Meeting of the Ecosystem Approach Correspondence Group on Pollution Monitoring

Videoconference, 27 and 30 May 2022

Agenda Item 7: Data Standards and Data Dictionaries for IMAP Common Indicators 18 and 20

Report on Training Course for Trace Elements (2021)

IAEA

Atoms For Peace

Environment Laboratories

VIRTUAL TRAINING COURSE
ON THE ANALYTICAL TECHNIQUES FOR THE DETERMINATION OF TRACE ELEMENTS IN ENVIRONMENTAL SAMPLES

Organized by:

International Atomic Energy Agency - Environment Laboratories
4, Quai Antoine 1er - MC 98000 MONACO

6 - 10 December 2021

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Prepared in collaboration with:

UN environment
United Nations Environment Programme

MAP
Mediterranean Action Plan
Barcelona Convention

CONTENTS

TRAINING COURSE ON THE ANALYTICAL TECHNIQUES FOR THE DETERMINATION OF TRACE ELEMENTS IN ENVIRONMENTAL SAMPLES

1. Background

A virtual training course on the analysis of trace elements in marine environmental samples was organized virtually from NAEL/MESL on behalf of the UN Environment Programme/Mediterranean Action Plan (UN Environment/MAP) - Programme for the Assessment and Control of Marine Pollution in the Mediterranean Sea (MEDPOL), referred to henceforth as MEDPOL, for participants from Mediterranean laboratories involved in the MEDPOL marine pollution monitoring program in the framework of the Land-based sources (LBS) Protocol of the Barcelona Convention.

A letter describing the course content was sent out beginning of March 2021 to all MEDPOL National Focal Points, inviting them to nominate candidates for the training course from their respective countries. The selection of the six successful candidates was done jointly by MESL staff and the MEDPOL monitoring and assessment officer, by applying criteria related to i) education, ii) employment and employers' relation to the MEDPOL programme, iii) English proficiency, iv) country distribution and v) overall merit of the nominees. Invitation letters were sent to the participants by IAEA/NAEL-MESL on 18 June 2021. The selected candidates were from Israel, Lebanon, Malta, Morocco, Slovenia and Turkey.

Before the training a questionnaire was sent to participants asking their background, needs and expectations from the training. This was helpful in the preparation of a training program more relevant for the selected participants. Five out of 6 participants replied to the questionnaires, which can be found at the end of this report.

The course was held from 6 to 10 December 2021 and all selected candidates could participate.

The Training Course began with an introduction to the principles of sampling and sample preparation methodologies for sediments and biological materials. Detailed videos of sampling techniques and material were broadcasted to the participants before discussion. Introduction to the basic concepts of trace elements analysis for monitoring studies, as well as the principles of sample preparation methodology and moisture determination were presented to the participants in the training course. Lectures were dedicated on the analytical techniques (e.g. Flame Atomic Absorption Spectrometry, Graphite Furnace Atomic Absorption Spectrometry, and Inductively Coupled Plasma Mass Spectrometry as well as to the hyphenated technique (Cold Vapour Atomic Fluorescence Spectrometry-CV-AFS), applied for trace elements and mercury speciation analysis in marine samples. The most important concepts of measurement science-metrology in chemistry as validation of measurement procedure, use of certified reference materials, traceability and

uncertainty of measurement results were also presented. The exercise on the estimation of measurement uncertainty for the AAS determination of lead in sediment sample using modelling approach was developed and all tutorial materials were provided to the participants. The uncertainty on samplings, which is the dominating contributor to the total uncertainty was discussed in detail. This was a new topic for most of participants in the training course.

The practical sessions were also carried out in virtual mode via Webex. During the sessions the sample preparation procedures and the quantification of trace elements in sediments and biota samples using different techniques were demonstrated and discussed. Detailed videos and photos were used as technical support to show the practical aspects of sample preparation and analysis. All practical sessions were followed by a round-table discussion in order to answer questions from trainees and to compare proposed protocols with protocols applied in trainees' laboratories

As a virtual training, the sampling field trip for sediment and water sampling was not possible to be organized in the field. However, the sampling procedures for surface sediment (grab sampler), surface water and water profile sampling (Niskin bottle) were shown from a video to the trainees, who could appreciate how samples are collected and handled following the strictest procedures to ensure the highest quality of the final results.

