



## Transnationally harmonized sediment laboratory analysis protocol for HSs in DRB's surface waters proposal

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## 1. Introduction

The most used method for determination of the heavy metals is Inductively-coupled plasma mass spectrometry (ICP-MS) and for the organic substances are Liquid chromatography and/or gas chromatography, linked to mass spectrometry (LC-MS and GC-MS).

Generally used method should satisfy the criteria recommended by WFD for HSs: they should be determined according to the ISO norms, have a limit of quantification (LOQ)  $\leq$  Environmental Quality Standard (EQS) and a measurement uncertainty  $\leq$  50 %.

Organic compounds are selected in the sediment according to their physico-chemical propensity for the solid phase. The more hydrophobic (water repelling) a compound is the less soluble it is in water, and therefore more likely to adsorb to sediment particles.

The octanol-water partition coefficient ( $K_{ow}$ ) is used as a measure of the hydrophobicity of the organic compound.

Compounds measured in sediment or suspended particulate matter (SPM) with  $\log K_{ow} > 5$ , and compounds with  $\log K_{ow} < 3$  should be measured in water.

Compounds with a  $\log K_{ow}$  between 3 and 5 will depend on the degree of contamination. If the degree of contamination for a hydrophobic compound is unknown or expected to be low, sediment should be an additional monitoring matrix (due to accumulation).

## 2. Selected HSs

Under the European Water Framework Directive (WFD) was developed the Environmental Quality Standards Directive 2008/105/EC in which were proposed hazardous substances for monitoring in sediment. The Directive 2013/39/EU amending the Directive 2008/105/EC and has proposed new HSs for monitoring and has changed some existing HSs. The ISO and/or EPA standards for chemical analytical methods for HSs are described in paragraphs 2.1. to 2.20. For some substances with a lack of standards were given literature where their analytical methods were described.

### 2.1. Anthracene

Anthracene is polycyclic aromatic hydrocarbons (PAH).

Anthracene is consisting of three benzene rings derived from coal-tar. It is primarily used as an intermediate in the production of dyes and smoke screens. It is ubiquitous in the environment as a product of incomplete combustion of fossil fuels. It has been identified in surface and drinking water, ambient air, exhaust emissions, smoke of cigarettes and cigars, and in smoked foods and edible aquatic organisms.

ISO 17993. 2002. Water quality - Determination of 15 polycyclic aromatic hydrocarbons (PAH) in water by HPLC with fluorescence detection after liquid-liquid extraction.

EPA method 8100. September 1986. Polynuclear Aromatic Hydrocarbons.

## **2.2. Brominated diphenylethers**

Polybrominated diphenyl ethers or PBDEs are organobromine compounds used as a fire retardant. People are exposed to low levels of PBDEs by ingestion of food and by inhalation. PBDE bioaccumulates in blood, breast milk and adipose tissue. People are also exposed to these chemicals in their home environment because of their prevalence in common household items.

ISO 22032. 2006. Water quality - Determination of selected polybrominated diphenylethers (PBDE) in sediment and sewage sludge - Method using extraction and gas chromatography/mass spectrometry.

EPA Method 1614. August 2007. Brominated Diphenyl Ethers in water soil, sediment and tissue by HRGC/HRMS. EPA-821-R-07-005. U.S. Environmental Protection Agency, Office of Water, Washington, DC, USA.

## **2.3. Cadmium and its compounds**

Cadmium is a trace element and transitional metal. It is toxic in moderate doses. It is used in the manufacture of batteries, electrical conductors and metal plating. Cadmium is also a byproduct of the mining and processing of iron, nickel and other metals and can be toxic to welders and industrial workers, producing a syndrome due to inhalation of excessive amounts known as cadmium fume fever. An increased level of cadmium has been observed in the environment due to contamination of the water supply from mining.

ISO 5961:1994 Water quality -- Determination of cadmium by atomic absorption spectrometry (reviewed and confirmed in 2015).

