Science for Diplomats at EC-83

Chemical Weapons Sample Stability and Storage

12 October 13:30 – 14:45
Ooms Room

(light lunch available at 13:00)

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Samples Collected in OPCW Investigations

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Samples Collected in OPCW Investigations

OPCW **Scientific Advisory Board**

Twenty-Third Session
18 – 22 April 2016

SAB-23/WP.2
25 May 2016
ENGLISH only

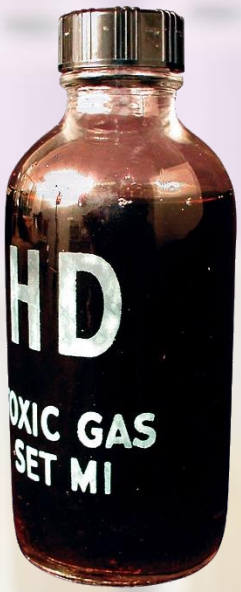
RESPONSE TO THE DIRECTOR-GENERAL'S REQUEST TO THE SCIENTIFIC ADVISORY BOARD TO PROVIDE FURTHER ADVICE ON CHEMICAL WEAPONS SAMPLE STABILITY AND STORAGE

1. EXECUTIVE SUMMARY

1.1 The Scientific Advisory Board (SAB) has considered the long-term storage and stability of samples collected in the context of the OPCW's investigations, including fact-finding missions and the Declaration Assessment Team, according to the Director-General's questions of 2 November 2015 (see Annex 1).

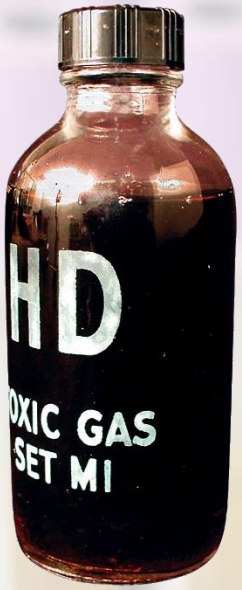
1.2 In the context of the OPCW's investigations, the Technical Secretariat has since 2013 received numerous samples, which are stored in the OPCW Laboratory at room temperature or refrigerated at 4 °C.

Sample Types



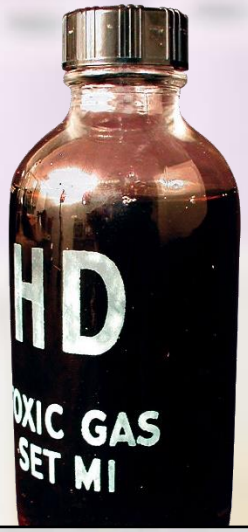
Bulk chemicals

Sample Types



Bulk chemicals

Sample Types



Bulk chemicals

Biomedical samples

Highly variable concentration and heterogeneous samples

Director-General's Request for Advice on Long-Term Storage and Stability of Samples Collected in Relation to the Potential Use of Chemical Weapons

In reference to the sample types described on the previous slide:

- Given the current storage conditions in the OPCW Lab (typically room temperature or 4°C), how quickly and through what process would samples degrade to a point where analysis of the samples would likely no longer return credible results?

- What are the best-practice conditions for long-term sample storage?

- Given these best-practice storage conditions, how quickly and through what type of process could samples degrade to a point where analysis of the samples would likely no longer return credible results?

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LABOR SPIEZ

SAB-23/WP.2
25 May 2016

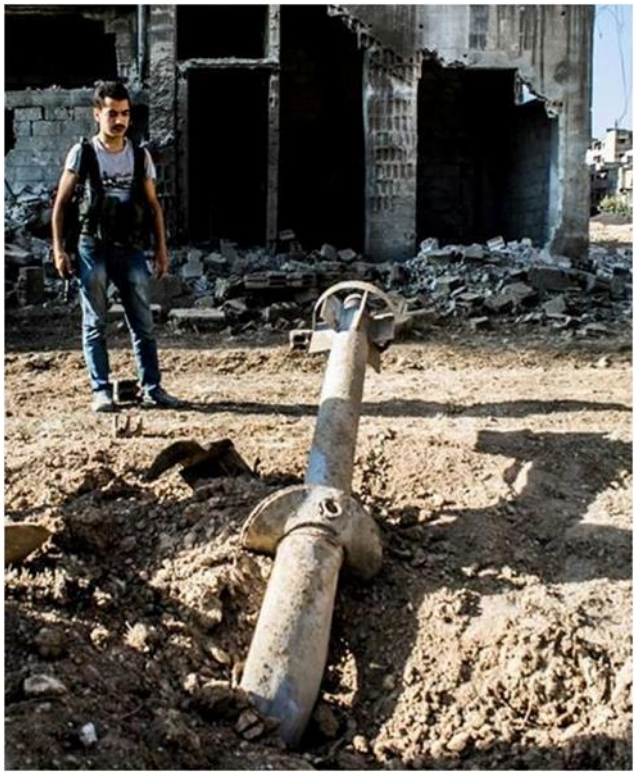
SAB Response to the Director-General's request to the SAB to provide further advice on CW sample stability and storage

Dr Christophe Curty
SAB Member
SPIEZ LABORATORY
12.10.2016

Agenda

- Background
- DG’s request
- What is known?
- SAB response to the DG’s request
- Conclusion

