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Report on Training Course for Organic Contaminants (2021)

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UNEP/MAP
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**VIRTUAL TRAINING COURSE
ON THE ANALYSIS OF ORGANOCHLORINE PESTICIDES AND
POLYCHLORINATED BIPHENYLS IN ENVIRONMENTAL SAMPLES**

Organized by:

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6 - 10 December 2021

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Prepared in collaboration with:



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TRAINING COURSE ON THE ANALYSIS
OF ORGANOCHLORINE PESTICIDES
AND POLYCHLORINATED BIPHENYLS IN
ENVIRONMENTAL SAMPLES

1. Background

A virtual training course on the analysis of Organochlorinated Pesticides (OCPs) and Polychlorinated Biphenyls (PCBs) 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 from their respective countries. MESL received 10 nominations of candidates for analysis of Organochlorinated Pesticides (OCPs) and Polychlorinated Biphenyls (PCBs) in marine environmental samples. 6 candidates were selected by MESL staff in collaboration with 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 seemed appropriate. Invitation letters were sent to the participants by IAEA/NAEL-MESL on 18 June 2021. The selected candidates were from Croatia, Lebanon, Montenegro, Morocco, Slovenia and Turkey. The course took place from 6th to 10th December 2021 and all participants could participate except the one from Croatia, who has withdrawn first day of the training course.

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. Then basic concepts and terminology on persistent organic contaminants analysis were provided. Several lectures were dedicated to the high-resolution gas chromatography techniques used for organochlorinated and other organic contaminants in marine samples, and on quality assurance/quality control principles. The most important concepts of measurement science - metrology in chemistry - validation of measurement procedure, use of reference materials, and uncertainty of measurement results, were also presented and discussed.

For the practical sessions of the Training Course, the procedures for marine sample preparation and quantification of polychlorinated biphenyls and organochlorinated pesticides in sediments and biota, using gas chromatography coupled to the electron capture detector, were demonstrated combining lectures and videos.

To set a working pace that everyone could follow, the entire laboratory procedures for both sediment and biota samples were prepared before the training course and the most important phases were highlighted. Intermediate steps and corresponding intermediate samples and solutions were prepared and filmed beforehand by the trainers. During the course, the entire procedures were presented to the trainees through recorded videos to focus on the most important steps of the analytical procedures. Then, discussion among trainers and participants occurred, and trainers responded and clarified all questions raised by the participants. This approach, applied for the first time in virtual mode, was welcomed by all trainees. At the end of the course, computer sessions were provided to let trainees to practice and assess the quality of their results. Those were compared with Reference Materials assigned values and used to prepare QA/QC charts.

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 samples. Theoretical and practical sessions were also devoted to sample preparation, storage, transport and pre-treatment of the samples. Within the practical section, biological samples as fish and mussels, were used as examples to show how they should be dissected. Participants were trained on the precautions to be taken during the removal of soft tissue from the organisms to avoid contamination from dissection tools, reagents, laboratory environment and the person carrying out the procedure.

During both, theoretical lectures and practical exercises, analytical methodologies, instrument optimization, quality assurance and quality control and quantitative calculations were discussed in detail. The details on the practical part of the course are given in the Practical Session section.

2. Evaluation

The experience of participants of the 2021 MEDPOL virtual training course on the analysis of Organochlorinated Pesticides (OCPs) and Polychlorinated Biphenyls (PCBs) in marine environmental samples in the field of organic contaminant analysis varied greatly within the group of participants, and not all of them were directly involved in sediments and biota matrices or this type of contaminant analyses in their institutions. This year, since the training course was virtual, all participants were more open to participate and generate debate during the wrap-up sessions but also allowed them to be more absent from delivered lessons. Almost all of them showed the required minimum level of English to follow the entire training course without the need of translating constantly into French

or other languages. This has been a big improvement from other years' experience. All participants, with the exception of one participant who had permanent Internet connection problems, showed a lot of interest in the theoretical and practical part. Most of them, excepting one participant had enough laboratory experience to understand the different steps of the analytical procedures, including the importance for obtaining accurate results in the analysis of organochlorinated compounds and pesticides in environmental matrices (sediment and biota). All of them were interested in implementing the learned procedures in their home laboratories and were keen to find out different solutions to make it possible. Also, three of the participant's laboratories provided results in the 2020 MEDPOL PT for chlorinated compounds. Two other participants came from laboratories that were not selected to participate for organic recent MEDPOL PTs.