## **2.4. C10-13-chloroalkanes**

These compounds are also known as short-chain polychlorinated paraffins (SCCPs).

C<sub>10-13</sub> -chloroalkanes are UVCB substances (substances of unknown or variable composition) with varying chlorine content (up to about 70% by weight) and carbon chain lengths (between C10 and C13). They are mainly released into the water as a result of runaway emissions during production and use as a metal fluid. Accidental discharges during transport and storage also resulted in environmental pollution. C<sub>10-13</sub> -

chloroalkanes have low water solubility (between 0.15 and 0.47 mg / l) and a strong tendency for adsorption to organic matter and soil.

ISO 12010:2019 Water quality -- Determination of short-chain polychlorinated alkanes (SCCP) in water -- Method using gas chromatography-mass spectrometry (GC-MS) and negative-ion chemical ionization (NCI)

### **2.5. Di (2-ethylhexyl)phthalate (DEHP)**

Di(2-ethylhexyl) phthalate is a colorless to pale yellow oily liquid and nearly odourless.

Di(2-ethylhexyl) phthalate (DEHP) is a product that is commonly added to plastics to make them flexible. DEHP has low solubility in water and binds strongly to soil particles or organic material.

ISO/AWI 24075 Determination for di(2-ethylhexyl)phthalate (DEHP) released from PVC medical devices (under development).

### **2.6. Fluoranthene**

Fluoranthene is a polycyclic aromatic hydrocarbon (PAH). The molecule can be viewed as the fusion of naphthalene and benzene unit connected by a five-membered ring. Although samples are often pale yellow, the compound is colorless. It is soluble in nonpolar organic solvents. Fluoranthene is found in many combustion products, along with other PAHs. Fluoranthene was added to the Candidate List of Substances of Very High Concern (SVHCs) due to the carcinogenic, toxic to reproduction, persistent, bioaccumulative and toxic (PBT) and very persistent and very bioaccumulative (vPvB) properties.

ISO 13877. 1998. Soil quality -- Determination of polynuclear aromatic hydrocarbons -- Method using high performance liquid chromatography.

ISO 17993. 2002. Water quality - Determination of 15 polycyclic aromatic hydrocarbons (PAH) in water by HPLC with fluorescence detection after liquid-liquid extraction.

ISO 15753. 2006. Animal and vegetable fats and oils -- Determination of polycyclic aromatic hydrocarbons.

EPA method 8100. September 1986. Polynuclear Aromatic Hydrocarbons.

### **2.7. Hexachlorobenzene**

Hexachlorobenzene appears as a white crystalline substance, insoluble in water and denser than water. Hexachlorobenzene was used as a fungicide and was also used as a chemical intermediate in

the manufacture of dyes, synthesis of organic chemicals, rubber and in wood preservation. Prolonged oral exposure to this substance results in a liver disease with associated skin lesions. It has the role of a persistent organic pollutant, carcinogenic agent and antifungal agrochemical agent.

ISO 6468. 1996. Water quality -- Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes -- Gas chromatographic method (ECD) after liquid-liquid extraction.

ISO 10301. 1997. Water quality -- Determination of highly volatile halogenated hydrocarbons – Gaschromatographic methods; ECD detection.

ISO 15680. 2003. Water quality -- Gas-chromatographic determination of a number of monocyclic aromatic hydrocarbons, naphthalene and several chlorinated compounds using purge-and-trap and thermal desorption.

EPA method 1625. 1989. Semivolatile Organic Compounds by Isotope Dilution GCMS.

### **2.8. Hexachlorobutadiene**

Hexachloro-1,3-butadiene is an organochlorine compound.

Hexachlorobutadiene is a colourless liquid with a mild odour. It is insoluble in water and denser than water, non-flammable and may be toxic by ingestion or inhalation.

It is also used as a solvent, and to make lubricants, in gyroscopes, as a heat transfer liquid, and as a hydraulic fluid.

ISO 6468. 1996. Water quality -- Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes -- Gas chromatographic method (ECD) after liquid-liquid extraction.