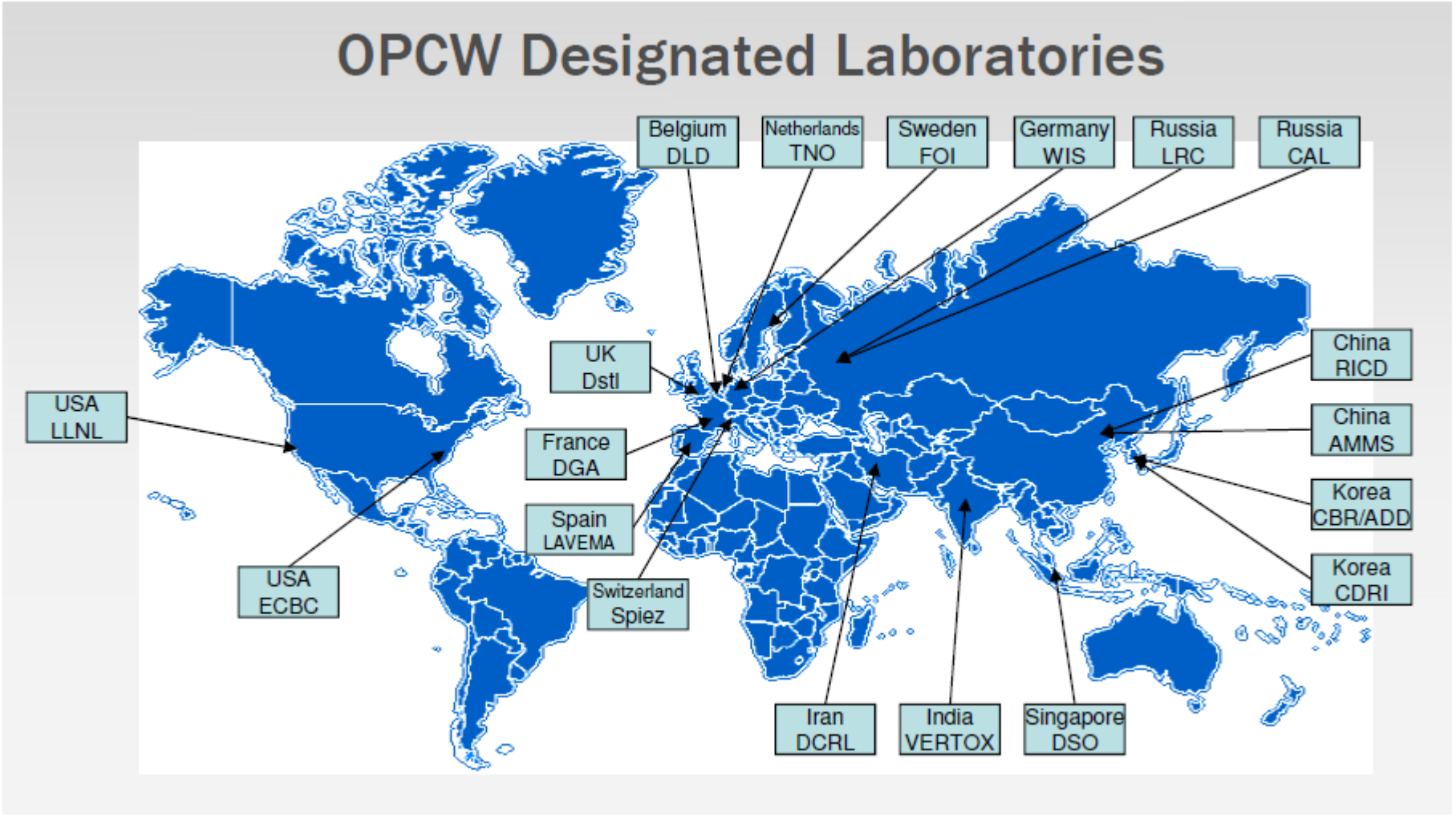
Background Facts



Background
Investigation & Sampling

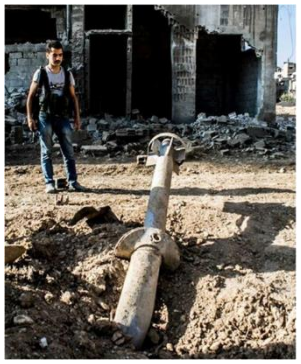
Background Samples

Background
Analysis ⟹ Results



Background
Overview: from the facts to the analysis

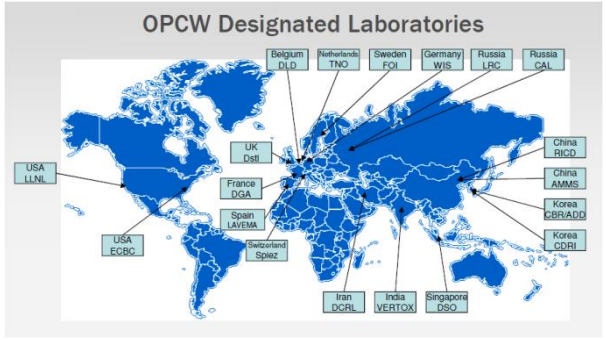
FACTS



MISSION

SAMPLES

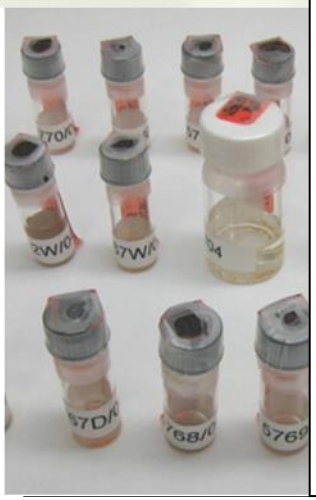
ANALYSIS



RESULTS

DG's request, 02.11.2015: Storage & Stability?

DG's request, 02.11.2015: Storage & Stability?



DIRECTOR-GENERAL'S REQUEST TO THE SCIENTIFIC ADVISORY BOARD TO PROVIDE ADVICE ON LONG-TERM STORAGE AND STABILITY OF SAMPLES COLLECTED IN RELATION TO POTENTIAL USE OF CHEMICAL WEAPONS

1. In order to be fully prepared to analyse any chemical potentially present in a wide range of types of samples in support of various operational missions, the OPCW must be able to store samples over several years and analyse those samples with high accuracy at any point in time.

2. In the context of the OPCW's investigations and fact-finding missions the Technical Secretariat has since 2013 received samples in relation to potential use of chemical weapons. These samples are stored at the OPCW Laboratory at room temperature or refrigerated at 4°C.

3. Sample types (whether current or future) – containing chemicals of interest, such as various nerve and blister agents as well as their immediate precursors and degradation products – may include in particular:

   (a) Relatively pure samples;

   (b) Liquid (including extracts) and solid samples containing either relatively high levels or trace levels of the chemicals of interest;

   (c) Highly heterogeneous unprocessed samples – such as soil, metal fragments, paint chips, fragments of highly absorbent material, or wipes – containing either relatively high levels or trace levels of the chemicals of interest; and

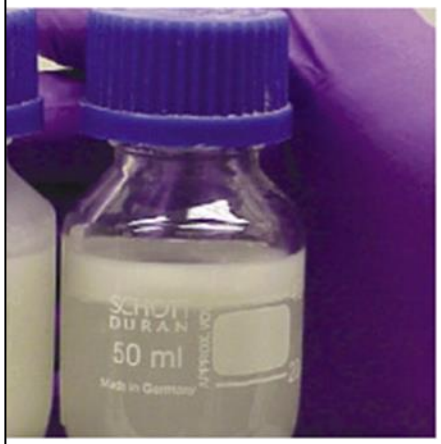
   (d) Biomedical samples: blood, plasma, urine, tissue.

4. The Director-General requests the Scientific Advisory Board (SAB) to address the following questions:

   (a) Given the current storage conditions (set out in paragraph 2), how quickly and through what process could the types of samples mentioned in paragraph 3 degrade to a point where analysis of the samples would likely no longer return credible results?

   (b) What are the best-practice conditions for long-term storage of the types of sample mentioned in paragraph 3?

   (c) Given the best-practice storage conditions set out in the SAB's answer to question (b), how quickly and through what process could the types of sample mentioned in paragraph 3 degrade to a point where analysis of the samples would likely no longer return credible results?



DG's request, 02.11.2015: Storage & Stability?

DIRECTOR-GENERAL'S REQUEST TO THE SCIENTIFIC ADVISORY BOARD TO PROVIDE ADVICE ON LONG-TERM STORAGE AND STABILITY OF SAMPLES COLLECTED IN RELATION TO POTENTIAL USE OF CHEMICAL WEAPONS

1. In order to be fully prepared to analyse any chemical potentially present in a wide range of types of samples in support of various operational missions, the OPCW must be able to store samples over several years and analyse those samples with high accuracy at any point in time.

Sample types

- Relatively pure samples
- Liquid (including extracts) and solid samples containing either relatively high levels or trace levels of the chemicals of interest
- Highly heterogeneous unprocessed samples – such as soil, metal fragments, paint chips, fragments of highly absorbent material, or wipes – containing either relatively high levels or trace levels of the chemicals of interest
- Biomedical samples: blood, plasma, urine, tissue

degrade to a point where analysis of the samples would likely no longer return credible results?

(b) What are the best-practice conditions for long-term storage of the types of sample mentioned in paragraph 3?

(c) Given the best-practice storage conditions set out in the SAB's answer to question (b), how quickly and through what process could the types of sample mentioned in paragraph 3 degrade to a point where analysis of the samples would likely no longer return credible results?

DG's request, 02.11.2015: Storage & Stability?

DIRECTOR-GENERAL'S REQUEST TO THE SCIENTIFIC ADVISORY BOARD TO PROVIDE ADVICE ON LONG-TERM STORAGE AND STABILITY OF SAMPLES COLLECTED IN RELATION TO POTENTIAL USE OF CHEMICAL WEAPONS

1. In order to be fully prepared to analyse any chemical potentially present in a wide range of types of samples in support of various operational missions, the OPCW must be able to store samples over several years and analyse those samples with high accuracy at any point in time.

Questions?

- Given the current storage conditions (set out in paragraph 2), how quickly and through what process could the types of sample mentioned in paragraph 3 degrade to a point where analysis of the samples would likely no longer return credible results?
- What are the best-practice conditions for long-term storage of the types of sample mentioned in paragraph 3?
- Given the best-practice storage conditions set out in the SAB's answer to question (b), how quickly and through what process could the types of sample mentioned in paragraph 3 degrade to a point where analysis of the samples would likely no longer return credible results?

(c) Given the best-practice storage conditions set out in the SAB's answer to question (b), how quickly and through what process could the types of sample mentioned in paragraph 3 degrade to a point where analysis of the samples would likely no longer return credible results?

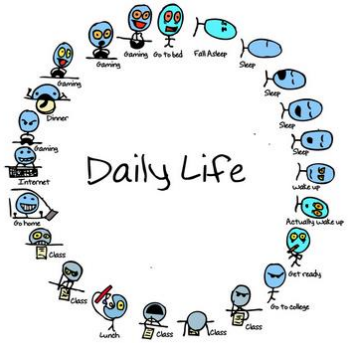
‘‘Credible results’’

Comment

- OPCW Proficiency Tests
- ISO 17025
- National Accreditation

«The SAB notes that the analytical findings of the Designated Laboratories (…) will always be scientifically accurate (…), the findings will always return ‘credible results’.»

