REGIONAL SEAS

UNITED NATIONS ENVIRONMENT PROGRAMME

25 March 1985

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PROJET-NE PAS DIVULGUER

Determination of total mercury in marine sediments and suspended solids by cold vapour atomic absorption spectrophotometry

Reference Methods For Marine Pollution Studies No. 26

Prepared in co-operation with

IAEA

UNEP 1985

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PREFACE

The Regional Seas Programme was initiated by UNEP in 1974. Since then the Governing Council of UNEP has repeatedly endorsed a regional approach to the control of marine pollution and the management of marine and coastal resources and has requested the development of regional action plans. The Regional Seas Programme at present includes ten regions and has over 120 coastal States participating in it. (1), (2)

One of the basic components of the action plans sponsored by UNEP in the framework of the Regional Seas Programme is the assessment of the state of the marine environment and of its resources, and of the sources and trends of the pollution, and the impact of pollution on human health, marine ecosystems and amenities. In order to assist those participating in this activity and to ensure that the data obtained through this assessment can be compared on a world-wide basis and thus contribute to the Global Environment Monitoring System (GEMS) of UNEP, a set of Reference Methods and Guidelines for marine pollution studies are being developed and are recommended to be adopted by Governments participating in the Regional Seas Programme.

The methods and guidelines are prepared in co-operation with the relevant specialized bodies of the United Nations system as well as other organizations and are tested by a number of experts competent in the field relevant to the methods described.

In the description of the methods and guidelines the style used by the International Organization for Standardization (ISO) is followed as closely as possible.

The methods and guidelines, as published in UNEP's series of Reference Methods for Marine Pollution Studies, are not considered as final. They are planned to be periodically revised taking into account the development of our understanding of the problems, of analytical instrumentation and the actual need of the users. In order to facilitate these revisions the users are invited to convey their comments and suggestions to:

International Laboratory of Marine Radioactivity
International Atomic Energy Agency
c/o Musee Oceanographique
MC98000 MONACO

which is responsible for the technical co-ordination of the development, testing and intercalibration of Reference Methods.

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(1) UNEP: Achievements and planned development of the UNEP's Regional Seas Programme and comparable programmes sponsored by other bodies. UNEP Regional Seas Reports and Studies No. 1 UNEP, 1982.

(2) P. HULM: A Strategy for the Seas. The Regional Seas Programme: Past and Future UNEP, 1983.

This draft issue of the Reference Method for Marine Pollution Studies No. 26 was prepared in co-operation with the International Atomic Energy Agency (IAEA). It includes comments received from a number of scientists who reviewed and tested the method. The assistance of all those who contributed to the preparation of the draft issue of this reference method is gratefully acknowledged.

CONTENTS

1. SCOPE AND FIELD OF APPLICATION

This Reference Method describes the determination of total mercury in marine sediments and suspended solids by cold vapour atomic absorption spectrophotometry after the extraction of mercury by acid leaching. The detection limit is 2 ng of total mercury. A productivity of at least one hundred samples per day can be achieved for previously dried samples.

2. REFERENCE

RANDLESOME, J.E. and ASTON, S.R. (1980) A rapid method for the determination of mercury in sediments, suspended solids and soils. Environmental Technology Letters, 1, 3-8.

3. PRINCIPLES

An aliquot of sample is digested in nitric/hydrochloric acid mixture at 50°C in a sealed test-tube. The mercuric ion is reduced with excess stannous chloride to metallic mercury which is volatilized by aeration and the total mercury determined as monoatomic vapour by absorption at 253.7 nm wavelength.

4. REAGENTS

All reagents, including the deionised double distilled water must be as low in mercury concentration as possible. All reagents must be checked for mercury contamination by analyzing blanks.

4.1 Deionised Double Distilled Water (DDDW). Water is deionised with a mixed-bed resin and double distilled.

4.2 HNO_3/HCl acid mixture. Mix 900 ml of AnalaR (or equivalent grade) 16M HNO_3 with 100 ml AnalaR (or equivalent grade) 12M HCl in a volumetric flask (5.9).