### **2.9. Hexachlorocyclohexane**

Hexachlorocyclohexane (HCH), formally known as benzene hexachloride (BHC), is a synthetic chemical is produced and used as an insecticide on fruit, vegetables, and forest crops, and animals and animal premises.

Macgregor, K., Oliver, I.W., Harris, L., Ridgway, I.M. 2010. Persistent organic pollutants (PCB, DDT, HCH, HCB & BDE) in eels (*Anguilla anguilla*) in Scotland: Current levels and temporal trends. *Environmental Pollution* 158, 2402-2411.

### 2.10. Lead and its compounds

Lead is a heavy metal that has major health implications. Even low levels of lead exposure have been associated with harmful effects on health, the major sources in the environment being paint and gasoline. In recent years, lead exposure has been decreased by regulatory actions in removing lead from paint and gasoline and limitation of occupational lead exposure. Lead has no medical uses. Lead toxicity is marked by neurotoxicity, neurodevelopmental defects, gastrointestinal, kidney and bone marrow toxicity.

ISO 17294-2. 2003. Water quality -- Application of inductively coupled plasma mass spectrometry (ICPMS) -- Part 2: Determination of 62 elements.

ISO 11885. 2007. Water quality - Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES).

### 2.11. Mercury and compounds

Mercury is silvery-white metal, liquid at room temperature, a rather poor conductor of heat and a fair conductor of electricity.

Mercury is a well-known toxin, second only to lead as a cause of heavy metal poisoning. Mercury is used in many areas of manufacturing and is present in dental and medical equipment. Because of the toxicity of acute and chronic exposure to metallic mercury, this metal is now used less and less in industry and attempts are made to remove it from household and medical equipment and appliances. Mercury is also present in fertilizers and pesticides.

ISO 5666. 1999. Water quality -- Determination of mercury.

ISO 16590. 2000. Water quality -- Determination of mercury -- Methods involving enrichment by amalgamation.

ISO 16772. 2004. Soil quality -- Determination of mercury in aqua regia soil extracts with cold-vapour atomic spectrometry or cold-vapour atomic fluorescence spectrometry.

ISO 17852. 2006. Water quality -- Determination of mercury -- Method using atomic fluorescence spectrometry.

### 2.12. Pentachlorobenzene

Pentachlorobenzene appears as white crystals. Pentachlorobenzene is a benzene in which five of the hydrogens are replaced by chlorines. Now classed as a persistent organic pollutant under the Stockholm Convention. It has a role as a persistent organic pollutant.

ISO 6468:1996 Water quality -- Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes -- Gas chromatographic method after liquid-liquid extraction

(reviewed and confirmed in 2014). The standard describes a method for determining certain organochlorine insecticides, polychlorinated biphenyls (PCBs) and chlorobenzenes (except the mono- and dichlorobenzenes) in drinking water, ground water, surface waters and waste waters. The method is applicable to samples containing up to 0.05 g/l of suspended solids.

### 2.13. Polyaromatic Hydrocarbons (PAH)

Polycyclic aromatic hydrocarbons (PAHs, also *polyaromatic hydrocarbons*) are hydrocarbons—organic compounds containing only carbon and hydrogen—that are composed of multiple aromatic rings. The simplest such chemicals are naphthalene, having two aromatic rings, and the three-ring compounds anthracene and phenanthrene.

PAHs are uncharged, non-polar molecules found in coal and in tar deposits. They are also produced by the thermal decomposition of organic matter (for example, in engines and incinerators or when biomass burns in forest fires).

Compounds with five or more rings have low solubility in water and low volatility; they are therefore predominantly in solid state, bound to particulate air pollution, soils, or sediments.

ISO 13877. 1998. Soil quality -- Determination of polynuclear aromatic hydrocarbons -- Method using high performance liquid chromatography.

ISO 17993. 2002. Water quality - Determination of 15 polycyclic aromatic hydrocarbons (PAH) in water by HPLC with fluorescence detection after liquid-liquid extraction.