Storage & Stability: what is known?



Degradation

Rotting

NATURAL PROCESS

Storage & Stability: what is known? Influenced by?

Storage & Stability: what is known? Influenced by?

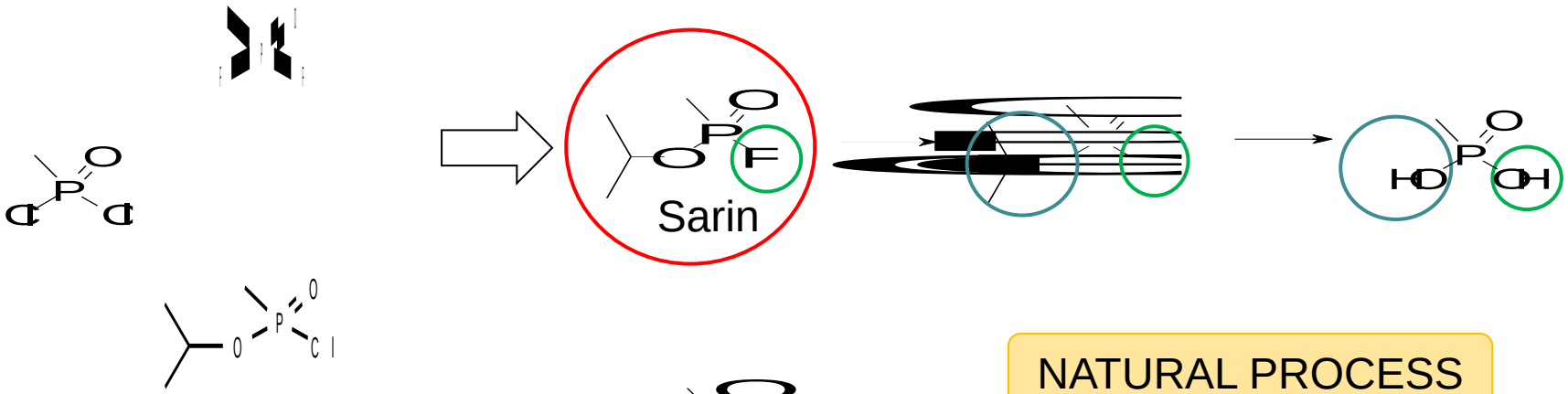
- Composition
- Temperature
- Packaging
- Atmosphere
- Duration
- Light
- Moisture
- …

Storage & Stability : what is known?
Chemistry

- Chemical
- Composition of sample
- Temperature
- Atmosphere
- Packaging
- Duration
- Light
- Moisture
- …

