BIOSOLIDS OBSERVATORY

CONTAMINANTS ANALYSIS TO IMPROVE RISK MANAGEMENT



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LIST OF ACRONYMS

- **ADEME**: *Agence de l'Environnement et de la Maîtrise de l'Énergie* (French Agency for Ecological Transition)
- **Afssa**: *Agence française de sécurité sanitaire des aliments* (French Food Safety Agency)
- **AM**: Arithmetic Mean
- **ANSES**: *Agence nationale de sécurité sanitaire de l'alimentation, de l'environnement et du travail* (French National Agency for Food, Environmental and Occupational Health & Safety)
- **AOX**: Adsorbable Organic Halogens
- **APE**: Aqua Publica Europea (The European Association of Public Water Operators)
- **As**: Arsenic
- **BAT**: Best Available Techniques
- **BBP**: Benzyl butyl phthalate
- **BEP**: Best Environmental Practices
- **BFR / BFH**: Brominated Flame Retardants
- **BML**: *Bundesministerium für Land- und Forstwirtschaft, Regionen und Wasserwirtschaft* (Austrian Federal Ministry of Agriculture, Forestry, Regions and Water Management)
- **BMUV**: *Bundesministerium für Umwelt, Naturschutz, nukleare Sicherheit und Verbraucherschutz* (German Federal Ministry for the Environment, Nature Conservation, Nuclear Safety and Consumer Protection)
- **BOD / BOD5**: Biochemical Oxygen Demand / 5-day Biochemical Oxygen Demand
- **BPA**: Bisphenol A
- **BPB**: Bisphenol B
- **BPS**: Bisphenol S
- **BTEX**: Benzene, Toluene, Ethylbenzene, and Xylenes
- **BW**: Body Weight (used in dietary toxicity calculations)
- **C10–13**: Carbon chain length range for short-chain hydrocarbons
- **C14–17**: Carbon chain length range for medium-chain hydrocarbons
- **C≥18**: Carbon chain length range for long-chain hydrocarbons
- **Ca / CaO**: Calcium / Calcium Oxide (Lime treatment form)
- **CAS**: Chemical Abstracts Service registry designation system
- **Cd**: Cadmium
- **COD**: Chemical Oxygen Demand
- **CP**: Chlorinated Paraffins
- **Cr**: Chromium

- **Cu**: Copper
- **DBP**: Di-n-butyl phthalate
- **DBT**: Dibutyltin
- **DecaBDE**: Decabromodiphenyl ether (BDE-209)
- **DEHP**: Di(2-ethylhexyl) phthalate
- **DEP**: Diethyl phthalate
- **DIBP**: Diisobutyl phthalate
- **DM**: Dry Matter
- **DMP**: Dimethyl phthalate
- **DNOP**: Di-n-octyl phthalate
- **DOT**: Dioctyltin
- **DS**: Dry Solids
- **E1**: Estrone
- **E2**: 17β-estradiol
- **EC**: European Community / European Commission
- **EDI**: Estimated Daily Intake
- **EE2**: 17α-ethinylestradiol
- **EEC**: European Economic Community
- **EFAR**: European Federation for Agricultural Recycling
- **EFSA**: European Food Safety Authority
- **EPA**: Environmental Protection Agency (e.g., US EPA, Danish EPA)
- **EU**: European Union
- **EurEau**: The European Association of Water Services
- **FATE SEES**: Pan-European analysis campaign (*Occurrence and levels of selected compounds in European Sewage Sludge Samples*)
- **GC-MS**: Gas Chromatography–Mass Spectrometry
- **GC-MS/MS**: Gas Chromatography–tandem Mass Spectrometry
- **HBCD**: Hexabromocyclododecane
- **Hg**: Mercury
- **HMW**: High-Molecular-Weight
- **ICP-MS**: Inductively Coupled Plasma Mass Spectrometry
- **IED**: Industrial Emissions Directive (Directive 2010/75/EU / 2024/1785)
- **INERIS**: *Institut National de l'Environnement Industriel et des Risques* (French National Institute for Industrial Environment and Risks)
- **ISO**: International Organization for Standardization
- **I-TEQ**: International Toxic Equivalent
- **JRC**: Joint Research Centre
- **K / K2O**: Potassium / Potassium Oxide (Potash calculation form)
- **kg/ha / kg/ha/year**: Kilograms per hectare / Kilograms per hectare per year
- **LANAE**: Partnering analytical laboratory network
- **LAS**: Linear Alkylbenzene Sulfonates
- **LC-MS/MS**: Liquid Chromatography–tandem Mass Spectrometry
- **LCCP**: Long-Chain Chlorinated Paraffins
- **LMW**: Low-Molecular-Weight
- **LOD**: Limit of Detection
- **LOQ**: Limit of Quantification
- **Max**: Maximum observed value