EPA method 8100. September 1986. Polynuclear Aromatic Hydrocarbons.

### 2.14. Tributyltin compounds (Tributyltin-cation)

Tributyltin (TBT) was used as a biocide in paint. Tributyltin has found its way into our environment following its use as an antifouling paint on ships and boats.

The TBT slowly leaches out into the marine environment where it is highly toxic.

Hamwijk, C., Schouten, A., Foekema, E.M., Ravensberg, J.C., Collombon, M.T., Schmidt, K., Kugler, M. 2005. Monitoring of the booster biocide dichlofluanid in water and marine sediment of Greek marinas. *Chemosphere* 60, 1316–1324.

Lee, S., Chung, J., Won, H., Lee, D., Lee, Y.-W. 2011. Analysis of antifouling agents after regulation of tributyltin compounds in Korea. *Journal of Hazardous Materials* 185, 1318–1325.



### 2.15. Dicofol

Dicofol is an organochlorine pesticide (acaricide; miticide) that is chemically related to DDT, and used for controlling mites that damage cotton, fruit trees and vegetables.

No analytical standard method is available for Dicofol.

DIN 38407-2. 1993. German standard methods for the determination of water, waste water and sludge; jointly determinable substances (group F); determination of low volatile halogenated hydrocarbons by gas chromatography.

### 2.16. Perfluorooctane sulfonic acid and its derivatives (PFOS)

Perfluorooctane sulfonic acid and its derivatives (PFOS) is a persistent organic pollutants (POP) and was included to the Annex B of the Stockholm Convention on Persistent Organic Pollutants. PFOS is persistent, bioaccumulative and toxic to mammalian species. PFOS is widely used as surface treatment agents for textiles, leather products, paper, furniture and carpets for its excellent waterproofing and oil-resistance performance.

ISO 25101. 2009. Water quality - Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) - Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry.

### 2.17. Quinoxifen

Quinoxifen is a fungicide often used to control powdery mildew infections on grapes and hops. It has a role as an antifungal agrochemical. It is aromatic ether, a member of quinolones, an organochlorine compound and a member of monofluorobenzenes.

No analytical "standard" method is available for Quinoxifen.

Quinoxifen is usually measured using LC-MS or LC-MS/MS, and can be determined significantly below the EQS level.

### 2.18. Dioxins and dioxin-like compounds

Dioxins and dioxin-like compounds comprise Polychlorinated dibenzo-p-dioxins (PCDDs), Polychlorinated dibenzofurans (PCDFs), Polychlorinated biphenyls (PCBs) (twelve of them have "dioxin-like" properties). There are 75 PCDDs, and seven of them are specifically toxic. There are 135 PCDF congeners, and ten of them have "dioxin-like" properties. Dioxins occur as by-products in the manufacture of some organochlorines, in the incineration of chlorine-containing substances such as

PVC (polyvinyl chloride), in the chlorine bleaching of paper, and from natural sources such as volcanoes and forest fires.

EPA method 1613. October 1994. Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. U.S. Environmental Protection Agency, Office of Water, Washington, DC, USA.

### **2.19. Hexabromocyclododecane (HBCDD)**

Hexabromocyclododecane (HBCDD) is a high production volume chemical used as a flame retardant, mainly within the polymer and textile industry. In theory, HBCDD consists of 16 stereoisomers, with water solubility in the range of 2-50µg/l.

No analytical “standard” method is available for HBCDD.

HBCDDs are persistent organic pollutants (POPs) which have been used as flame retardants. LC-MS methods are most commonly used, including those where other polluting flame retardants are determined simultaneously.

Morris, S., Bersuder, P., Allchin, C.R., Zegers, B. 2006. Determination of the brominated flame retardant, hexabromocyclododecane, in sediments and biota by liquid chromatography-electrospray ionisation mass spectrometry. *Trends in Analytical Chemistry* 25, 343-349.