2. Evaluation

The participants seemed to have appreciated the content of the training course. Almost all of them showed the required minimum level of English with the exception of one country to follow the entire training course. This has been a big improvement from other years' experience. All participants showed a lot of interest in the theoretical and practical parts and shared with us their experiences and daily challenges. All of them were interested in implementing the learned procedures in their home laboratories, if not already in place. The fact that this year the training course was held virtually has inevitably impacted the output and the participants' feedback. During the round table discussions, all participants have expressed their willingness to physically take part to real practical sessions in the laboratory.

A questionnaire was distributed to the trainees to receive feedback on the organization, content and structure of the training. Only three out of six participants filled and sent back their questionnaires. 67% of participants declared to have found the course satisfactory and to be able to better do their job after the training. They also stated that they would recommend the training course to others (100%).

In their comments they expressed their particular appreciation for the practical examples of uncertainty calculation, seawater analysis, method validation, use of CRMs and the examples on monitoring case studies. One of the applicants stressed out the fact that the analytical background was rather different from one participant to another, making the selection of the topic sometimes not relevant for all of them. A more harmonized selection could be necessary to further improve the quality of the information received. They all expressed their wish to practice in the laboratory and to apply the newly learned knowledge in practical experiences. Two out of three participants found that the workshop was too theoretical as no "real practical" laboratory experiences were possible. The questionnaires and the summary of the evaluation forms can be found at the end of this report.

3. Conclusion and Recommendations

The theoretical knowledge on the good laboratory practice, sampling, different analytical techniques for trace element analysis and quality assurance principles were presented and were beneficial to all the participants. Knowledge obtained during the training course was very well accepted from all participants, even if their theoretical background was not always at the level requested for this training. Practical sessions were also well accepted by all trainees even in this particular situation but all of them expressed the disappointment for not being able to physically participate to real practical sessions. Not all participants had the correct practical background for the training. One trainee reported to work on the monitoring of trace elements only occasionally in marine sediments, and mostly working on different types of waters. A second trainee apparently only worked on nutrient analysis until now and never on trace elements, so her involvement in the practical session was limited due to the lack of experience. Another participant did not practically perform trace element analysis yet as a dedicated instrumentation (AAS) will be soon installed in the respective laboratory. All of this means that the capacity built during the training might not directly be beneficial for the MEDPOL programme.

The insufficient level of English language was a serious obstacle for one of the trainees to follow lectures and to be fully involved in the practical sessions, and the communication with this participant was only possible in French.

Although, it was mandatory that the national laboratories nominated by focal points had participated in the 2020 MED POL PT, this criterion was not fulfilled for two participants. Thus,

despite our efforts to link both the training course and the PT, in order to have high-capacity building impact, this concept was not fully implemented by the national focal points.

<u>Recommendations</u>:

- ✓ The selection procedure for the participants in MEDPOL training course may need to be further improved and selection criteria, as provided in chapter 1, further adjusted.
- ✓ Language tests should be introduced as integral part of the selection process.
- ✓ MEDPOL focal points should only nominate candidates that are actively involved in implementation of Pollution and Marine Litter Cluster of IMAP/MEDPOL monitoring programme therefore being staff members of the laboratories responsible for IMAP implementation at national level.
- ✓ Additional efforts are needed to ensure the laboratories participating in the TCs are those taking part in PTs in order to make the most of the training received.
- ✓ MEDPOL Focal Points should follow up more closely with the nominated national laboratories participating in the implementation of MEDPOL IV/IMAP monitoring programme and experts participating in the TC for trace elements, with a view of further supporting national efforts to implement the QA/QC measures for the marine monitoring data reported to MEDPOL.

