Observations and comments for Transnationally harmonized sediment laboratory analysis protocol for HSs in DRB's surface waters proposal

Deliverable D 4.3.2

Project title	Sediment-quality Information, Monitoring and Assessment System to support transnational cooperation for joint Danube Basin water management
Acronym	**SIMONA**
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Observation notes of the chapters:

The comments and suggestions on the D 4.2.2 Transnationally harmonized sediment laboratory analysis protocol for HSs in DRB's surface waters proposal are presented in this deliverable.

1. Observation of Introduction

HGI-CGS corrected paragraph: *"The Danube river basin is under great pressure from a diverse range of human activities. River sediments have an important role in the monitoring of the environment as they are considered as the final sink of most contaminants. Many aquatic species live in the sediment. Microbial processes cause regeneration of nutrients and functioning of nutrient cycles for the whole water body. Accordingly, a healthy river needs sediment as a source of life. One of the aims of this study is to investigate the spatial and temporal variation in heavy metals and organic compound concentrations in sediment for the risk assessment status in the Danube river, with review to determine its ability to sustain aquatic life."*

GI-BAS comment: *"there is no legalised BG-national derived EQSs for sediments"*

HGI-CGS suggested: *"The overviews of the laboratory methods and procedures (norms) for analysis of HSs from agricultural activities, organic pollutants predominantly released from the industrial activates and heavy metals and its compounds in DTP countries are presented in Appendix 1, 2, 3."*

2. Observation of Selected priority HSs

TUCN created Excel table with the prescribed HSs in sediments according to the various EU documents. After Water Framework Directive was established in EU documents were some changes of number and status of priority hazardous substances. The table with the correlation between proposed PSs in Guidance Document No. 25 Guidance on chemical monitoring of sediment and biota under the Water Framework Directive, the Directive 2013/39/EU amending the Directive 2008/105/EC and List of Priority Substances for the Danube River Basin is presented in **Appendix 1.**

HGI-CGS added: Additionally in this protocol in paragraphs 2.21. to 2.25. added 5 heavy metals and its compounds from the List of Priority Substances for the Danube River Basin (ICPDR, 2003).

IGR suggested: *"There are reference guides for establishing the sediment quality (Dutch General Environmental Quality Standards, Canadian CSQG, Canadian PSQG), that list more metals (As, Cu, Cr, Mo, Ni, Pb, Zn). Some of the partner countries in the SIMONA project already monitor these metals in*

sediments (mainly for establishing the quality of rivers waters).That is why it might be appropriate to specify more metals besides Cd, Pb and Hg."

2.1. Anthracene

HGI-CGS has changed description of the substance.

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

GI-BAS suggested: ISO 18287 Soil quality - Determination of polycyclic aromatic hydrocarbons (PAH) - Gas chromatographic method with mass spectrometric detection (GC-MS).

GI-BAS suggested: EN 16181 Soil, treated biowaste and sludge - Determination of polycyclic aromatic hydrocarbons (PAH) by gas chromatography (GC) and high performance liquid chromatography (HPLC).

HGI-CGS added years in norms: ISO 18287: **2006.** and EN 16181: **2018.**

HGI-CGS suggested: EN 16181: 2018 Soil, treated biowaste and sludge - Determination of polycyclic aromatic hydrocarbons (PAH) by gas chromatography (GC) and high performance liquid chromatography (HPLC).

2.2. Brominated diphenylethers

HGI-CGS has changed description of the substance.

2.3. Cadmium and its compounds

HGI-CGS has changed description of the substance.

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

GI-BAS suggested: EN 16171 Sludge, treated biowaste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS).

HGI-CGS added year in norm: EN 16171: **2016.**

HGI-CGS suggested: ISO 22036: 2008. Soil quality – Determination of trace elements in extracts of soil by inductively coupled plasma – atomic emission spectrometry (ICP-AES).

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

2.4. C10-13-chloroalkanes

HGI-CGS has changed description of the substance.

GI-BAS deleted: 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).