Storage & Stability: what is known? Chemistry

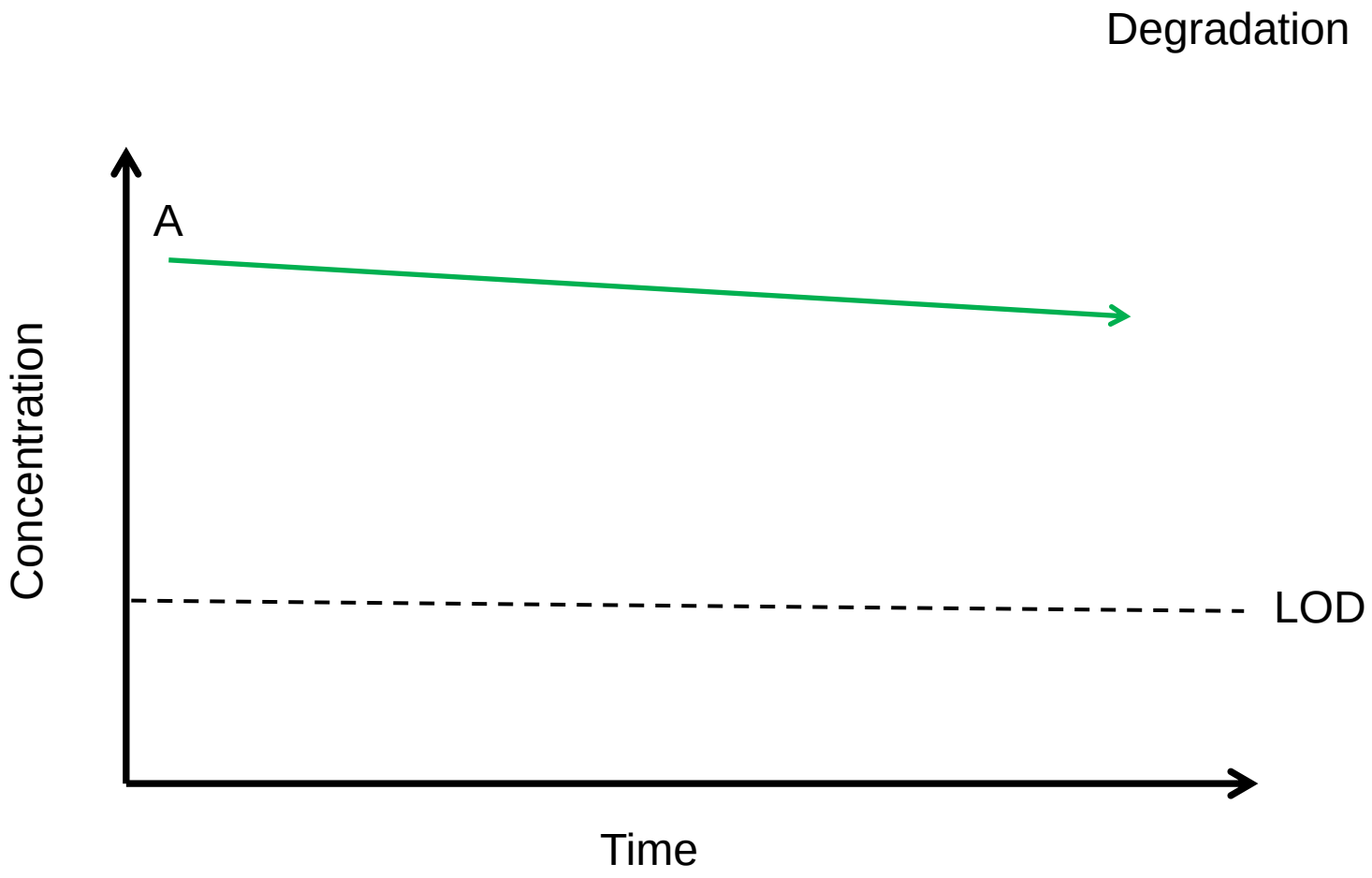
Preparation **CWA** **Degradation**



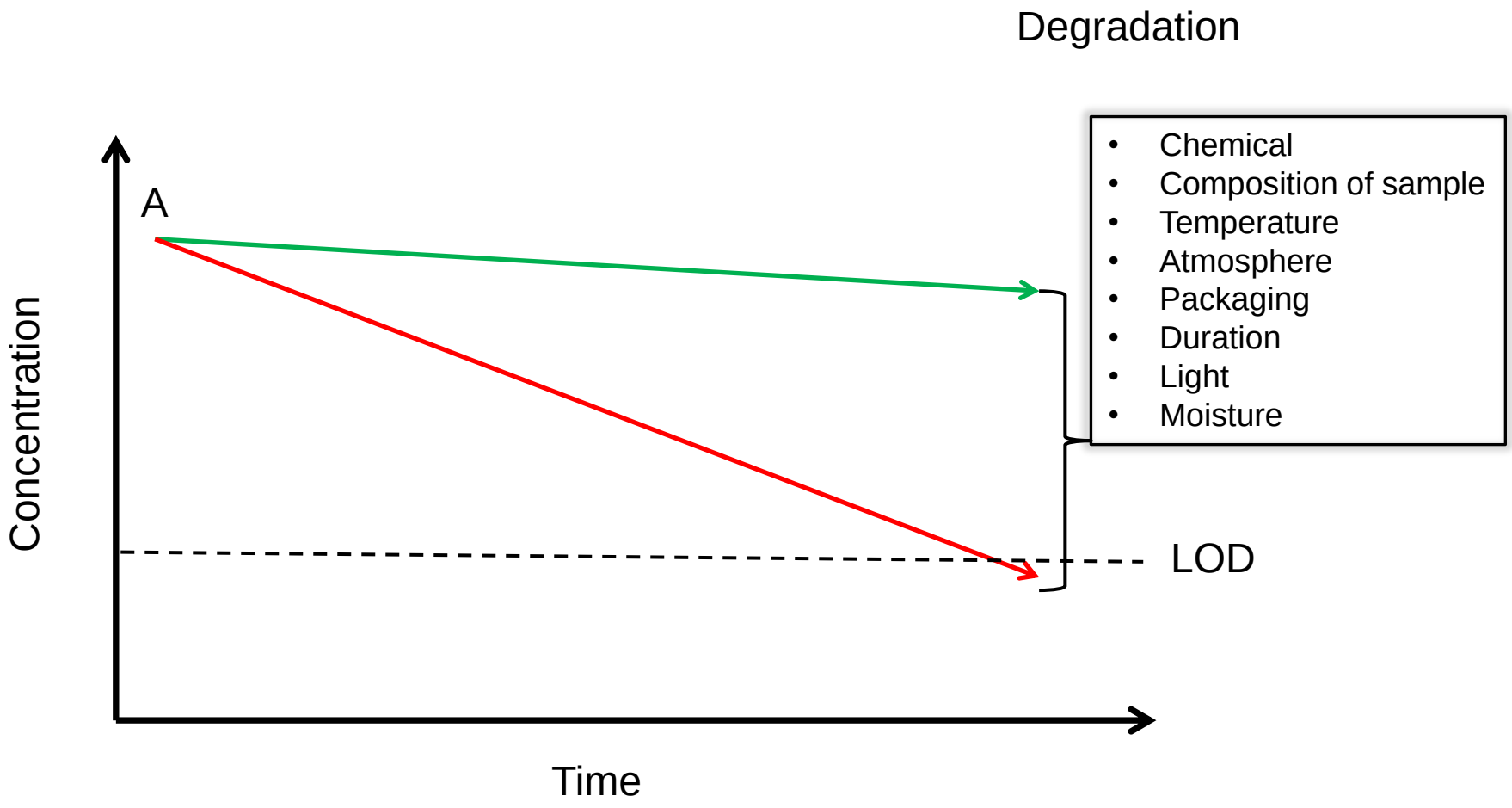
NATURAL PROCESS

By-product

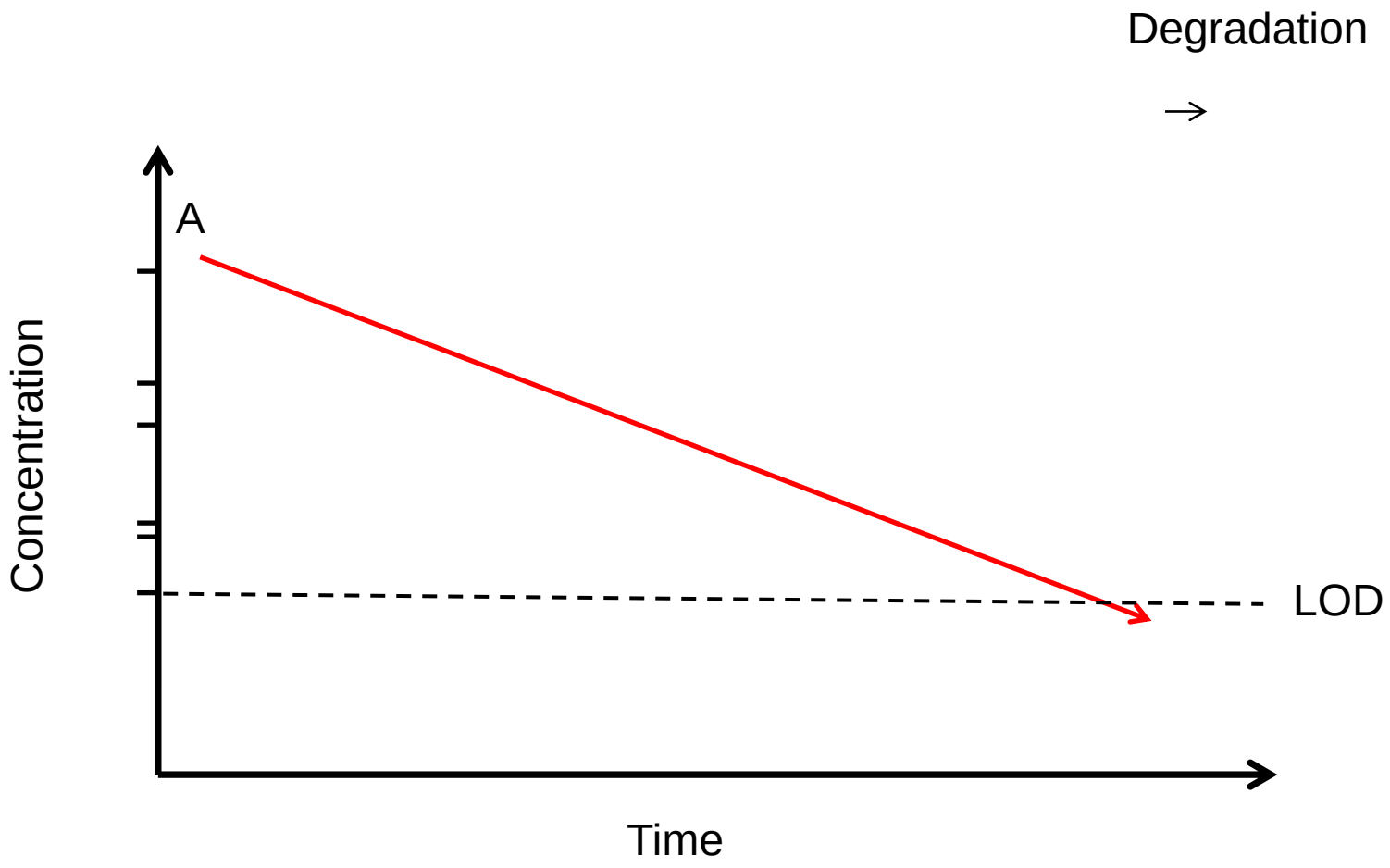
Storage & Stability: what is known? Chemistry



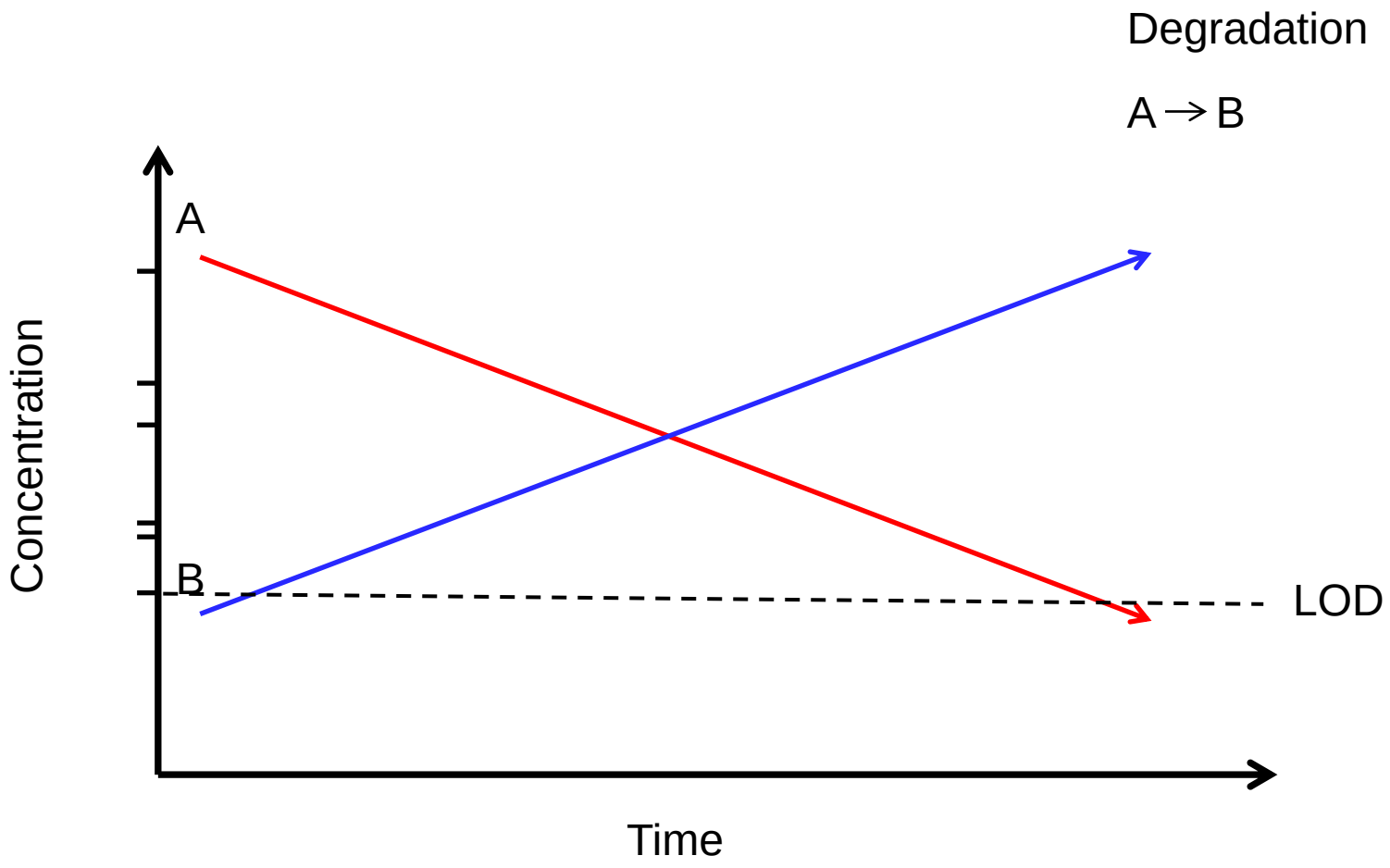
Storage & Stability: what is known? Chemistry