4.3 $SnCl_2$ solution. Heat 20 g of "Low in Mercury" $SnCl_2$ (B.D.H.Ltd. or equivalent) and 10 ml of 12M AnalaR (or equivalent grade) HCl with 20 ml of DDDW in a beaker while stirring on a magnetic hot-plate. Cool and make up to 100 ml with DDDW in a volumetric flask (5.9).

4.4 $KMnO_4$ solution. Prepare 50 ml of a saturated solution of AnalaR

(or equivalent grade) $KMnO_4$ in DDDW. This reagent should be replaced in the mercury trap (5.13) at intervals of about two weeks.

4.5 Nitric acid ($d_{20}{}^{o}C$=1.4 gml^{-1}).

4.6 Mercury standard solutions.

4.6.1 Stock mercury solution: Prepare a solution containing 0.0677 g $HgCl_2$ per litre of 0.5M AnalaR HNO_3. This stock solution, which contains 50 ug Hg l^{-1} is stable indefinitely.

4.6.2 Mercury standard solution: From the stock solution (4.6.1) prepare, by appropriate dilutions using micropipettes (5.8), mercury standard solutions. Prepare these solutions at least weekly, using as dilutent 1 ml nitric acid (4.5) diluted in 250 ml (5.9) of deionised double distilled water (4.1).

4.7 Working matrix: Prepare the working matrix by homogenizing a sufficiently large sample (e.g. 300 g of fresh weight) of the same type of sediment or suspended soils which will be analyzed. Test the homogenity of the working matrix by analyzing 5 subsamples for their mercury content (6), including the digestion (6.3). If the coefficient of variation of the five analyses is less than 5% the working matrix is ready for use. Otherwise homogenize the working matrix until the above coefficient of variation is obtained or prepare a new working matrix.

5. APPARATUS

5.1 Twenty or more 60 ml pyrex test-tubes with ground glass stoppers and joints.

5.2 Clean cabinet for air drying sediment and filter samples.

5.3 Oven for drying glassware at 105°C.

5.4 Pestle and mortar for homogenizing samples.

5.5 Petri dishes to store filters and glass screw-cap bottles for sediments.

5.6 Electronic balance, preferably "top-load" variety, capable of weighing to $\pm$ 0.001 g.

5.7 Autodispensers to deliver 3 ml aliquots of reagents.

5.8 Micropipettes for standard mercury solution preparations.

5.9 Volumetric flasks, 100, 250, 1000 ml.

5.10 Aluminium heating block, thermostatically controlled, to accomodate the 60 ml test-tubes.

5.11 Seive with 63 μm screen.

5.12 Atomic absorption spectrophotometer (AAS), double beam with deuterium background corrector and mercury hollow-cathode lamp. Optimum operating conditions will depend on the actual instrument available (see manufacturer's instructions).

5.13 Cold vapour system. A cylindrical silica faced flowcell (10 cm length, 2 cm diameter) with end windows transparent to UV light is mounted on the burner head. The circuit is made of silicone rubber tubing (0.3 cm I.D.) and a peristaltic pump giving an air flow rate of 120 ml min^{-1} and a sintered glass bubbler mounted on a ground glass stopper to fit the 60 ml test tubes. The circuit is fitted with a two-way valve to allow flow to be diverted to a small Dreschel bottle. This apparatus is shown diagramatically in figure 1.

5.14 Chart recorder for AAS output.

NOTE: All glassware used for the first time must be washed with nitric acid (4.5) and several times with deionised double distilled water (4.1).

6. METHOD

6.1 Sample collection and storage

The collection, transport and storage of sediment and suspended solid samples constitute critical stages in the determination of trace metals. These stages are particularly pertinent for the analysis of mercury in sediments and suspended soils. For optimum accuracy in the determination of mercury in environmental samples, a full consideration must be given to these factors, and the final accuracy and precision of the result should be assessed from this broad point of view. Several points in connection with these pre-analysis stages must be taken into account:

a) methods of sampling;
b) container material and methods of cleaning;
c) transport to the laboratory;
d) storage before analysis.