A questionnaire was distributed to the trainees to receive feedback on the organization, content and structure of the training. Overall, the course was rated as excellent by 100% (5/5). 60% (3/5) of participants thought that the course met their needs and another 40% (2/5) considered that to some extent, so in general they felt they will be better able to do their job after attending this course (80% replied yes and 20% to some extent). On one hand, the balance of lectures, group discussions and group exercises were found to be correct, but most participants wished to have the opportunity to practice in the laboratory to apply the newly learned knowledge "*in situ*".

3. Conclusion and Recommendations

The training course was beneficial for all trainees. During the virtual training course, each participant had a chance to observe and apply validated analytical protocols with a strict quality assurance system in place, following the Eurachem guidelines¹ and according to the ISO 17025.² Most participants acknowledged that they will have to improve or modify their laboratory procedures to reach a quality of analysis required for the MEDPOL monitoring program.

Although most participants were familiar with concepts like surrogate and internal standards, reference materials and quality assurance, they showed genuine interest and commitment to improve the quality of their work. More advanced participants took advantage of discussing specific problems with fellow trainees and MESL staff providing the training. This year, three of the laboratories' trainees participated in the 2020 MEDPOL PT. In this respect, we consider that the nomination process of this year

¹ B. Magnusson and U. Örnemark (eds) Eurachem Guide : The Fitness for Purpose of Analytical Methods -A laboratory Guide to Method Validation and Related Topics (2nd ed. 2014).

² INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, ISO/IEC 17025:2017. General requirements for the competence of testing and calibration laboratories, Geneva, (2017).

continues to show an improvement compared to the previous years where laboratories' trainees never provided data results for the PT MEDPOL exercises.

In the future the nomination process should continue to make sure that the right people from laboratories providing analysis data to the IMAP/MEDPOL monitoring programs are receiving the training. Focus should be on laboratory experience to benefit most from the capacity building efforts provided.

Based on the experience from this training course, expert missions to national designated laboratories participating in national marine environment monitoring programs for IMAP/MEDPOL IV are under preparation as to assist at laboratories with greatest needs to improve their QA/QC and data quality. Given the fact that some laboratories need to build up expertise and infrastructure to be able to provide good quality data especially for organic contaminants, this should include the identification of technical (e.g. acquisition of laboratory equipment, analytical standards, reference materials) and knowledge needs. These missions are supported by the MEDPOL Focal Points to reinforce the importance and motivation.

MEDPOL Focal Points should follow up more closely with national laboratories participating in implementation of IMAP MEDPOL IV/monitoring program and experts participating in the TC organized for organic compounds, with a view of further supporting national efforts to implement the QA/QC measures in order to warrant good quality of monitoring data reported to MEDPOL.

MEDPOL Focal Points should continue to make all possible efforts to ensure nominated participants of the TC are with adequate background and from laboratories actively participating in national marine environment monitoring programs within the implementation of IMAP/ MEDPOL IV. Similarly, additional efforts are needed to ensure the laboratories participating in TCs are those taking part in PTs in order to make the most of the training received.