GI-BAS suggested: ISO 22032 Water quality - Determination of selected polybrominated diphenyl ethers in sediment and sewage sludge - Method using extraction and gas chromatography/mass spectrometry (ISO 22032:2006).

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

HGI-CGS has changed description of the substance.

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

GI-BAS suggested: ISO 13913 Soil quality - Determination of selected phthalates using capillary gas chromatography with mass spectrometric detection (GC/MS).

HGI-CGS added year in norm: ISO 13913: **2014.**

HGI-CGS suggested: ISO 13913 2014. Soil quality - Determination of selected phthalates using capillary gas chromatography with mass spectrometric detection (GC/MS).

2.6. Fluoranthene

HGI-CGS has changed description of the substance.

GI-BAS suggested: ISO 18287 Soil quality - Determination of polycyclic aromatic hydrocarbons (PAH) - Gas chromatographic method with mass spectrometric detection (GC-MS) and

EN 16181. Soil, treated biowaste and sludge - Determination of polycyclic aromatic hydrocarbons (PAH) by gas chromatography (GC) and high performance liquid chromatography (HPLC).

HGI-CGS added years in norms: ISO 18287: **2006.** and EN 16181: **2018.**

HGI-CGS suggested: ISO 18287 2006. Soil quality - Determination of polycyclic aromatic hydrocarbons (PAH) - Gas chromatographic method with mass spectrometric detection (GC-MS) and

EN 16181 2018. Soil, treated biowaste and sludge - Determination of polycyclic aromatic hydrocarbons (PAH) by gas chromatography (GC) and high performance liquid chromatography (HPLC).

2.7. Hexachlorobenzene

HGI-CGS has changed description of the substance.

GI-BAS deleted: 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 and

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.

GI-BAS suggested: ISO 10382 Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection.

HGI-CGS added year in norm: ISO 10382: **2002.**

HGI-CGS suggested: ISO 10382: 2002. Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection.

2.8. Hexachlorobutadiene

HGI-CGS has changed description of the substance.

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

GI-BAS suggested: EN ISO 15009 Soil quality - Gas chromatographic determination of the content of volatile aromatic hydrocarbons, naphthalene and volatile halogenated hydrocarbons - Purge-and-trap method with thermal desorption (ISO 15009:2016).

HGI-CGS suggested: ISO 10382: 2002. Soil quality – Determination of organochlorine pesticides and polychlorinated biphenyls – Gas-chromatographic method with electron capture detection.

2.9. Hexachlorocyclohexane

HGI-CGS has changed description of the substance.

GI-BAS suggested: ISO 10382 Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection.

HGI-CGS added year in norm: ISO 10382: **2002.**

HGI-CGS suggested: ISO 10382: 2002. Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection.

2.10. Lead and its compounds

HGI-CGS has changed description of the substance.

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

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

GI-BAS suggested: EN 16171 Sludge, treated biowaste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS).

HGI-CGS added year in norm: EN 16171: **2002.**

HGI-CGS suggested: EN 16171: 2016. Sludge, treated biowaste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS) and

ISO 22036: 2008. Soil quality- Determination of trace elements in extracts of soil by inductively coupled plasma – atomic emission spectrometry (ICP-AES).

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

2.11. Mercury and compounds

HGI-CGS has changed description of the substance.

GI-BAS deleted: ISO 17852. 2006. Water quality -- Determination of mercury -- Method using atomic fluorescence spectrometry.

GI-BAS suggested: EN 16171 Sludge, treated biowaste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS).

HGI-CGS added year in norm: EN 16171: **2016.**

HGI-CGS suggested: EN 16171: 2016. Sludge, treated biowaste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS).

ISO 12846: 2012. Water quality – Determination of mercury – Method using atomic absorption spectrometry (AAS) with and without enrichment and

Mercury determination in soils and sediments is based on ISO 12846 using flameless AAS.

IGR suggested: EPA7473 Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry.