6.1.1 Surface Sediment Samples

A 20 cm length Teflon spatula may be used as a sampling tool for accessible (e.g. intertidal) sediments, and sediments should be scraped from the top 0.5 cm over an area of approximate 10cm radius for each sample. Suitable sample containers are polyethylene screw-cap bottles precleaned with nitric acid (4.5) and rinsed with DDDW (4.1). Samples are then transported in plastic bags containing solid carbon dioxide, or at ambient temperature when the transport time is less than 30 minutes. On arrival at the laboratory the samples should be dried and analysed as soon as possible or stored at 5°C.

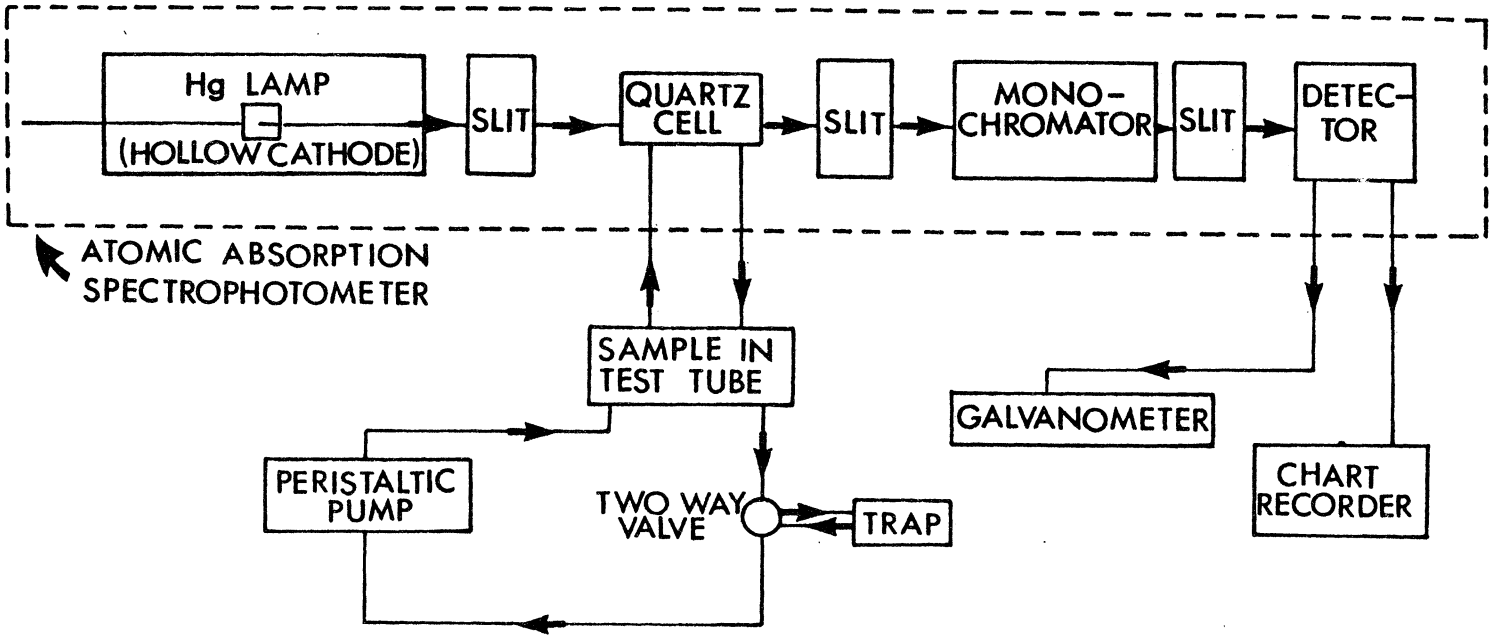

FIGURE 1: DIAGRAMMATIC REPRESENTATION OF THE COLD VAPOUR SYSTEM FOR USE IN THE DETERMINATION OF TOTAL MERCURY IN MARINE SEDIMENTS AND SUSPENDED SOLIDS.