### **2.20. Heptachlor and heptachlor epoxide**

Heptachlor is an insecticide which is banned and not used anymore in the EU. Heptachlor epoxide is its degradation product. Heptachlor epoxide is a white powder. Bacteria and animals break down heptachlor to form heptachlor epoxide. The epoxide is more likely to be found in the environment than heptachlor.

EN ISO 6468. 1996. Water quality - Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes - Gas chromatographic method after liquid-liquid extraction.

EPA Method 1699. December 2007. Pesticides in water, soil, sediment, biosolids, and tissue by HRGC/HRMS. EPA-821-R-08-001. U.S. Environmental Protection Agency, Office of Water, Washington, DC, USA.

Thomas, M., Lazardigues, A., Banas, D., Brun-Bellut, J., Feidt, C. 2012. Organochlorine pesticides and polychlorinated biphenyls in sediments and fish from fresh water cultured fish ponds in different agricultural contexts in north-eastern France. *Ecotoxicology and Environmental Safety* 77, 35–44.

Zhou, R., Zhu, L., Yang, K., Chen, Y. 2006. Distribution of organochlorine pesticides in surface water and sediments from Qiantang River, East China. *Journal of Hazardous Materials* 137, 68–75.

### 3. Sieving and drying

Sieving and drying are carried out in the reference laboratory. Once the sample is submitted to the laboratory, it must be dried (drying temperature max. 40 °C), then sieving at a fraction <63 µm. Homogenization of a dry sample is very important and a homogeneity test must be performed during homogenization. The reference laboratory divides the homogenized sample into three parts: one part remains in the reference laboratory for analysis, the other part is sent to the national analysis laboratory while the third part is archived.

Air-drying is not recommended because of the high risk of pollution. The samples were dried in an oven at 25-30 ° C till more or less constant weight. Prior to analyses of inorganic constituents (e.g. metals), sediment samples may be dried at 105°C (except for mercury determination, which needs a drying step at <50°C) (recommendation Guidance No. 25).

Different types of sediments may require different preparation (drying, sieving, and homogenization) as described in ISO 5667-3:2018 Water quality -- Sampling -- Part 3: Preservation and handling of water samples.

According to recommendation Guidance No. 25, all samples must be sieved over 2 mm mesh as soon as possible after collection to remove large detritus and benthic organisms. Otherwise, during subsequent sample handling and processing, biotic material will deteriorate and become part of the sediment sample. Sieving should take place in the laboratory under controlled conditions. Wet sieving is best performed with ambient water. The same water should be reused to prevent changing the equilibrium. The silt and clay could be separated by sieving over a 63 µm mesh sieve (fraction <63 µm).

Sieves are traditionally made of corrosion-resistant brass (rim and mesh). Today, stainless steel is preferred for organics analyses. These must not be used for the analysis of trace metals, however. For trace metals, polymer sieves are recommended (PVC or acrylic rim, with e.g. nylon or polyester mesh), (recommendation Guidance No. 25).

### 4. Sample storage and archive

The samples are stored in plastic and dark glass bottles and stored in a cool place (usually at 2 °C to 8 °C). For storage for short periods (up to 24 h), cooling at 2 ° C to 8 ° C in designated laboratory refrigerators may be applied. For sample storing during longer periods (more than a month) it is recommended to freeze sample at -20°C. Before the analysis of such sample, care should be taken to thaw the sample thoroughly (HRN ISO 5667-12:2001, ISO 5667-12:2017, ISO 5667-15:2009 (reviewed and confirmed in 2015)).

All storage methods will affect the sample to some extent, and the choice of preservation technique depends mainly on the objective of the sample collection.

According to recommendation Guidance No. 25, a technique for one group of analyses may interfere with other analyses. To overcome this problem, a sufficient sample volume should be collected to allow specific preservation or storage techniques for each specific group of analytes. Sample can be stored at 4°C for about a week and up to 3 months when frozen at -20°C, unless otherwise specified in the analytical methods for specific degradable compounds. Whenever possible, freezing should be avoided because it can change the grain size distribution of the sediment.