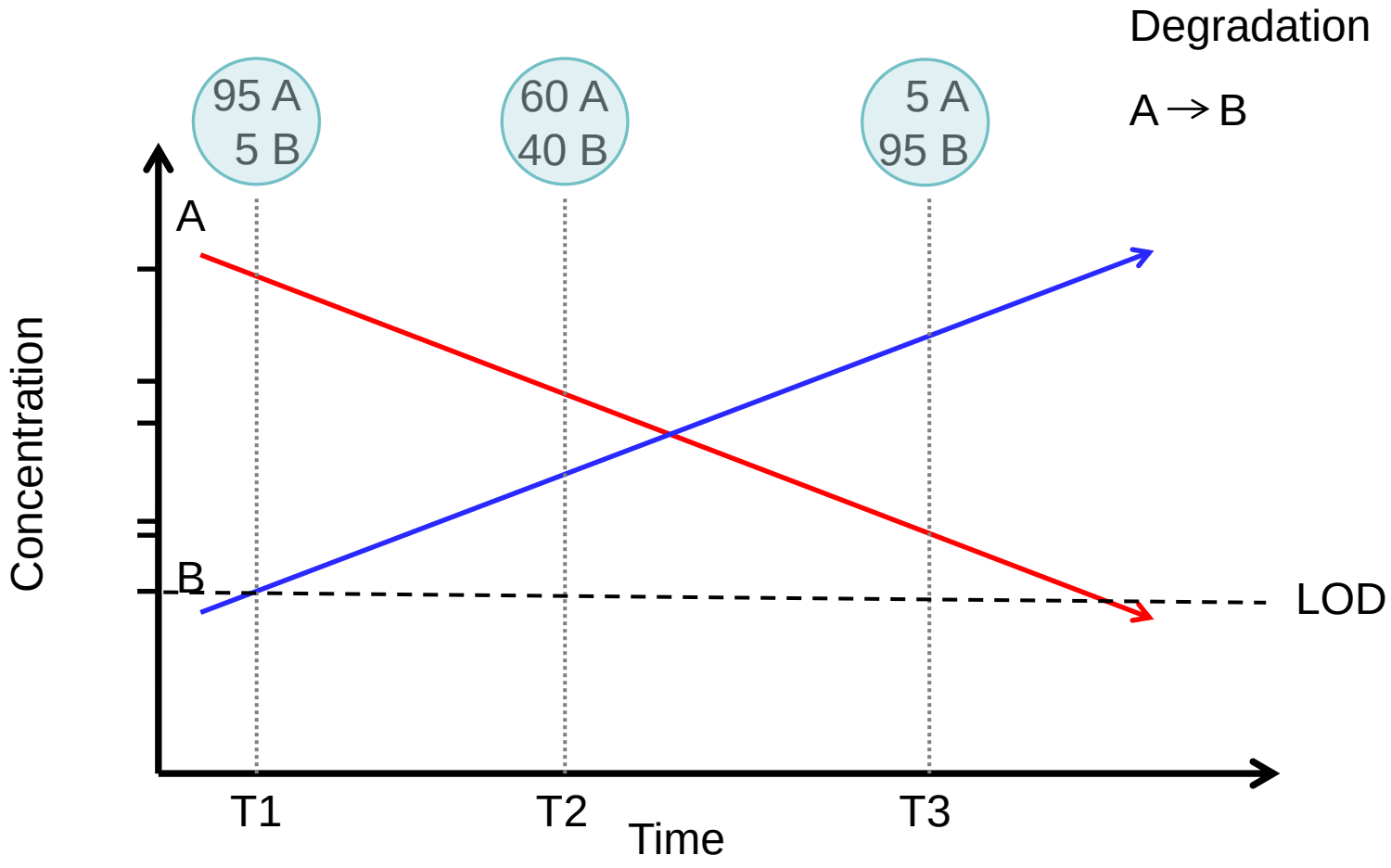
Storage & Stability: what is known? Chemistry



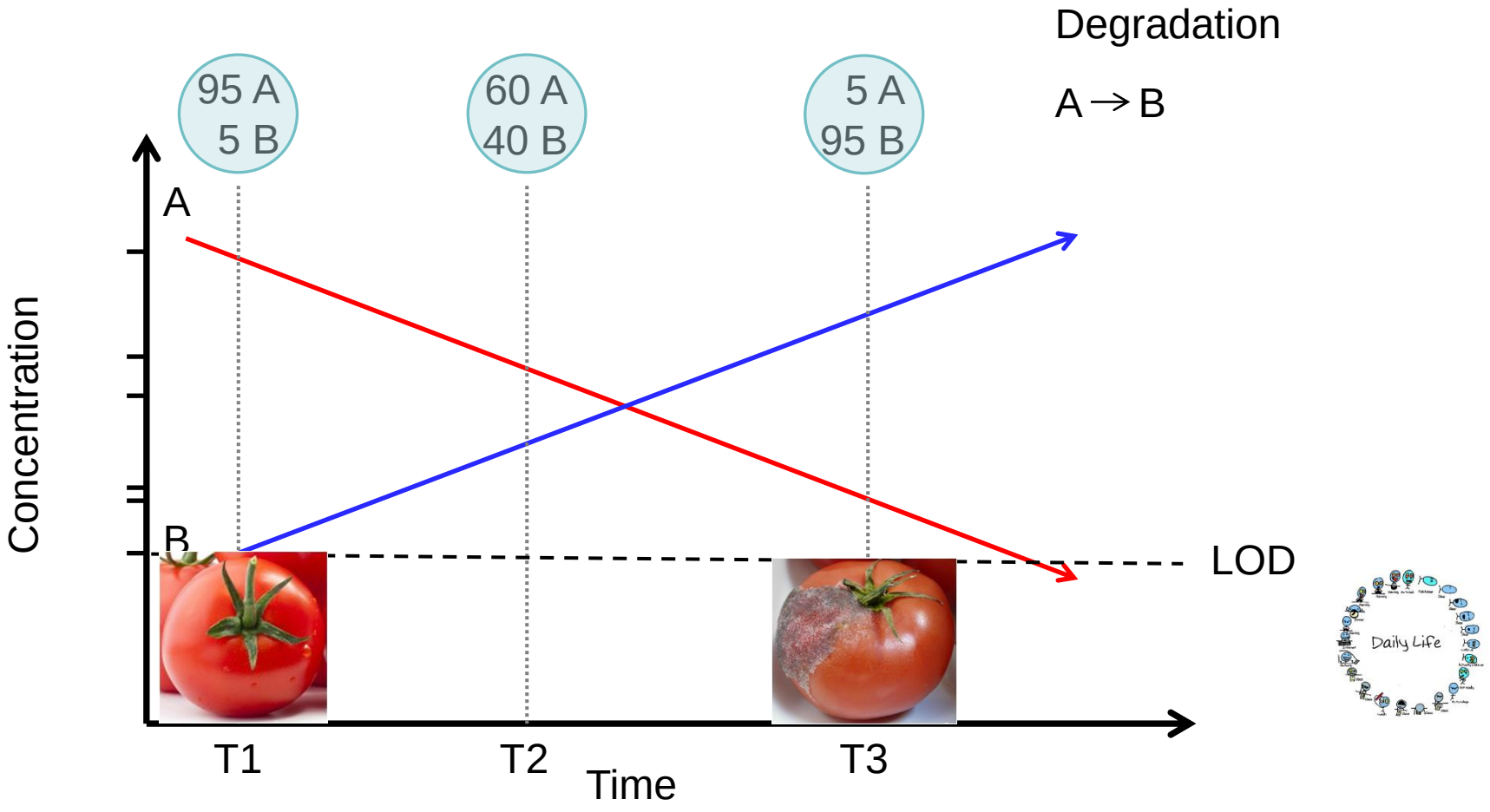
Storage & Stability: what is known? Chemistry