- **MBBR**: Moving Bed Biofilm Reactor
- **MBT**: 2-mercaptobenzothiazole
- **MCCP**: Medium-Chain Chlorinated Paraffins
- **Med**: Median value
- **Mg / MgO**: Magnesium / Magnesium Oxide
- **mg/kg / mg/kg DS / mg/kg DM**: Milligrams per kilogram / of Dry Solids / of Dry Matter
- **Min**: Minimum observed value
- **MOT**: Monooctyltin
- **MTBE**: Methyl tert-butyl ether (or methyl tertiobutyl ether)
- **N / n**: Number of samples / total sample population size
- **N**: Nitrogen (Total N, NH4-N for Ammonium, NO3-N for Nitrate)
- **ng WHO-TEQ/kg**: Nanograms of World Health Organization Toxic Equivalents per kilogram
- **Ni**: Nickel
- **NOAEL**: No Observed Adverse Effect Level
- **NP**: Nonylphenol
- **NPE / NPEO**: Nonylphenol Ethoxylates
- **OBT**: 2-hydroxybenzothiazole
- **OctaBDE**: Octabromodiphenyl ether (includes BDE-203)
- **OM**: Organic Matter
- **OP / OPE**: Octylphenol / Octylphenol Ethoxylates
- **P / P2O5**: Phosphorus / Phosphorus Pentoxide (Phosphate equivalent unit)
- **p.e.**: Population Equivalent (standardized wastewater plant sizing unit)
- **PAH / PAH 13 / PAH 16**: Polycyclic Aromatic Hydrocarbons (grouped testing sets)
- **Pb**: Lead
- **PBDE**: Polybrominated Diphenyl Ethers
- **PCB / i-PCB**: Polychlorinated Biphenyls / Indicator PCBs (PCBs 28, 52, 101, 118, 138, 153, 180, plus dioxin-like congener 169)
- **PCDD**: Polychlorinated dibenzo-p-dioxin
- **PCDF**: Polychlorinated dibenzofuran
- **PCN**: Polychlorinated Naphthalenes
- **PentaBDE**: Pentabromodiphenyl ether (includes BDE-99, BDE-100, BDE-47)
- **PFAS**: Per- and Polyfluoroalkyl Substances
- **PFBA**: Perfluorobutanoic acid
- **PFBS**: Perfluorobutane sulfonic acid
- **PFDA**: Perfluorodecanoic acid
- **PFDoDA**: Perfluorododecanoic acid
- **PFHpA**: Perfluoroheptanoic acid
- **PFHxA**: Perfluorohexanoic acid / Undecafluorohexanoic acid
- **PFHxS**: Perfluorohexane sulfonic acid
- **PFNA**: Perfluorononanoic acid
- **PFOA**: Perfluorooctanoic Acid
- **PFOS**: Perfluorooctane Sulfonate
- **PFPeA**: Perfluoropentanoic acid
- **PFUnDA**: Perfluoroundecanoic acid