IGR suggested: U.S.EPA245.5 The Determination of Mercury in Soils, Sediments and Sludges by Cold Vapor Atomic Absorption Spectroscopy.

2.12. Pentachlorobenzene

HGI-CGS has changed description of the substance.

GI-BAS deleted: 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.

GI-BAS suggested: ISO 10382 Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection.

HGI-CGS added year in norm: ISO 10382: **2002.**

HGI-CGS suggested: ISO 10382: 2002. Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection.

2.13. Polyaromatic Hydrocarbons (PAH)

HGI-CGS has changed description of the substance.

GI-BAS suggested: EN 16181: 2018. Soil, treated biowaste and sludge - Determination of polycyclic aromatic hydrocarbons (PAH) by gas chromatography (GC) and high performance liquid chromatography (HPLC).

ISO 18287 Soil quality - Determination of polycyclic aromatic hydrocarbons (PAH) - Gas chromatographic method with mass spectrometric detection (GC-MS).

HGI-CGS added years in norms: ISO 18287: **2008.** and ISO 16181: **2018.**

HGI-CGS suggested: ISO 18287: 2006. Soil quality - Determination of polycyclic aromatic hydrocarbons (PAH) - Gas chromatographic method with mass spectrometric detection (GC-MS).

2.14. Tributyltin compounds (Tributyltin-cation)

HGI-CGS has changed description of the substance.

GI-BAS suggested: EN ISO 23161 Soil quality. Determination of selected organotin compounds. Gas-chromatographic method.

HGI-CGS added year in norm: ISO 23161: **2018.**

HGI-CGS suggested: EN ISO 23161: 2018. Soil quality. Determination of selected organotin compounds. Gas-chromatographic method.

2.15. Dicofol

HGI-CGS has changed description of the substance.

HGI-CGS deleted: No analytical standard method is available for Dicofol.

HGI-CGS added: ISO 10382: 2002. Soil quality – Determination of organochlorine pesticides and polychlorinated biphenyls – Gas-chromatographic method with electron capture detection (DIN ISO 10392: 2003-05).

2.16. Perfluorooctane sulfonic acid and its derivatives (PFOS)

HGI-CGS has changed description of the substance.

GI-BAS deleted: 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.

HGI-CGS added: DIN 38407-42: 2011-03. German standard methods for the examination of water, waste water and sludge – Jointly determinable substances (group F) – Part 42: Determination of selected polyfluorinated compounds (PFC) in water – Method using high performance liquid chromatography and mass spectrometric detection (HPLC/MS-MS) after solid-liquid extraction (F42).

2.17. Quinoxyfen

HGI-CGS has changed description of the substance.

HGI-CGS deleted: No analytical "standard" method is available for Quinoxyfen.

HGI-CGS added: DIN 38407-35:2010-10. German standard methods for the examination of water, waste water and sludge – Jointly determinable substances (group F) – Part 35: Determination of selected phenoxyalkyl carbonic acids and further acid plant treatment agents – Method using high performance liquid chromatography and mass spectrometric detection (HPLC-MS/MS) (F35).

2.18. Dioxins and dioxin-like compounds

HGI-CGS has changed description of the substance.

HGI-CGS suggested: DIN 38414-24: 2000-10. German standard method for the examination of water, waste water and sludge – Sludge and sediments (group 5) – Part 24: Determination of polychlorinated dibenzodioxins (PCDD) and polychlorinated dibenzofurans 8PCDF) (S 24) and

ISO 10382: 2002. Soil quality – Determination of organochlorine pesticides and polychlorinated biphenyls – Gas-chromatographic method with electron capture detection.

2.19. Hexabromocyclododecane (HBCDD)

HGI-CGS has changed description of the substance.

HGI-CGS deleted: No analytical "standard" method is available for HBCDD.

HGI-CGS suggested: ISO 22032: 2006. Water quality – Determination of selected polybrominated diphenyl ethers in sediment and sewage sludge – Method using extraction and gas-chromatography/mass spectrometry (DIN EN ISO 22032: 2009-07).