4. List of participants

PARTICIPANTS OF THE TRAINING COURSE ON THE ANALYTICAL TECHNIQUES FOR THE DETERMINATION OF TRACE ELEMENTS IN ENVIRONMENTAL SAMPLES

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5. Course outline

VIRTUAL
MED POL INORGANIC CONTAMINANTS ANALYSIS
TRAINING COURSE
6 – 10 December 2021

AGENDA
(local time = Monaco time)

DAY 1		
MONDAY 6 DECEMBER 2021		
	SESSION 1	
9:30 – 10:00	Brief welcome. Check IT platform, roll call and self-introduction of participants. Expectations from the Training Course.	*Ms Emilia Vasileva-Veleva* *Research Scientist* *Acting MESL Section Head* *All inorganic and organic participants*
10:00 – 11:00	MEDPOL Monitoring guidelines/protocols.	*Ms Jelena Knezevic* *MEDPOL programme officer*
11:00 – 11:15	BREAK	
11:15 – 12:15	Sampling principles and techniques. Samples storing, transport and pre-treatment. **Practical** : sample preparation. Dissection of biological samples (fish, mussels, oysters).	*Mr Roberto Cassi* *Laboratory Technician* *All participants in inorganic and organic courses*
11:15 – 12:15	Wrap-up way forward and discussions.	
12:15 – 14:00	BREAK / LUNCH	
	SESSION 2	
14:00 – 15:00	Trace elements determination for monitoring studies.	*Ms Emilia Vasileva* *Research Scientist* *Acting MESL Section Head*
15:00–15:15	BREAK	
15:15 – 16.30	Sample preparation for trace elements in sediments and biota sample. Mineralisation techniques and moisture determination.	*Ms Emilia Vasileva* *Research Scientist* *Acting MESL Section Head*
16.30 - 17.00	Wrap-up way forward and discussions.	

DAY 2		
TUESDAY 7 DECEMBER 2021		
SESSION 3		
9:30 – 10:30	**Practical**: Sample preparation: mineralization of biological and sediment samples for trace element analysis. Dilution of sediment and biota digests to appropriate, specified volumes.	*Ms Sabine Azemard* *Ms Anna Maria Orani* *Laboratory Technician*
10:30 – 11:00	**Practical**: Moisture determination, comparative studies on different methods.	
11:00 – 11:15	BREAK	
11:15 – 12:00	ICP-MS Spectrometry - Main principles and application for trace element analysis of Environment Samples.	*Ms Anna Maria Orani* *Laboratory Technician*
12:00 – 12:30	Wrap-up way forward and discussions.	*Ms Sabine Azemard* *Ms Anna Maria Orani* *Laboratory Technician*
12:30 – 14:00	BREAK / LUNCH	
SESSION 4		
14:00 – 15:00	**Practical**: Quantitative determination of trace elements in marine environment samples- sediments and biota. Gravimetric dilution and preparation of calibration standards. LOD, procedural blanks, Internal Standards.	*Ms Anna Maria Orani* *Laboratory Technician*
15:00–15:15	BREAK	
15:15 – 16.30	**Practical**: Determination of Cd and Pb in marine biota samples and in marine sediments with ICP-MS. Correction of interferences. Data treatment.	*Ms Anna Maria Orani* *Laboratory Technician*
16.30 - 17.00	Wrap-up way forward and discussions.	

DAY 3		
WEDNESDAY 8 DECEMBER 2021		
SESSION 5		
9:30 – 10:15	Introduction to the determination of trace elements by Atomic Absorption Spectrometry (AAS).	*Ms Emilia Vasileva* *Research Scientist* *Acting MESL Section Head*
10:15 – 11:00	Method validation and key study for method validation for trace elements in marine samples	

11:00 – 11:15	BREAK	
11:15 – 12:45	**Practical**: Flame Atomic Absorption Spectrometry and application of the method for determination of trace elements in marine samples. Preparation of calibration curve for Zn by Flame Atomic Absorption Spectrometry. Measurement conditions. Data treatment.	*Ms Sabine Azemard* *Laboratory Technician*
12:45 – 13:30	Wrap-up way forward and discussions.	*Ms Emilia Vasileva* *Research Scientist* *Acting MESL Section Head* *Ms Sabine Azemard* *Laboratory Technician*
13:30 – 14:00	BREAK / LUNCH	
	SESSION 6	
14:00 – 15:00	Proper use of CRMs. IAEA CRMs for trace elements and methyl mercury.	*Ms Emilia Vasileva* *Research Scientist* *Acting MESL Section Head*
15:00–15:15	BREAK	
15:15 – 16.30	**Practical**: PT and ILC with marine environmental samples. Interpretation of results. Corrective actions.	*Ms Sabine Azemard* *Laboratory Technician*
16.30 - 17.00	Wrap-up way forward and discussions.	*Ms Emilia Vasileva* *Research Scientist* *Acting MESL Section Head* *Ms Sabine Azemard* *Laboratory Technician*