Storage & Stability: what is known? Chemistry



Storage & Stability: what is known? Chemistry



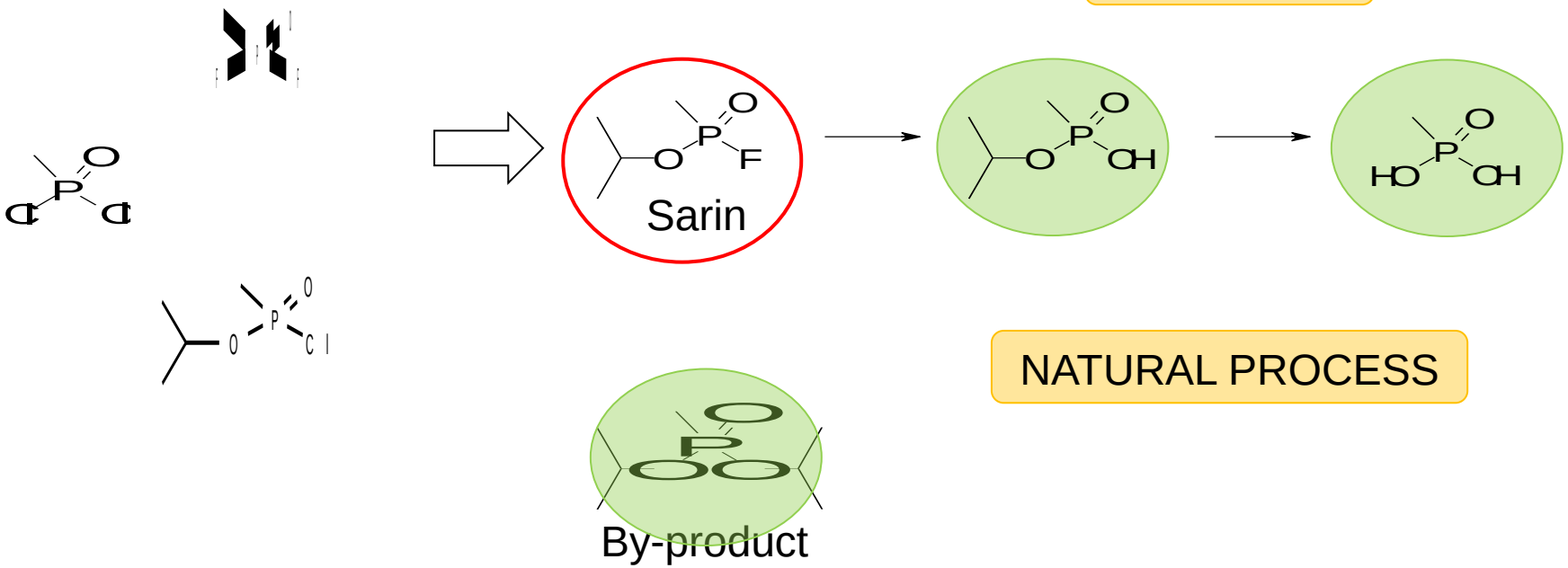
Storage & Stability: what is known? Chemistry

CWC-related chemicals

Degradation

Preparation

CWA



NATURAL PROCESS

Storage & Stability: what is known? Chemistry

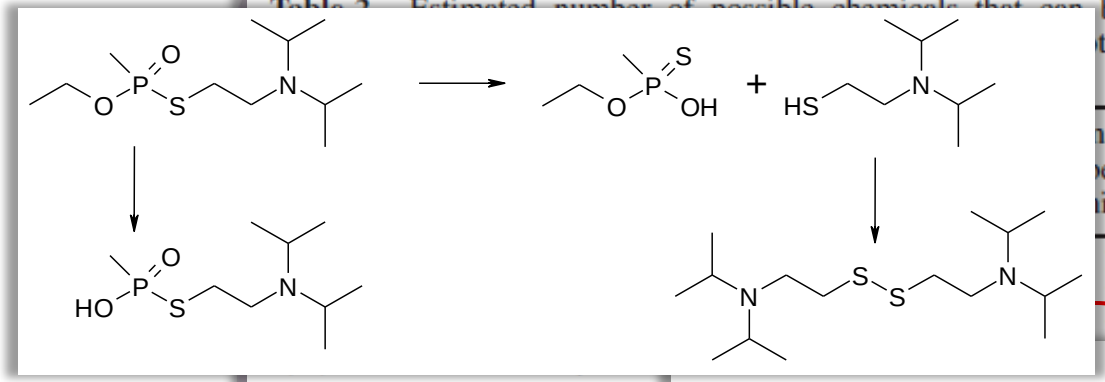
Table 2. Estimated number of possible chemicals that can be derived from the definitions for scheduled chemicals contained in the Annex on Chemicals of the CWC not counting corresponding protonated or alkylated salts, where this is applicable

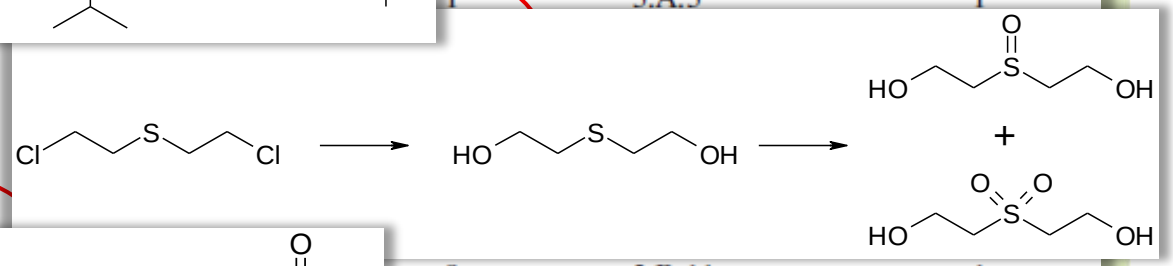
Schedule number	(Estimated) number of chemicals	Schedule number	(Estimated) number of chemicals	Schedule number	Number of chemicals
1.A.1	>20 000[a]	2.A.1	1	3.A.1	1
1.A.2	>50 000[a]	2.A.2	1	3.A.2	1
1.A.3	>200 000[a]	2.A.3	1	3.A.3	1
1.A.4	9	2.B.4	Millions	3.A.4	1
1.A.5	3	2.B.5	20[b]	3.B.5	1
1.A.6	3	2.B.6	100	3.B.6	1
1.A.7	1	2.B.7	1	3.B.7	1
1.A.8	1	2.B.8	1	3.B.8	1
1.B.9	4	2.B.9	1	3.B.9	1
1.B.10	>200 000[a]	2.B.10	10	3.B.10	1
1.B.11	1	2.B.11	8	3.B.11	1
1.B.12	1	2.B.12	10	3.B.12	1
–	–	2.B.13	1	3.B.13	1
–	–	2.B.14	1	3.B.14	1
–	–	–	–	3.B.15	1
–	–	–	–	3.B.16	1
–	–	–	–	3.B.17	1

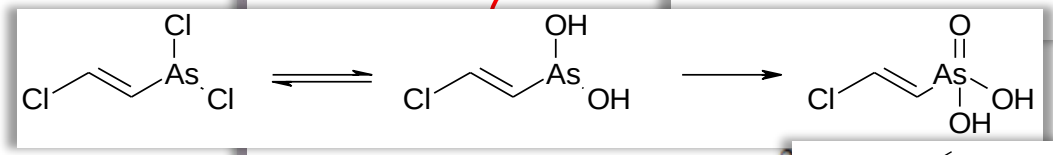
[a]Including branched chains and cyclo alkane chains, not including bicyclo alkane chains and stereoisomers and not including corresponding protonated and alkylated salts

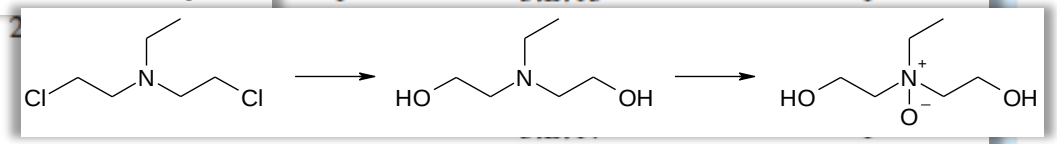
Chemical Weapons Convention Chemicals Analysis: Sample Collection, Preparation and Analytical Methods, Ed. M. Mesilaakso, John Wiley & Sons, 2005.