## 5. Normalization

Numerous impacts in bulk sediment composition, such as particle size fraction, the dilution of contaminants by material like quartz or carbonates, organic matter content and background values influence the reliable interpretation of laboratory results. Normalizing could include grain size correction, quartz correction, common normalizers are Al and Li, with some limitations Fe, then organic carbon and trace elements (Cs, Eu, Rb, Sc, Sm, and Th, etc.)

### 5.1. Grain size correction

Coarser grain fraction dilutes pollution. The fine fraction, especially the clay-silt fraction (>63µm) is suitable for analyses. The first step is sieving to isolate the fine fraction. Some minerals could remain in the fine fraction it is necessary to make correction for them. Silica minerals and carbonates naturally contain negligible amounts of heavy metals and therefore dilute pollution in sediments.

### 5.2. Quartz correction

The high concentration of the quartz fraction in the sample produces a dilution of the existent contaminants which is known as a „matrix effect“. The quartz correction produces values of trace metals with the realistic assessment of the degree of metal concentration or adsorption by the clay fine fraction (UNESCO, WHO, UNEP, 1992). According to Thomas (1972) the quartz correction could be calculate by formula:  $C_c = (C_o \times 100) / (100 - q_z)$ , where  $C_c$  is the quartz corrected concentration,  $C_o$  is the trace metal observed concentration and  $q_z$  is the % of quartz content. Quartz content could be determined by X-ray diffraction (Till and Spears, 1969) or by the gravimetric determination of total quartz plus feldspar (Trostell and Wynne, 1940).

### 5.3. Al- and Li-normalization

The content of Al and Li has significant correlations with heavy metals. Aluminium is one of the most abundant naturally-occurring metals which could be found in aluminosilicate mineral fraction. It is

immobile and has negligible anthropogenic impact. Lithium is better normalizer for the sediment enriched by phyllosilicates.

## 6. Quality Control

The laboratory should check during reception of samples all relevant information according to preservation and transport conditions of the sample (ISO 5667-15:2009 (reviewed and confirmed in 2015)).

Quality control should be properly ensured in the laboratory using the following recommendations:

- Proper quality assurance / quality control of method validation by analytical laboratories, routine Internal QC procedures, and independent external QC procedures;
- The validation of an analytical method, including the determination of measurement reliability, bias, etc., requires the use of certified reference materials;
- In CIS Guidance No. 19-“ANNEX III a complete list of sediment certified reference materials (CRM) is reported (available for the determination of metals, PAHs and chlorinated pesticides in sediment (no CRM developed for other substances));
- Internal QC procedures should routinely monitor the performance of analytical methods for example by including duplicate samples or (laboratory) reference materials in analytical batches. The results are evaluated using standard statistical methods, such as Shewhart charts, to ensure that the methods remain under statistical control;
- The laboratory should regularly participate in external inter-laboratory comparisons.

It is recommended that quality control is carried out in accordance with ISO/IEC 17025:2017.

## 7. Reference

DIN 38407-2. 1993. German standard methods for the determination of water, waste water and sludge; jointly determinable substances (group F); determination of low volatile halogenated hydrocarbons by gas chromatography.

EC 2010. Common Implementation Strategy for the Water Framework Directive (2000/60/EC): Guidance Document No. 25 Guidance on chemical monitoring of sediment and biota under the Water Framework Directive Luxembourg: Office for Official Publications of the European Communities.

EC 2018. Common Implementation Strategy for the Water Framework Directive (2000/60/EC): Technical Guidance for deriving Environmental Quality Standards, Guidance Document No. 27 Updated version 2018.

EPA method 1613. October 1994. Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS. U.S. Environmental Protection Agency, Office of Water, Washington, DC, USA.

EPA Method 1614. August 2007. Brominated Diphenyl Ethers in water soil, sediment and tissue by HRGC/HRMS. EPA-821-R-07-005. U.S. Environmental Protection Agency, Office of Water, Washington, DC, USA.