DAY 4
THURSDAY 9 DECEMBER 2021

	SESSION 7	
9:30 – 10:15	Determination of Mercury and Methyl Mercury in marine samples.	*Ms Sabine Azemard* *Laboratory Technician*
10:15 – 11:00	**Practical**: Set up of trace element analytical run. QA/QC of data obtained. Procedural blanks, recovery factors. Data treatment. Control charts.	*Ms Sabine Azemard* *Ms Anna Maria Orani* *Laboratory Technician*
11:00 – 11:15	BREAK	
11:15 – 12:00	Determination of organic Hg by AMA. Calibration curves. Data treatment. Case study: Determination of organic Hg mass fraction in marine biota sample.	*Ms Sabine Azemard* *Laboratory Technician*

12:00 – 12:30	Wrap-up way forward and discussions.	*Ms Sabine Azemard* *Ms Anna Maria Orani* *Laboratory Technician*
12:30 – 14:00	BREAK / LUNCH	
SESSION 8		
14:00 – 15:00	**Practical**: trace element determination in seawater. Analytical challenges, different techniques, LOD, procedural blanks	*Ms Anna Maria Orani* *Laboratory Technician*
15:00–15:15	BREAK	
15:15 – 16.30	Monitoring studies for trace elements in marine environment: case studies.	*Ms Anna Maria Orani* *Laboratory Technician*
16.30 - 17.00	Wrap-up way forward and discussions.	

DAY 5
FRIDAY 10 DECEMBER 2021

SESSION 9		
9:30 – 11:00	Reliability of Measurement Results Application of metrology concepts for uncertainty and traceability of measurement results.	*Ms Emilia Vasileva-Veleva* *Research Scientist* *Acting MESL Section Head* *All inorganic and organic participants*
11:00 – 11:15	BREAK	
11:15 – 12:00	Uncertainty estimation by the "Nordtest approach".	*Ms Sabine Azemard* *Laboratory Technician*
12:00 – 12:30	Wrap-up way forward and discussions.	*Ms Emilia Vasileva* *Research Scientist* *Acting MESL Section Head* *Ms Sabine Azemard* *Laboratory Technician*
12:30 – 14:00	BREAK / LUNCH	
SESSION 10		
14:00 – 15:00	Questionnaires. Presentation of results. Course evaluation.	*All inorganic and organic participants*
15:00–15:15	Closure of the training course. Closing discussion.	

6. Evaluation of participants' questionnaire

1.   What is your overall impression of the training course?

33% Excellent   67% Satisfactory   ❐   Poor   ❐   Better than expected

2.   Do you feel that this training met your needs? (if NOT, please, explain)

33% Yes   67% To some extent ❐   Uncertain   ❐   No

3.   Do you feel that you will be better able to do your job after attending this course?

67% Yes   33% To some extent ❐   Uncertain   ❐   No

4.   Do you have a better attitude to your job having completed this course?

67% Yes   33% To some extent ❐ Uncertain   ❐   No

5.   Would you recommend that others in your field should attend this course?

100% Yes   ❐   To some extent   ❐   Uncertain   ❐   No

6.   Do you think that similar workshops with other topics would be useful?

100% Yes   ❐   No

If YES, please indicate relevant topics:
2 Trace elements by ICP-OES   1 Trace elements by ICP-MS
1 Others (specify)

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TRAINING CONTENT

7. How do you rate the balance of theoretical and practical material in the workshop?

67% Too theoretical 33% Good balance ❐   Too practical

8. How do you rate the balance of lectures, group discussions, and group exercises?

33% Good   67% Too many lectures ❐ Too many discussion sessions

9. How do you rate the training's length?

33% Too short

67% Just right   ❐   Too long

How did you
     feel about the pacing of the course?

❐   Too fast   ❐   Too slow
100% Just right

10. How do you
     rate the training's sequence?

67% Suitable   ❐   Poorly sequenced
33% Very well sequenced

11. How helpful were the group exercises?

33% Very helpful ❐   Helpful   33% Not helpful

12. Did you have enough skills practice
     time?

33% Yes   67% No   ❐   Uncertain

How valuable was the training content to your current job?

33% Very valuable   67% Of some value ❐   No real value