2.20. Heptachlor and heptachlor epoxide

HGI-CGS has changed description of the substance.

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

GI-BAS suggested: ISO 10382 Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection.

HGI-CGS added year in norm: ISO 10382: **2002.**

HGI-CGS suggested: ISO 10382: 2002. Soil quality — Determination of organochlorine pesticides and polychlorinated biphenyls — Gas-chromatographic method with electron capture detection.

Additionally, to meet requirements of the ICPDR, HGI-CGS added following heavy metals, from 2.21. to 2.25.:

2.21. Nickel and its compounds

HGI-CGS added description of the substance and standards:

EN 16171: 2016. Sludge, treated bio-waste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS).

ISO 22036: 2008. Soil quality – Determination of trace elements in extracts of soil by inductively coupled plasma – atomic emission spectrometry (ICP-AES).

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

RO-IGR added: ISO 18227:2014 (Soil quality - Determination of elemental composition by XRF) specifies the procedure for a quantitative determination of major and trace element concentrations in homogeneous solid waste, soil, and soil-like material by energy dispersive X-ray fluorescence (EDXRF) spectrometry or wavelength dispersive X-ray fluorescence (WDXRF) spectrometry using a calibration with matrix-matched standards

RO-IGR added: ISO 13196:2013Soil quality -- Screening soils for selected elements by energy-dispersive X-ray fluorescence spectrometry using a handheld or portable instrument

2.22. Arsenic and its compounds

HGI-CGS added description of the substance and standards:

EN 16171: 2016. Sludge, treated bio-waste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS).

ISO 22036: 2008. Soil quality – Determination of trace elements in extracts of soil by inductively coupled plasma – atomic emission spectrometry (ICP-AES).

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

2.23. Zinc and its compounds

HGI-CGS added description of the substance and standards:

EN 16171: 2016. Sludge, treated bio-waste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS).

ISO 22036: 2008. Soil quality – Determination of trace elements in extracts of soil by inductively coupled plasma – atomic emission spectrometry (ICP-AES).

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

RO-IGR added: ISO 18227:2014 (Soil quality - Determination of elemental composition by XRF) specifies the procedure for a quantitative determination of major and trace element concentrations in homogeneous solid waste, soil, and soil-like material by energy dispersive X-ray fluorescence (EDXRF) spectrometry or wavelength dispersive X-ray fluorescence (WDXRF) spectrometry using a calibration with matrix-matched standards.

RO-IGR added: ISO 13196:2013Soil quality -- Screening soils for selected elements by energy-dispersive X-ray fluorescence spectrometry using a handheld or portable instrument.

2.24. Chromium and its compounds

HGI-CGS added description of the substance and standards:

EN 16171: 2016. Sludge, treated bio-waste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS).

ISO 22036: 2008. Soil quality – Determination of trace elements in extracts of soil by inductively coupled plasma – atomic emission spectrometry (ICP-AES).

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

RO-IGR added: ISO 18227:2014 (Soil quality - Determination of elemental composition by XRF) specifies the procedure for a quantitative determination of major and trace element concentrations in homogeneous solid waste, soil, and soil-like material by energy dispersive X-ray fluorescence (EDXRF) spectrometry or wavelength dispersive X-ray fluorescence (WDXRF) spectrometry using a calibration with matrix-matched standards.

RO-IGR added: ISO 13196:2013Soil quality -- Screening soils for selected elements by energy-dispersive X-ray fluorescence spectrometry using a handheld or portable instrument.

2.25. Copper and its compounds

HGI-CGS added description of the substance and standards:

EN 16171: 2016. Sludge, treated bio-waste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS).

ISO 22036: 2008. Soil quality – Determination of trace elements in extracts of soil by inductively coupled plasma – atomic emission spectrometry (ICP-AES).

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

RO-IGR added: ISO 18227:2014 (Soil quality - Determination of elemental composition by XRF) specifies the procedure for a quantitative determination of major and trace element concentrations in homogeneous solid waste, soil, and soil-like material by energy dispersive X-ray fluorescence (EDXRF) spectrometry or wavelength dispersive X-ray fluorescence (WDXRF) spectrometry using a calibration with matrix-matched standards.