6.1.2 Core and Grab Samples

Core samples may be collected using gravity or piston devices with P.V.C. core-liners. During transport, the cores should be sealed at their top and bottom ends using polythene bungs. Extrusion of the cores should be performed immediately on shipboard or on arrival at the laboratory after storing at 5°C.

6.1.3 Suspended Solids Samples

Suspended solids samples can be collected by filtering large volumes of sea water through prewashed and dried GF/C glassfibre filters. The volume required will vary with location but 200 mg total solids will usually be required for analysis. Short-term and longer term storage experiments have shown that there appears to be little variation in the Hg content of a suspended solids sample after a few days storage at 5°C, but dramatic increases in the Hg content of the suspended solids are often observed after several weeks.

For long-term storage of suspended solids samples, freezing the samples in plastic containers is an effective way to prevent contamination.

6.2 Sample preparation

Air dry sediment or filter samples in Petri dishes in the clean cabinet (5.2) at room temperature. This will usually take one night. Weigh the samples after drying and check constant weight before analysis. Homogenize dried sediment with a pestle and mortar. In order to normalize for variations in grain-size distributions, the dried sediment samples should be sieved through a 63 um screen (5.11) for the analysis of the silt size-fraction (< 63 um). Weigh out accuratly about 1 g of dried sediment into a test-tube or weigh a dried filter in a test-tube cleaned with concentrated nitric acid (4.5) and deionised double distilled water (4.1) and dried at 105°C (5.3).

6.3 Digestion

Add 3 ml of the HNO_3/HCl mixture (4.2) to the test-tubes using an auto-dispenser (5.7). Heat for one hour at 50°C in the aluminium heating block (5.10). Cool to room temperature and add 12 ml of DDDW from an auto-dispenser.

6.4 Mercury reduction and AAS measurement

Add 3 ml of $SnCl_2$ reagent (4.3) from an auto-dispenser (5.7) and immediately attach the test-tube directly to the sintered bubbling head of the recycling cold vapour system (5.13). Switch on the peristaltic pump and record the absorption peak height on the chart recorder.

Prior to the next analysis, direct the flow of the system using the two-way valve to allow the air flow through the Dreschel bottle containing 50 ml of permanganate solution (4.4). Continue pumping until the absorption signal returns to baseline.

For samples with widely differing concentrations, a test-tube containing DDDW (4.1) should be run in-between analyses to ensure that all the mercury vapour has been removed from the system.

6.5 Calibration and blanks

The atomic absorption instrument (5.12) should be calibrated with a series of standard solutions representing a range of 0-800 ng Hg; these are prepared by dilutions using the stock mercury solution (4.6.1, 4.6.2). Aliquots of the solution are analysed in the same manner as the samples, commencing at step 6.3. Calibration should be performed on a daily basis at least. Perform blanks (in triplicate) using the procedure from 6.3 with no sample in the test-tube.

7. EXPRESSION OF RESULTS

From the height of the absorption peaks in the calibration procedure (6.5) construct a calibration curve. Subtract any blank peak height and plot the height of peak versus ng mercury employed. Using the calibration curve, read off the weights of mercury found in the sample determinations and divide these by the known weights of samples used to calculate the final results in $\mu g\ kg^{-1}$ Hg (dry weight). An analytical report form is presented in Annex 1.

8. ESTIMATION OF PRECISION, ACCURACY AND QUALITY CONTROL

8.1 Precision

Estimate the precision of the entire analytical procedure (6 through 7) by digesting ten samples of the working matrix (4.7) and analyzing them separately. Calculate the standard deviation (S) and the coefficient of variation (CV) where CV=S.100/mean value. If the CV is greater than 5% check the whole procedure for possible errors and/or contamination. The precision of the method is normally better than $\pm$ 5% at the 95% confidence level.

