTRAL OPCW ANALYTICAL DATABASE

1. In general, the name of a chemical (spelling, punctuation, spaces, etc.) should be based on the name given in the Convention's Annex on Chemicals.
2. The following additional rules should be followed in cases where the information in the Annex on Chemicals is insufficient to designate only one name.
 - 2.1 The first letter of the name should be capitalised (but not the structural and stereo-descriptors sec-, tert-, cis- and trans-).
 - 2.2 Use the trivial names for the following radicals:
Saturated branched: Isopropyl, Isobutyl, sec-Butyl, tert-Butyl. Also use Pinacolyl.
Unsaturated: Vinyl, Allyl, Isopropenyl.
 - 2.3 If a compound has several substituents, these substituents are to be listed in alphabetical order, disregarding the presence of the prefixes O- or S-.
 - 2.4 Parentheses are to be used in the following cases: around prefixes defining substituted substituents; after the numerical multiplicative prefixes 'bis', 'tris', etc.; around simple substituent prefixes to separate locants of the same type referring to different structural elements; and to avoid ambiguities.
 - 2.5 For a radical with a branching structure, the name should be derived from the longest continuous chain, starting (position 1) at the conjunction with the parent structure. For example: Methylphosphonofluoridate made using 5-Methyl-3-hexanol is 1-Ethyl-3-methylbutyl methylphosphonofluoridate.
 - 2.6 A distinction is to be made between thiolate and thionate, depending on whether the sulfur (S) atom is single or double bonded to the phosphorus atom.
 - 2.7 Names should be as short as possible, and unnecessary characters should be omitted; for example,
 - (a) the n- in n-alkyl;
 - (b) the 1- before 1-alkyl in case of a normal alkyl chain;
 - (c) the O in O-Alkyl alkylphosphonohalidates;
 - (d) the O in O-Alkyl S-2-dialkylaminoethyl alkylphosphonothiolates; and
 - (e) any unnecessary brackets and parentheses.

3. The following tables illustrate the application of these naming rules for scheduled chemicals and their associated derivatives.

Examples of names of scheduled chemicals

Schedule	Name
1.A.01	Alkyl alkylphosphonofluoridate
1.A.02	Alkyl N,N-dialkylphosphoramidocyanidate
1.A.03	Alkyl S-2-dialkylaminoethyl alkylphosphonothiolate
1.A.04	2-Chloroethylchloromethylsulfide
	Bis(2-chloroethyl)sulfide
	Bis(2-chloroethylthio)methane
	1,2-Bis(2-chloroethylthio)ethane
	1,3-Bis(2-chloroethylthio)propane
	1,4-Bis(2-chloroethylthio)butane
	1,5-Bis(2-chloroethylthio)pentane
	Bis(2-chloroethylthiomethyl)ether
	Bis(2-chloroethylthioethyl)ether
1.A.05	2-Chlorovinylchloroarsine
	Bis(2-chlorovinyl)chloroarsine
	Tris(2-chlorovinyl)arsine
1.A.06	Bis(2-chloroethyl)ethylamine
	Bis(2-chloroethyl)methylamine
	Tris(2-chloroethyl)amine
1.A.07	Saxitoxin
1.A.08	Ricin
1.B.09	Alkylphosphonic difluoride
1.B.10	Alkyl 2-dialkylaminoethyl alkylphosphonite
1.B.11	Isopropyl methylphosphonochloridate
1.B.12	Pinacolyl methylphosphonochloridate
2.A.01	O,O-Diethyl S-2-diethylaminoethyl phosphorothiolate
2.A.02	1,1,3,3,3-Pentafluoro-2-(trifluoromethyl)-1-propene
2.A.03	3-Quinuclidinyl benzilate
2.B.04	To avoid any confusion the O and S groups should be indicated. Examples :
	Methylphosphonic dichloride
	Dimethyl methylphosphonate
	Methyl methylphosphonate instead of methyl methylphosphonic acid
	O-Ethyl S-phenyl ethylphosphonothiolothionate
2.B.05	N,N-Dialkylphosphoramidic dihalide
2.B.06	Dialkyl N,N-dialkylphosphoramidate
2.B.07	Arsenic trichloride
2.B.08	2,2-Diphenyl-2-hydroxyacetic acid
2.B.09	3-Quinuclidinol
2.B.10	2-N,N-Dialkylaminoethyl chloride
2.B.11	2-N,N-Dialkylaminoethanol
2.B.12	2-N,N-Dialkylaminoethanethiol
2.B.13	Bis(2-hydroxyethyl)sulfide
2.B.14	3,3-Dimethyl-2-butanol

3.A.01	Carbonyl dichloride
3.A.02	Cyanogen chloride
3.A.03	Hydrogen cyanide
3.A.04	Trichloronitromethane
3.B.05	Phosphorous oxychloride
3.B.06	Phosphorous trichloride
3.B.07	Phosphorous pentachloride
3.A.08	Trimethyl phosphite
3.A.09	Triethyl phosphite
3.A.10	Dimethyl phosphite
3.A.11	Diethyl phosphite
3.B.12	Sulfur monochloride
3.B.13	Sulfur dichloride
3.B.14	Thionyl chloride
3.B.15	Ethyldiethanolamine
3.B.16	Methyldiethanolamine
3.B.17	Triethanolamine

Examples of names of derivatives (D.S.)

D.S.	Type of name
1.A.05	2-(2-Chlorovinyl)-5-methyl-1,3,2-benzodithiarsole
2.A.03	Bis(trimethylsilyl)benzilate
	3-Quinuclidinyl trimethylsilyl ether
2.B.07	2-Chloro-5-methyl-1,3,2-benzodithiarsole
2.B.11	2-N,N-Dialkylaminoethyl trimethylsilyl ether
	2-N,N-Dialkylaminoethyl tert-butyldimethylsilyl ether
2.B.12	2-N,N-Dialkylaminoethyl trimethylsilyl sulfide
2.B.13	Bis(2-trimethylsiloxyethyl)sulfide
3.B.15	Bis(2-trimethylsiloxyethyl)ethylamine
3.B.16	Bis(2-trimethylsiloxyethyl)methylamine
3.B.17	Tris(2-trimethylsiloxyethyl)amine
	Tris(2-tert-butyldimethylsiloxyethyl)amine

Annex 3

LIST OF EVALUATORS FOR THE FOUR ANALYTICAL TECHNIQUES

IR evaluators:

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