4. List of participants

**PARTICIPANTS OF THE VIRTUAL TRAINING COURSE
ON THE ANALYSIS OF ORGANOCHLORINE PESTICIDES AND
POLYCHLORINATED BIPHENYLS IN ENVIRONMENTAL SAMPLES**

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5. Course outline

VIRTUAL EVENT
MED POL ORGANIC 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.	<i>Ms Emilia Vasileva-Veleva</i> <i>Research Scientist</i> <i>Acting MESL Section Head</i> <i>All inorganic and organic participants</i>
10:00 - 11:00	MEDPOL Monitoring guidelines/protocols.	<i>Ms Jelena Knezevic</i> <i>MEDPOL programme officer</i>
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).	<i>Mr Roberto Cassi</i> <i>Laboratory Technician</i> <i>All participants in inorganic and organic courses</i>
12:15 - 12:30	Wrap-up way forward and discussions.	
12:30 - 14:00	BREAK / LUNCH	

SESSION 2

14:00 - 15:00	Sources, properties and fate of organochlorinated compounds (OCs). The past, the present, and the future.	<i>Ms Imma Tolosa</i> <i>Research Scientist</i>
15:00-15:15	BREAK	
15:15 - 16.30	Analytical techniques for the determination of OCs. Extraction and clean-up methods.	<i>Ms Imma Tolosa</i> <i>Research Scientist</i>
16.30 - 17.00	Wrap-up way forward and discussions.	

DAY 2
TUESDAY 7 DECEMBER 2021

SESSION 3

9:30 - 10:30	Quantitative determination of OCs by GC-ECD.	<i>Ms Imma Tolosa Research Scientist</i>
10:30 - 11:00	Practical: Extraction of sediment and biological samples with microwave oven. Filtration of samples and blank.	<i>Mr David Huertas Laboratory Technician</i>
11:00 - 11:15	BREAK	
11:15 - 12:00	Practical: Activation of copper. Removal of sulfur on sediment samples and blank.	<i>Mr David Huertas Laboratory Technician</i>
12:00 - 12:30	Wrap-up way forward and discussions.	
12:30 - 14:00	BREAK / LUNCH	

SESSION 4

14:00 - 15:00	Practical: Sample concentration: rotatory evaporator, multi-evaporator and nitrogen stream. Solid Phase Extraction (SPE) column chromatography for sediment samples. Elution and concentration of all fractions obtained.	<i>Mr David Huertas Laboratory Technician</i>
15:00-15:15	BREAK	
15:15 - 16.30	Practical: Transfer of samples and calibrating standards in auto- injector vials. Spiking of GC internal standards. Instrumental Injection (GC-ECD).	<i>Mr David Huertas Laboratory Technician</i>
16.30 - 17.00	Wrap-up way forward and discussions.	

DAY 3
WEDNESDAY 8 DECEMBER 2021

SESSION 5

9:30 - 10:30	Confirmation analyses. Quantitative determination of OCs by GC-MS.	<i>Ms Imma Tolosa Research Scientist</i>
10:30 - 11:00	Quality assurance/quality control requirements.	
11:00 - 11:15	BREAK	

11:15 - 12:00	Practical: Determination of lipid content for biological samples. Samples clean-up using sulfuric acid.	<i>Mr David Huertas Laboratory Technician</i>
12:00 - 12:30	Wrap-up way forward and discussions.	
12:30 - 14:00	BREAK / LUNCH	

SESSION 6

14:00 - 15:00	Practical: Solid Phase Extraction (SPE) column chromatography for biological samples (F3). Elution and concentration of the third fraction. Transfer of samples and calibrating standards in auto- injector vials. Spiking of GC internal standards. Instrumental Injection (GC-ECD).	<i>Mr David Huertas Laboratory Technician</i>
15:00-15:15	BREAK	
15:15 - 16.30	Practical: Introduction to GC-ECD data retreatment software. Computer session. Data quantification of organochlorine compounds. Determination and use of limits of detection.	<i>Mr David Huertas Laboratory Technician</i>
16.30 - 17.00	Wrap-up way forward and discussions.	