8.2 Accuracy

Using this Reference Method, analyze a certified standard, with a matrix similar to the material under study, together with your own working matrix chosen from among your samples (4.7). Calculate the mean and the standard deviation for the certified standard and the working matrix. If the value given for the certified standard is within the interval of your mean $\pm$ standard deviation, your method has the required accuracy and the working matrix can be used as a standard for checking the accuracy of your procedure. If not, check the whole procedure for errors.

NOTE: In addition, by participating in intercalibration exercises involving several analytical laboratories, the accuracy of the method as used by the analyst can be checked and compared with the accuracy obtained by other participants in the exercise.

8.3 Quality Control

Analyse periodically, at least once a week or whenever the routine has been interrupted for more than a week, the working matrix, in order to guarantee the precision and accuracy of your results. If, on any occasion, the calibration curve is found to be altered by more than 5% from the previous ones, check your routine for possible errors.

9. ANALYTICAL REPORT

Fill in the Analytical report (table 1) giving full details in every column.

Table 1: Analytical Report on Total Mercury Concentration in Sediment and Suspended Solids

1. Sample code:_____

2. Determination of dry weight in oven:_____

   2.1 Duration of drying:_____hours

   2.2 Date of drying:_____day;_____month;_____year

3. Digestion

   3.1 Duration of digestion:_____hours

   3.2 Temperature used for digestion:_____^{o}C

   3.3 Date of digestion:_____day;_____month;_____year

   3.4 Anomalies observed which may influence results:

_____

_____

_____

4. Standardization (calibration)

   4.1 Date:_____day;_____month;_____year

   4.2 Result:

digestion vessel	1	2	3	4	5	6	7	8
								(blanks)
added stand.sol.(ml)	-	0.1	0.2	0.3	0.4	-	-	-

mass of Hg (ng)

---

units of recorded signal

µg Hg kg^{-1}

---

5. Analytical result and estimation of precision using subsamples of same sample.

5.1 Date:_____day;_____month;_____year

5.2 Result:

digestion vessel	1	2	3	4	5	6	7	8	9	10
sample mass (g)										
units of recorded signal										
µg Hg kg^{-1}										

mean_____µg Hg kg^{-1}; Stand. deviation_____

coeff. of variation_____%

6. Estimation of accuracy

6.1 Date:_____day;_____month;_____year

6.2 Type of certified standard used:_____

6.3 Declared µg Hg kg^{-1} certified standard:_____

6.4 Results:

digestion vessel	1	2	3	4	5	6	7	8
mass certified standard (g)					-	-	-	-
mass working matrix (g)	-	-	-	-				
units of recorded signal								
µg Hg kg^{-1}								

mean_____µg Hg kg^{-1}; Stand. deviation_____

coeff. of variation_____%

certified standard identity

working matrix identity

7. Anomalies observed during analysis and other remarks relevant to the interpretation of results:

8. Intercalibration exercise (give details):_____

9. Full address of the institution which carried out the analysis:

_____

_____

_____

10. Name(s) and signature(s) of the person(s) who carried out the analysis:

_____

_____

_____

Date: _____

LIST OF REFERENCE METHODS FOR MARINE POLLUTION STUDIES

LISTE DES METHODES DE REFERENCE POUR LES ETUDES DE POLLUTION MARINE

UNEP/WHO : Guidelines for monitoring the quality of coastal recreational and shellfish-growing waters. (Draft) Reference Methods for Marine Pollution Studies No. 1, UNEP 1984.

UNEP/WHO : Determination of total coliforms in sea-water by the membrane filtration culture method. Reference Methods for Marine Pollution Studies No. 2 Rev. 1, UNEP 1983.

PNUE/OMS : Détermination des coliformes totaux dans l'eau de mer par la méthode de culture sur membranes filtrantes. Méthodes de Références pour les Etudes de Pollution Marine No 2, Rév. 1, PNUE 1983.

UNEP/WHO : Determination of faecal coliforms in sea-water by the membrane filtration culture method. Reference Methods for Marine Pollution Studies No. 3 Rev. 1, UNEP 1983.