RO-IGR added: ISO 13196:2013Soil quality -- Screening soils for selected elements by energy-dispersive X-ray fluorescence spectrometry using a handheld or portable instrument.

3. Observation of Sieving and drying

HGI-CGS deleted text: *"Sieving and drying should be 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."*

HGI-CGS added text: *"A wet-sieving should be done in the field or if not possible in the reference laboratory. Once the sample is submitted to the laboratory, it must be dried at max. temperature 25-30 °C (some compounds needs freeze drying)."*

SK-SIGDS comment: *"Sieving after drying is problematic. The sediment containing silt and clay is mostly baked and must be crushed. When crushing, the original granulometric composition of the sediment cannot be guaranteed."*

HGI-CGS correction: *"it must be dried at max. temperature 25-30 °C. At temperatures > 30 °C mercury escapes."*

GI-BAS comment: some compounds needs freeze drying

HGI-CGS added: *"For a wet sieved sample fraction < 63 µm (wet sieving in the field) and for a subsequent analysis or organic compounds, the freeze-drying of samples is recommended."*

HGI-CGS added: ISO 5667-15: 2009. Water quality – Sampling – Part 15: Guidance on the preservation and handlingof sludge and sediment samples (reviewed and confirmed in 2015).

ISO 5567-13: 2011. Water quality – Sampling – Part 12: Guidance on sampling of sludges.

4. Observation of Sample storage and archive

No comments.

5. Observation of Normalization

HGI-CGS added: *"and parent material (natural geochemical background)"*

5.1. Grain size correction

HGI – CGS correction of typo: "<63 µm"

5.2. Quartz correction

No comments.

5.3. Al- and Li-normalization

No comments.

6. Observation of Quality Control

HGI-CGS added text:

"The field quality assurance involves a series of steps, procedures, and practise. The quality of data generated in a laboratory depends, to a large degree, on the integrity of the samples that arrive there. Consequently, the field investigator must take the necessary precautions to protect the samples from contamination and deterioration.

There are many sources of contamination; the following are some basic precautions to consider:

- *Sample containers must be cleaned according to recognised methods and certified by the issuing laboratory as 'contamination free'.*
- *Only the recommended type of sample container for each analysis should be used.*
- *The inner portion of the sample container and caps must not be touched by anything other than the sample itself.*
- *Sample containers must be kept in a clean environment, away from dust, dirt, flames and grime. Containers must be capped at all times and possibly stored in coolers before and after sampling.*

- *Petroleum products (e.g., gasoline, oil, exhaust fumes) are prime sources of contamination. Exhaust fumes and cigarette smoke can contaminate samples with lead and other trace elements.*
- *Samples must never be permitted to get warm. They should be stored in a cool place, coolers packed with ice packs are recommended.*

In addition to the standardised field procedure, field quality control requires the submission of replicate and reference samples. Replicate samples detect heterogeneity, allow the precision of the measurement process to be estimated, and provide a check that the sample is reproducible. Reference samples are used primarily to document the bias of the analytical (laboratory) process. The frequency of replicate and reference samples should be established in the project design. Reference samples should be submitted to the analysing laboratory with the samples collected in the field."

and

"The analytical laboratories had to prove a valid accreditation for the required parameters and analytical methods as well as according to ISO/IEC 17025."

HGI-CGS added standard: ISO/IEC 17025: 2017. General requirements for the competence of testing and calibration laboratories (DIN EN ISO/IEC 17025: 2018-03).

7. Observation of References

The following References were deleted from protocol by GI-BAS:

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

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

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

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

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

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

The following References were added in protocol by HGI-CGS, GI-BAS, IGR and JCI:

Abdel-Shafy, H.I., Mansour, M.S. 2016. A review on polycyclic aromatic hydrocarbons: Source, environmental impact, effect on human health and remediation. Egyptian Journal of Petroleum 25, 107–123.