Storage & Stability: what is known? Chemistry









Schedule number	Number of chemicals
3.A.1	1
3.A.2	1
3.A.3	1
1.A.5	3
1.A.6	3
1.A.7	1
1.A.8	1
1.B.9	4
3.B.11	1
3.B.12	1
3.B.13	1

[a]Including branched chains and cyclo alkane chains, not including bicyclo alkane chains and stereoisomers and not including corresponding protonated and alkylated salts

Chemical Weapons Convention Chemicals Analysis: Sample Collection, Preparation and Analytical Methods, Ed. M. Mesilaakso, John Wiley & Sons, 2005.

Response to the DG's request

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Response to the DG's request

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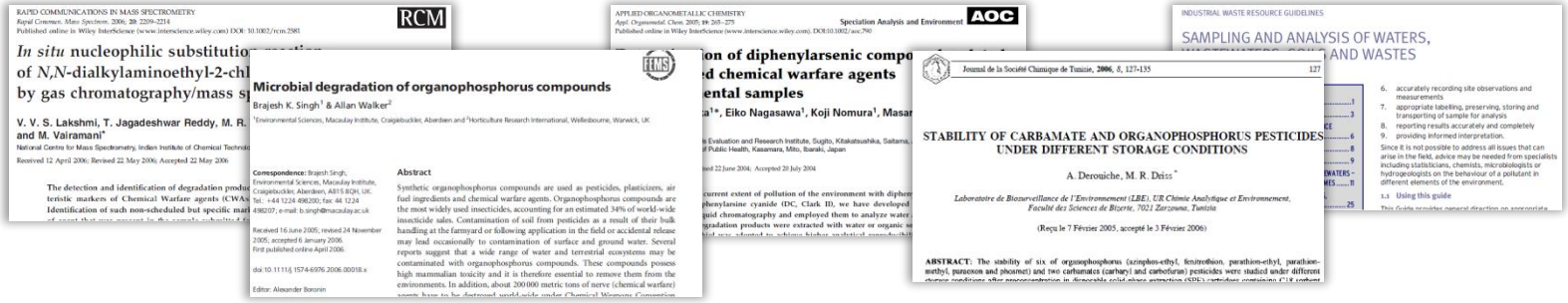
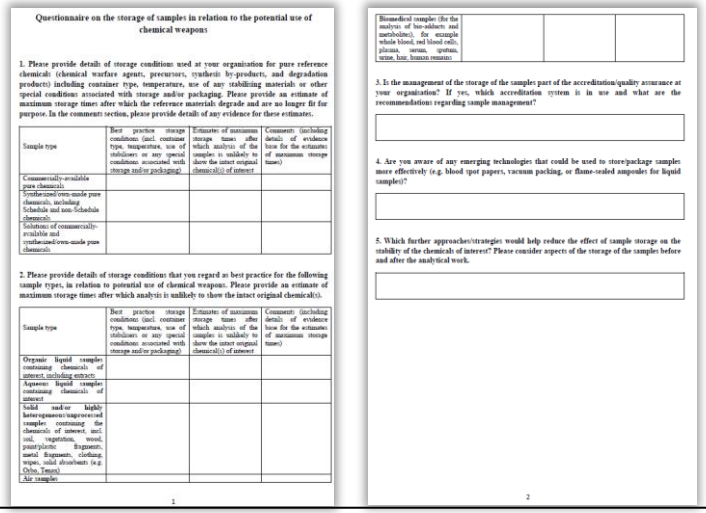
- Review of > 180 scientific papers

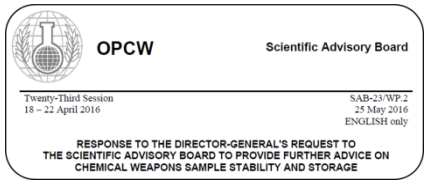


- Questionnaire to Designated Laboratories



Chemical	Type	Storage Condition	until assignment completed	manufacturer's recommendation	until signs of degradation	up to 2 weeks	2 weeks	up to 1 month	several months	up to 3 months	up to 6 months	at least 6 months	up to 1 year	several years	up to 3 years	up to 5 years	more than 10 years
Sample, organic solutions	General	refrigerator	■					■			■						
		freezer (-18°C)	■								■						
		glass container	■			■											
		original container				■					■						
		sealed															
Sample, aqueous solutions	General	refrigerator	■								■						
		glass container	■			■					■						
		original container				■											
		PP container									■						
		sealed															
Sample, solid/hetero-geneous	General	freezer (-18°C)	■								■						
		refrigerator	■			■					■						
		glass container				■					■						
		HDPE container									■						
		original container				■											
		sealed									■						
Sample, air	General	refrigerator	■														
		freezer (-18°C)	■														

Executive Summary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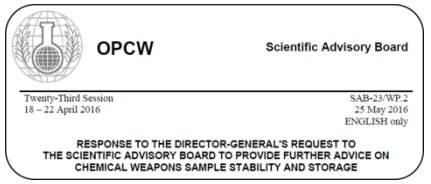
1. Given the current storage conditions in the OPCW Laboratory, how quickly and through what process could the aforementioned types of sample degrade to a point where analysis of the samples would no longer return credible results?

Storage conditions at the OPCW Laboratory

→ RT and 4°C

- Always credible analytical results
- Depends on many factors!
- Hydrolysis, oxidation and polymerisation
- Inevitable and natural (weeks to months!)
- Reduce time between collection and analysis

Executive Summary



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1. Given the current storage conditions in the OPCW Laboratory, how quickly and through what process could the aforementioned types of sample degrade to a point where analysis of the samples would no longer return credible resu[illegible]?

Process

Recommendation 1. Samples should be analysed as soon after collection possible and the need for storage eliminated or, less favourably, the storage time minimised. Prompt analysis should be viewed as urgent, as the intact original chemicals will provide the strongest basis for confirming the use of chemicals prohibited by the Chemical Weapons Convention. (This is because the sample stability, and potential impacts of any matrix or environmental factors on the stability of any CWC-relevant chemicals in the sample, will not be known prior to analysis.)

Recommendation 2. Further work on the storage of samples just after sampling and during transport to the OPCW Laboratory, sample handling during splitting, handling and storage of samples at the OPCW Laboratory, should be pursued.

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2. What are the best-practice conditions for long term-storage of the different types of sample?

Types of sample

Recommendation 3. Commercial chemical samples should be stored in gl containers with Teflon-lined caps in the dark: those in

(i) Schedules 1A01, 1A02, 1A03, 1A06, 1B09, 1B10, 1B11 and 1B12 at -18 °C under argon (to enable stability for 5-10 years).

(ii) Schedules 1A04 and 1A05 at room temperature (for stability > 10 years).

(iii) Schedule 1A08 (ricin) as a precipitate in 6 M ammonium sulfate at 4 °C (for stability > 10 years).

Recommendations 3 to 8 Type of containers, extracts, biomedical samples, splitting

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2. What are the best-practice conditions for long term-storage of the different types of sample?