PNUE/OMS : Détermination des coliformes fécaux dans l'eau de mer par la méthode de culture sur membranes filtrantes. Méthodes de Références pour les Etudes de Pollution Marine No 3, Rév. 1, PNUE 1983.

UNEP/WHO : Determination of faecal streptococci in sea-water by the membrane filtration culture method. Reference Methods for Marine Pollution Studies No. 4 Rev. 1, UNEP 1983.

PNUE/OMS : Détermination des streptocoques fécaux dans l'eau de mer par la méthode de culture sur membranes filtrantes. Méthodes de Références pour les Etudes de Pollution Marine No 4, Rév. 1, PNUE 1983.

UNEP/WHO : Determination of faecal coliforms in bivalves by multiple test tube method. Reference Methods for Marine Pollution Studies No. 5 Rev. 1, UNEP 1983.

PNUE/OMS : Détermination des coliformes fécaux dans les bivalves par le test des tubes multiples. Méthodes de Références pour les Etudes de Pollution Marine No 5, Rév. 1, PNUE 1983.

UNEP/FAO/IAEA : Guidelines for monitoring chemical contaminants in marine organisms. Reference Methods for Marine Pollution Studies No. 6, UNEP. (in preparation)

UNEP/FAO/IOC/ IAEA : Sampling of selected marine organisms and sample preparation for trace metal analysis. Reference Methods for Marine Pollution Studies No. 7 Rev. 2, UNEP 1984.

UNEP/FAO/IOC/ IAEA : Determination of total mercury in selected marine organisms by cold vapour atomic absorption spectrophotometry. Reference Methods for Marine Pollution Studies No. 8 Rev. 1, UNEP 1984.

UNEP/FAO/IAEA : Determination of total arsenic in selected marine organisms by hydride generation atomic absorption spectrophotometry. Reference Methods for Marine Pollution Studies No. 9, UNEP 1985.

UNEP/FAO/IAEA : Determination of total selenium in selected marine organisms by hydride generation atomic absorption spectrophotometry. Reference Methods for Marine Pollution Studies No. 10, UNEP 1984.

UNEP/FAO/IOC/ IAEA : Determination of total cadmium, zinc, lead and copper in selected marine organisms by flameless atomic absorption spectrophotometry. Reference Methods for Marine Pollution Studies No. 11 Rev. 1, UNEP 1984.

UNEP/FAO/IAEA : Sampling of selected marine organisms and sample preparation for the analysis of chlorinated hydrocarbons. Reference Methods for Marine Pollution Studies No. 12 Rev. 1, UNEP 1984.

UNEP/FAO/IAEA : Determination of methylmercury in selected marine organisms by gas chromatography. Reference Methods for Marine Pollution Studies No. 13, UNEP 1984.

UNEP/FAO/IOC/ IAEA : Determination of DDTs and PCBs in selected marine organisms by packed column gas chromatography. Reference Methods for Marine Pollution Studies No. 14 Rev. 1, UNEP 1985.

UNEP/IOC/IAEA : Monitoring of tar on marine beaches. Reference Methods for Marine Pollution Studies No. 15, UNEP 1985.

UNEP/IAEA : Determination of DDTs, PCBs, PCCs and other hydrocarbons in sea-water by gas chromatography. (Draft) Reference Methods for Marine Pollution Studies No. 16, UNEP 1982.

UNEP/IAEA : Determination of DDTs, PCBs and other hydrocarbons in marine sediments by gas-liquid chromatography. (Draft) Reference Methods for Marine Pollution Studies No. 17, UNEP 1982.

UNEP/IOC : Determination of total dissolved cadmium in sea-water by differential pulse anodic stripping voltammetry. (Draft) Reference Methods for Marine Pollution Studies No. 18, UNEP 1983.

UNEP/IOC/IAEA : Determination of mercury in estuarine waters and suspended sediment by cold vapour atomic absorption spectrophotometry. Reference Methods for Marine Pollution Studies No. 19, UNEP 1985.