ATSDR 2005. Toxicological Profile for Alpha-, Beta-, Gamma-, and Delta-Hexachlorocyclohexane (Update). Department of Public Health and Human Services, Public Health Service, Agency for Toxic Substances and Disease Registry. Atlanta, GA, U.S, 325p.

Alloway, B.J. (Ed.) 2013. Heavy Metals in Soils Trace Metals and Metalloids in Soils and their Bioavailability. Springer Netherlands, 613p.

Antizar-Ladislao, B. 2008. Environmental levels, toxicity and human exposure to tributyltin (TBT)-contaminated marine environment. A review. Environment International 34, 292–308.

Barber, J.L., Sweetman, A.J., Van Wijk, D., Jones, K.C. 2005. Hexachlorobenzene in the global environment: Emissions, levels, distribution, trends and processes. Science of the Total Environment 349, 1-44.

Danish EPA 2013. Evaluation of health hazards by exposure to Chlorinated paraffins and proposal of a health-based quality criterion for ambient air. Ed. Elsa Nielsen, Ole Ladefoged 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. German Institute for Standardisation.

DIN 38407-35:2010-10 German standard methods for the examination of water, waste water and sludge – Jointly determinable substances (group F) – Part 35: Determination of selected phenoxyalkyl carbonic acids and further acid plant treatment agents – Method using high performance liquid chromatography and mass spectrometric detection (HPLC-MS/MS) (F35). German Institute for Standardisation.

DIN 38407-42:2011-03 German standard methods for the examination of water, waste water and sludge – Jointly determinable substances (group F) – Part 42: Determination of selected polyfluorinated compounds (PFC) in water – Method using high performance liquid chromatography and mass spectrometric detection (HPLC/MS-MS) after solid-liquid extraction (F 42). German Institute for Standardisation.

DIN 38414-24:2000-10 German standard method for the examination of water, waste water and sludge – Sludge and sediments (group 5) – Part 24: Determination of polychlorinated dibenzodioxins (PCDD) and polychlorinated dibenzofurans 8PCDF) (S 24). German Institute for Standardisation.

Emsbo-Mattingly, S.D., Litman, E. 2016. Polycyclic aromatic hydrocarbon homolog and isomer fingerprinting. In: Stout, S.A., Wang, Z. (Eds.). Standard Handbook Oil Spill Environmental Forensics. Elsevier, 255-312.

EN ISO 15009:2016 Soil quality – Gas chromatographic determination of the content of volatile aromatic hydrocarbons, naphthalene and volatile halogenated hydrocarbons – Purge-and-trap method with thermal desorption. European Standardisation Organisation and International Organization for Standardization.

EN ISO 23161:2018 Soil quality – Determination of selected organotin compounds. Gas-chromatographic method. European Standardisation Organisation and International Organization for Standardization.

EN 16171:2016 Sludge, treated bio-waste and soil. Determination of elements using inductively coupled plasma mass spectrometry (ICP-MS). European Standardisation Organisation.

EN 16181:2018 Soil, treated biowaste and sludge – Determination of polycyclic aromatic hydrocarbons (PAH) by gas chromatography (GC) and high performance liquid chromatography (HPLC). European Standardisation Organisation.

EPA 2016. Health Effects Support Document for Perfluorooctane Sulfonate (PFOS). U.S. Environmental Protection Agency, Office of Water, Washington, DC, USA, 245p.

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Conclusion:

- **The most ISO standards for water could be, especially for organic parameters, used in modified form for solids (soils, sewage sludge, and river sediments). To avoid any misunderstanding, all such standards in protocol were deleted;**
- **The new norms were added;**
- **The following heavy metals from ICPDR list were added: Nickel and its compounds, Arsenic and its compounds, Zinc and its compounds, Chromium and its compounds and Copper and its compounds;**
- **The QC is detailed explained;**
- **According to changes in protocol were some References deleted and some added.**