Types of sample

Recommendation 3. Commercial chemical samples should be stored in gl
containers with Teflon-lined caps in the dark: those in

Recommendation 4. Extracts of chemicals should be made in dichloromethane and stored in glass containers at 4 °C with Teflon-lined caps in the dark, to ensure stability of the intact original chemical for up to one year. (Swabs or wipes should be analysed within one month of collection or otherwise disposed of due to likely storage instability; wherever possible they should be extracted as soon as possible into dichloromethane and the extracts stored instead).

(iii) Schedule 1A08 (ricin) as a precipitate in 0 M ammonium sulfate at 4 °C (for stability > 10 years).

Recommendations 3 to 8 Type of containers, extracts, biomedical samples, splitting

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2. What are the best-practice conditions for long term-storage of the different types of sample?

Types of sample

Recommendation 3. Commercial chemical samples should be stored in gl containers with Teflon-lined caps in the dark: those in

Recommendation 4. Extracts of chemicals should be made in dichloromethane and stored in glass containers at 4 °C with Teflon-lined caps

Recommendation 5. Highly heterogeneous unprocessed samples – such as soil, metal fragments, paint chips, or fragments of highly absorbent material – containing relatively high levels or trace levels of the chemicals of interest, should be stored in sealed glass or high-density polyethylene containers at -18 °C, to guarantee the stability of the samples for up to 6 months.

Recommendations 3 to 8 Type of containers, extracts, biomedical samples, splitting

Executive Summary

OPCW — Scientific Advisory Board

Twenty-Third Session
18 – 22 April 2016

SAB-23/WP.2
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Recommendation 6. Biomedical samples – for example, urine or plasma – should be stored in polypropylene or polyethylene terephthalate containers in a freezer at -80 °C (except for whole blood which should be refrigerated at 4 °C) to ensure the integrity of the samples for as long as possible (up to several years).

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Recommendation 7. Larger volumes of chemicals/samples should be split into subsamples and the subsamples used for repeated analytical manipulations. This will reduce the number of warming-cooling cycles the samples have to encounter. This is important especially for materials stored in a freezer or deep freeze (-80 °C). It will also help to minimise degradation of the chemical(s) in the unused portions of samples.

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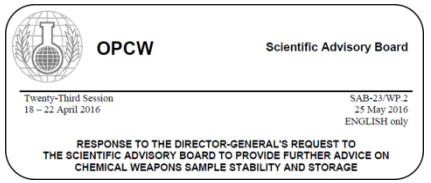
Recommendation 8. Samples of neat Scheduled chemicals required for long-term banking within the OPCW Laboratory should be flame-sealed in glass ampoules; the use of the flame-sealed ampoule technique appears to offer some storage and shipping advantages for which there is an evidence base.

Recommendations 3 to 8 Type of containers, extracts, biomedical samples, splitting

Executive Summary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3. Given these best-practice storage conditions, how quickly and through what type of process could the different types of samples degrade to a point where analysis of the samples would no longer return credible results?

- Always credible analytical results
- Inevitable and natural (weeks to months!)
- Extend "life"of original chemical
- Chemical forensics

Future work

Recommendation 9. The Technical Secretariat should monitor advances in sampling and analysis, and with the SAB, any new innovations relevant to chemical forensics.

Recommendation 10. A reference sample collection at the OPCW Laboratory should be kept to provide a range of chemical forensic options for current and future samples suspected of containing CWC-relevant chemicals.[7]

Conclusion

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Based on this review of processes by which CWC-relevant chemicals degrade, it is assessed that it is difficult, given the incomplete knowledge worldwide of the fate of CWAs in different matrices, to specify precisely when analysis of a sample 'would likely no longer identify the intact original chemicals'. Analytical results, produced under stringent quality control in OPCW Designated Laboratories, are always 'credible'. The main conundrum is how long after sample collection and storage will key markers of CWA use, or other CWC-prohibited activity, remain detectable? The passage of time will certainly lower the probability of identifying the original intact chemical(s), but the degradation products will remain detectable, proving CWA use.

ORGANISATION FOR THE PROHIBITION OF CHEMICAL WEAPONS

Working Together For a World Free of Chemical Weapons

Recommendations From The OPCW Scientific Advisory Board's Report on Verification

Recommendation 1

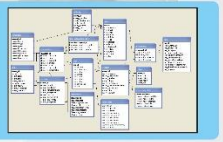
The Secretariat should consider adopting a comprehensive, more analytical approach to verification utilising all available and verifiable information.

Recommendation 2

The Secretariat should acquire the capability to use open-source information on a routine basis.

Recommendation 3

The Secretariat should put in place an information management structure that can provide the support required for the verification process.

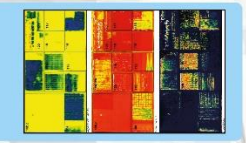


Recommendation 4

Remote/automated monitoring technologies should be added to the list of approved inspecti on equipment.

Recommendation 5

The Secretariat should look into the option of using satellite imagery for the planning of non-routine missions, in particular for IAU and CI.



Recommendation 6

The Secretariat should visit the National Authorities to obtain assurance on the accuracy and completeness of declarations. The outcome of such visits may impact on the inspection frequency.

Recommendation 7

The Secretariat must commission an independent review of all activities pertaining to the missions carried out in the Syrian Arab Republic.

Recommendation 8

The list of declarable OCPFs submitted by States Parties should include all facilities which fall under the definition/requirement of paragraph 1 of Part IX of the Verification Annex, regardless of the purity level of a DOC or DOC mixtures produced.

Recommendation 9

Not all facilities that fall under Part IX of the Verification Annex should be considered of the same relevance to the object and purpose of the Convention. The TWG recommends a practical approach for enhancing the utilisation of verification resources for OCPF declaration and on-site inspection processes.

Recommendation 10

The verification thresholds for OCPFs producing highly relevant chemicals, and the possibility of revision of the product group codes, should be addressed by the SAB as well as the industry cluster.

Recommendation 11

The OPCW should increase the staff of the OPCW Laboratory to cope with various aspects of IAU, biomedical samples, trace environmental analysis, toxins, and on-site analysis. Establishing a network of DLs for biomedical sample analysis should be a high priority.

Recommendation 12

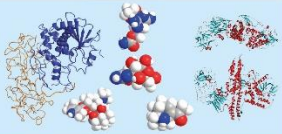
Lessons on chemical sampling and analysis from the OPCW's support to the 2013 United Nations Mission to Investigate the Use of Chemical Weapons in the Syrian Arab Republic, and all subsequent OPCW activities in relation to the Syrian Arab Republic must be identified and implemented.

Recommendation 13

PTs should incorporate a broader range of chemicals, and at a wider range of concentrations, to prepare laboratories for IAU-type scenarios.

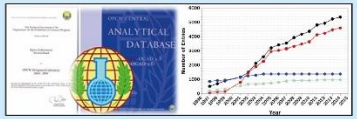
Recommendation 14

The Secretariat should expedite toxin identification exercises.



Recommendation 15

Continuous additions to the OPCW Central Analytical Database (OCAD) are recommended to allow the OPCW to meet all its mandated inspection aims, including IAU.

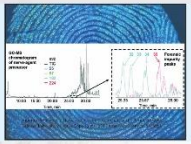


Recommendation 16

Developments in analytical instrument portability, miniaturisation and disposable biosensors should be periodically reviewed by the Secretariat and the SAB for potential applicability to on-site analysis.

Recommendation 17

The Secretariat should monitor developments in attribution analysis/chemical forensics.



Recommendation 18

The Secretariat should augment its capability to monitor and forecast developments in science and technology of relevance to the Convention and its verification regime.

https://www.opcw.org/special-sections/science-technology/science-technology-monitor/

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Report available at: https://www.opcw.org/fileadmin/OPCW/SAB/en/Final_Report_of_SAB_TWG_on_Verification_-_as_presented_to_SAB.pdf

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Report available at: https://www.opcw.org/fileadmin/OPCW/SAB/en/Final_Report_of_SAB_TWG_on_Verification_-_as_presented_to_SAB.pdf

SAB Report on Sample Stability and Storage

- A comprehensive review of scientific literature combined with best practices on storing samples relevant to chemical agents.
- Provides reference information on breakdown products of chemical warfare agents.
- No other compilation of this kind is known to be available in scientific literature.
- The Director-General has encouraged the SAB to publish the review in an appropriate scientific journal (*See EC-82/DG.13*).
- A resource for those whose work involves sampling and analysis of chemical agents