DAY 4

THURSDAY 9 DECEMBER 2021

SESSION 7

9:30 - 10:30	Practical: Computer session. Evaluation of organochlorinated results on sediment samples, QA/QC of data obtained.	<i>Mr David Huertas Laboratory Technician</i>
10:30 - 11:00	Practical: Computer sessions. Quantification of biota, Recoveries.	
11:00 - 11:15	BREAK	
11:15 - 12:00	Practical: Evaluation of organochlorinated results on biota samples, QA/QC of data obtained.	<i>Mr David Huertas Laboratory Technician</i>
12:00 - 12:30	Wrap-up way forward and discussions.	
12:30 - 14:00	BREAK / LUNCH	

SESSION 8

14:00 - 15:00	High resolution gas chromatography, theory and instrumentation.	<i>Ms Imma Tolosa Research Scientist</i>
15:00-15:15	BREAK	

15:15 - 16:30	The stationary phase. Capillary columns. Sample introduction. Detectors. Temperature effects.	<i>Ms Imma Tolosa Research Scientist</i>
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.	<i>Ms Emilia Vasileva-Veleva Research Scientist Acting MESL Section Head</i> <i>All inorganic and organic participants</i>
11:00 - 11:15	BREAK	
11:15 - 12:00	Uncertainty estimation by the "Nordtest approach".	<i>Ms Imma Tolosa Research Scientist</i>
12:00 - 12:30	Wrap-up way forward and discussions.	
12:30 - 14:00	BREAK / LUNCH	

SESSION 10

14:00 - 15:00	Questionnaires. Presentation of results. Course evaluation.	<i>All inorganic and organic participants</i>
15:00-15:15	Closure of the training course. Closing discussion.	

6. Evaluation of training by participants

1. What is your overall reaction to the workshop?

[100%] Excellent Better than expected Satisfactory Poor

2. Do you feel that the workshop met your needs? (If NOT, please explain)

[60%] Yes
[40%] To some extent Uncertain No
that you will be better able to do

3.

[80%] Yes
[20%] To some extent Uncertain No

4. Do you have a better attitude about your job thanks to this course??

[60%] Yes
[20%] To some extent Uncertain [20%] No

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

[100%] Yes To some extent Uncertain No

6. In your opinion, the number of participants in the workshop was:

[60%] Just right [40%] Too few Too many

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

[100%] Yes [] No

If YES, please recommend topics:

[4] Other pesticides [2] Heavy metals [2] Others (*specify*): PBDE, Dioxins, Sea water analysis, organic and inorganic parameters

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

[20%] Too many lectures [] Too many discussions [80%] Good

9. How helpful were the group exercises?

[40%] Very helpful [60%] Helpful [] Not helpful

10. What do you think of the speed of the course?

[] Too fast [100%] Just right [] Too slow

11. Did you have enough skills practice time?

[80%] Yes [20%] No [] Uncertain

WORKSHOP CONTENT

15. How do you rate the workshop length?

[100%] Just right Too short Too long

16. What's your opinion on the workshop content sequence?

[40%] Very well sequenced [60%] Suitable Poorly sequenced

17. How valuable was the workshop content to your current job?

[80%] Very valuable [20%] Some value No real value

18. How do you rate the balance of theoretical and practical sessions?

[20%] Too theoretical [80%] Good balance Too practical

INSTRUCTIONAL MATERIAL

20. In your opinion, was the number of handouts you received sufficient?

[100%] Just right Too few Too many

21. How do you rate the quality of the handout material?

[60%] High quality [40%] Sufficient [] Below expectations

LABORATORY AND FACILITIES

22. How do you rate the laboratory sessions?

Fair Poor

[60%] Excellent [20%] Very good [20%] Good

24. Did you like the seating arrangements of the class room?

Yes

No

Uncertain

25. How do you rate the service (breaks, lunch, e.c.)?

Excellent

Very Good

Good

Fair

Poor

26. Overall evaluation of the course?

[60%] Excellent

[20%] Very good

[20%] Good

[] Fair [] Poor

Note: Questions that required comments were not reported.