- **PMC**: PubMed Central (Digital health/science journal archive)
- **PM2.5 / PM10**: Fine Particulate Matter fractions (sub-2.5 and sub-10 micrometers)
- **PNEC / PNEC Soil**: Predicted No Effect Concentration (in soil matrix environments)
- **POP**: Persistent Organic Pollutant
- **PPCPs**: Pharmaceuticals and Personal Care Products
- **PVC**: Polyvinyl chloride
- **QAC**: Quaternary Ammonium Compounds
- **REACH**: Registration, Evaluation, Authorisation and Restriction of Chemicals (EU Regulation)
- **S**: Sulfur
- **SBR**: Sequencing Batch Reactor
- **SCCP**: Short-Chain Chlorinated Paraffins (C10–C13)
- **t DS/year**: Tons of dry solids per year
- **TBT**: Tributyltin
- **TEF**: Toxicity Equivalent Factor
- **TEQ**: Toxic Equivalency / Toxicity Equivalent
- **TFT**: Trialkyltin derivatives / Tri-n-butyltin
- **TOC**: Total Organic Carbon
- **TS**: Total Solids
- **µg/kg DS**: Micrograms per kilogram / of Dry Solids
- **µg/kg BW/day**: Micrograms per kilogram of Body Weight per day
- **UKWIR**: United Kingdom Water Industry Research
- **UTILITALIA**: Federation of Italian water, environment, and energy utilities
- **UV / UV Filters**: Ultraviolet stabilizers / light blocking additives (e.g., UV-327, UV-328)
- **UWWTD**: Urban Wastewater Treatment Directive (Directive 91/271/EEC)
- **UWWTP / WWTP**: Urban Waste Water Treatment Plant / Waste Water Treatment Plant
- **VOC**: Volatile Organic Compound
- **VS**: Volatile Solids
- **WHO**: World Health Organization
- **WW**: Wet Weight (used in specific national sludge limit criteria)
- **Zn**: Zinc

1 EXECUTIVE SUMMARY

The Biosolids Observatory was established to address the critical need for updated, representative data on biosolids quality across the European Union. Biosolids are organic materials derived from urban wastewater treatment that are primarily managed in the EU through land application due to their benefits in nutrient recovery, carbon sequestration, and soil health. The main objectives of the Biosolids Observatory are to provide a set of reliable data to support risk assessment and to propose a structured approach to guarantee the quality of biosolids.

Scope and Methodology

- **Sample representation**: The campaign analyzed **30 representative samples** from eight EU countries: Italy, Spain, Sweden, Germany, Slovenia, Austria, Greece, and France.

- **Plant diversity**: Samples were collected from various Urban Waste Water Treatment Plant (UWWTP) sizes, categorised by population equivalent (p.e.), including plants <50,000 p.e. (23.3%), 50,000–250,000 p.e. (30%), and >250,000 p.e. (26.6%), as well as hub treatment sites (20%).

- **Quality assurance**: To ensure data reliability, the campaign utilized standardized sampling protocols (continuous and batch) and partnered with ISO/IEC 17025 accredited laboratories.

- **Substance selection**: Contaminants were selected for analysis based on a rigorous "Global Score" system that weighted risk contribution findings from existing scientific literature.

Key Findings: agronomic value

- **Soil improvement**: biosolids were confirmed as a source of organic matter, which improve soil structure, water retention, and microbial diversity. Regular biosolids land application can sequester up to 180 kg of Carbon per hectare per year.

- **Nutrient cycling**: biosolids provide essential nutrients (N, P) and secondary elements (Ca, S, Mg) to the plants.

- **Micronutrients**: biosolids are long-term sources of micronutrients like Zinc (Zn) and Copper (Cu).

Key Findings: Contaminants

- **Inorganic Elements**: Copper and Zinc are the most abundant, while high-risk elements like **Cadmium and Mercury remained consistently low** (averaging <1 mg/kg dry matter) and well below the limit values set by EU Directive 86/278/EEC

- **Organic Substances**:

  - **Alkanes**

  Short and medium chain chlorinated paraffins (SCCP & MCCP) which are mixtures of polychlorinated n-alkanes have been analysed in 23 biosolids samples. All the results were under the quantification limit of 500 µg/kg DS.