UNEP/IOC/IAEA : Monitoring of petroleum hydrocarbons in sediments. Reference Methods for Marine Pollution Studies No. 20, UNEP. (in preparation)

UNEP/WHO/IAEA : Determination of total coliforms in sea-water by multiple test tube (MPN) method. Reference Methods for Marine Pollution Studies No. 21, UNEP 1985.

UNEP/WHO/IAEA : Determination of faecal coliforms in sea-water by multiple test tube (MPN) method. Reference Methods for Marine Pollution Studies No. 22, UNEP 1985.

UNEP/WHO/IAEA : Determination of faecal streptococci in sea-water by multiple test tube (MPN) method. Reference Methods for Marine Pollution Studies No. 23, UNEP 1985.

UNEP/WMO/IAEA : Sampling of aerosols and wet precipitation for analysis of chemical pollutants. Reference Methods for Marine Pollution Studies No. 24, UNEP 1985.

SPC/UNEP : Coral reef monitoring handbook. Reference Methods for Marine Pollution Studies No. 25, UNEP 1984.

UNEP/IAEA : Determination of total mercury in marine sediments and suspended solids by cold vapour atomic absorption spectrophotometry. Reference Methods for Marine Pollution Studies No. 26, UNEP 1985.

UNEP/IAEA : Determination of total cadmium in marine sediments by flameless atomic absorption spectrophotometry. Reference Methods for Marine Pollution Studies No. 27, UNEP 1985.

UNEP : Sampling and identification of common Mediterranean Scyphomedusae and evaluation of their occurrence. (in preparation)

UNEP/IOC/IAEA : Monitoring of petroleum hydrocarbons in sea-water. (in preparation)

UNEP/IAEA : Guidelines for monitoring of estuarine waters and suspended matter. (in preparation)

UNEP/WHO/IAEA : Determination of faecal coliforms in estuarine waters, suspended matter and sediments. (in preparation)

UNEP/WHO/IAEA : Determination of phosphorus in suspended matter and sediments. (in preparation)

UNEP/WHO/IAEA : Determination of nitrogen in suspended matter and sediments. (in preparation)

UNEP/WHO/IAEA : Determination of BOD_5 and COD in estuarine waters. (in preparation)

UNEP/FAO/IAEA : Acute toxicity tests. (in preparation)

UNEP/IOC/IAEA : Determination of total cadmium in estuarine waters and suspended matter. (in preparation)

UNEP/FAO/IAEA : Biological non-acute toxicity tests. (in preparation)

UNEP/IOC/IAEA : Determination of basic oceanographic and meteorological conditions. (in preparation)

UNEP/IOC/IAEA : Determination of standard physical and chemical parameters. (in preparation)

UNEP/WHO/IAEA : Statistical methods for the evaluation of results from monitoring the quality of coastal recreational and shellfish-growing waters. (in preparation)

UNEP/FAO/IOC/IAEA : Determination of DDTs and PCBs in selected marine organisms by capillary column gas chromatography. (in preparation)

UNEP/IAEA : Determination of selected trace metals in aerosol and in wet precipitation. (in preparation)

UNEP/IAEA : Determination of halogenated hydrocarbons in aerosol and in wet precipitation. (in preparation)

UNEP/WMO/IAEA : Sampling of dry deposition. (in preparation)

UNEP/WHO/IAEA : Determination of staphylococcus aureus in sea-water and sewage by the membrane filtration culture method. (in preparation)

UNEP/WHO/IAEA : Determination of pseudomonas aeruginosa in sea-water and sewage by the membrane filtration culture method. (in preparation)

UNEP/WHO/IAEA : Isolation/Enumeration of salmonella from sea-water and sewage. (in preparation)

UNEP/WHO/IAEA : Determination of methylmercury, total mercury and selenium in human hair. (in preparation)

UNEP/WHO/IAEA : Guidelines for monitoring and epidemiological studies on health effects of methylmercury. (in preparation)

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