EPA method 1625. 1989. Semivolatile Organic Compounds by Isotope Dilution GCMS.

EPA Method 1699. December 2007. Pesticides in water, soil, sediment, biosolids, and tissue by HRGC/HRMS. EPA-821-R-08-001. U.S. Environmental Protection Agency, Office of Water, Washington, DC, USA.

EPA method 8100. September 1986. Polynuclear Aromatic Hydrocarbons.

Hamwijk, C., Schouten, A., Foekema, E.M., Ravensberg, J.C., Collombon, M.T., Schmidt, K., Kugler, M. 2005. Monitoring of the booster biocide dichlofluanid in water and marine sediment of Greek marinas. *Chemosphere* 60, 1316–1324.

HRN ISO 5667-12:2001, Water quality - Sampling - Part 12: Guidance for bottom sediment sampling (ISO 5667-12:1995)

ISO/AWI 24075 Determination for di(2-ethylhexyl)phthalate (DEHP) released from PVC medical devices (under development).

ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories

ISO 5666. 1999. Water quality -- Determination of mercury.

ISO 5667-3:2018 Water quality -- Sampling -- Part 3: Preservation and handling of water samples

ISO 5667-15:2009 Water quality -- Sampling -- Part 15: Guidance on the preservation and handling of sludge and sediment samples (reviewed and confirmed in 2015).

ISO 5961:1994 Water quality -- Determination of cadmium by atomic absorption spectrometry (reviewed and confirmed in 2015).

ISO 6468:1996 Water quality -- Determination of certain organochlorine insecticides, polychlorinated biphenyls and chlorobenzenes -- Gas chromatographic method after liquid-liquid extraction (reviewed and confirmed in 2014).

ISO 10301. 1997. Water quality -- Determination of highly volatile halogenated hydrocarbons – Gas chromatographic methods; ECD detection.

ISO 12010:2019 Water quality -- Determination of short-chain polychlorinated alkanes (SCCP) in water -- Method using gas chromatography-mass spectrometry (GC-MS) and negative-ion chemical ionization (NCI)

ISO 11885. 2007. Water quality - Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES).

ISO 13877. 1998. Soil quality -- Determination of polynuclear aromatic hydrocarbons -- Method using high performance liquid chromatography.

ISO 15680. 2003. Water quality -- Gas-chromatographic determination of a number of monocyclic aromatic hydrocarbons, naphthalene and several chlorinated compounds using purge-and-trap and thermal desorption.

ISO 15753. 2006. Animal and vegetable fats and oils -- Determination of polycyclic aromatic hydrocarbons.

ISO 16590. 2000. Water quality -- Determination of mercury -- Methods involving enrichment by amalgamation.

ISO 16772. 2004. Soil quality -- Determination of mercury in aqua regia soil extracts with cold-vapour atomic spectrometry or cold-vapour atomic fluorescence spectrometry.

ISO 17294-2. 2003. Water quality -- Application of inductively coupled plasma mass spectrometry (ICPMS) -- Part 2: Determination of 62 elements.

ISO 17852. 2006. Water quality -- Determination of mercury -- Method using atomic fluorescence spectrometry.

ISO 17993. 2002. Water quality - Determination of 15 polycyclic aromatic hydrocarbons (PAH) in water by HPLC with fluorescence detection after liquid-liquid extraction.

ISO 22032. 2006. Water quality - Determination of selected polybrominated diphenylethers (PBDE) in sediment and sewage sludge - Method using extraction and gas chromatography/mass spectrometry.

ISO 25101. 2009. Water quality - Determination of perfluorooctanesulfonate (PFOS) and perfluorooctanoate (PFOA) - Method for unfiltered samples using solid phase extraction and liquid chromatography/mass spectrometry.

Lee, S., Chung, J., Won, H., Lee, D., Lee, Y.-W. 2011. Analysis of antifouling agents after regulation of tributyltin compounds in Korea. *Journal of Hazardous Materials* 185, 1318–1325.

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