  - Bisphenols

  All bisphenol compounds were systematically detected. Bisphenol A and B were by far the most prevalent, with average values of 920 and 380 µg/kg DS, respectively.

  - Chlorobenzenes

  All chlorobenzenes compounds were systematically detected in the biosolids samples. The concentrations of monochlorobenzenes were very close to the quantification limit. For the other chlorobenzenes, the average concentrations were all very close to 0.05 mg/kg DS except for one sample.

  - Chlorophenols

  2,3,4,5-Tetrachlorophenol is the only compound that was detected in 2 biosolids samples (out of 29), with concentrations of 103 and 115 µg/kg DS, which is very close to the limit of quantification of 100 µg/kg DS.

  - Dioxins and furanes

  The concentration of dioxins and furanes compounds in biosolids were systematically above the quantification limits. The average Toxicity

Equivalent Factor (TEF) is 3.3 ng/kg DM with a maximum value is 14 ng/kg DM, which is lower than the strictest limit existing in EU Member States.

- Nonylphenol and nonylphenol ethoxylates

There is significant heterogeneity in the nonylphenol content of the analyzed samples. Three biosolids samples exceeded the strictest limit of 10 mg/kg DS set by the Danish EPA.

- Organotins

Monooctyltin, dioctyltin, and tributyltin are the organotins with the highest average concentrations, with 158, 123, and 101 µg/kg DS, respectively. For these three compounds, the maximum values represent between 10 and 20 times the average values.

- Polycyclic aromatic hydrocarbons PAHs

Sixteen different PAHs were analysed in 30 biosolids samples. All the results were above the quantification limit. Among the 30 samples tested, none was above the strictest limit set for 16 PAHs by the German EPA.

- Polybrominated diphenyl ethers (PBDEs)

Eighteen different PBDEs were analysed in 29 biosolids samples. Sixteen PBDEs were almost systematically detected. The PBDE with the highest average concentrations is by far PBDE 209 (0.3 mg/kg DS).

- Polychlorinated biphenyls PCBs

Most of the recorded values were close to the limit of quantification. Three samples alone account for the maximum recorded values. One sample shows relatively high concentrations of PCB 169 (0.3 mg/kg DS). Highest sum for PCB 28, 52, 101, 118, 138, 153, 180 is 0.068 mg/kg DS which is significantly lower than the most stringent regulatory limit of 0.2 mg/kg DS.

- Polychlorinated naphthalenes (PCNs)

The three PCNs (1-chloronaftalene, 2-cloronaftalene, 1,2,3,4-tetracloronaftalene) analysed in 23 biosolids samples were all under the quantification limit of 0.2 mg/kg DS.

- Per- and polyfluoroalkyl substances (PFAS)

FTCA5:3 was the most often detected PFAS but also the one with the mean and max concentrations, respectively of 14.2 and 80 µg/kg DS. PFOS was detected in half of the biosolids samples with an average concentration of 9.1 µg/kg DS while PFOA was detected in less than 20% of the samples with an average concentration of 6.8 µg/kg DS.The threshold limit value of 100 µg/kg DS for PFOS + PFOA set by Germany and Austria was systematically complied with for the different biosolids samples.

- Pharmaceuticals

Twenty three different pharmaceuticals were analysed in 30 biosolids samples, for all of which the detection limit was exceeded in all the biosolids samples. Families of pharmaceutical compounds with the highest average concentrations include antibiotics, antibacterials, hormones and antidepressants. Some pharmaceuticals have maximum values 10 to 20 times higher than average values.

- Phthalates

DEHP is the only phthalate present in significant concentrations with an average value of 11,9 mg/kg DS and a maximum value of 41,3 mg/kg DS. All samples are compliant with the most stringent limit of 50 mg/kg DS set by the Danish regulation.

- **Siloxanes**

Siloxanes were detected in more than two thirds of biosolids samples at average concentrations varying from 2 to 5 mg/kg DS.

- **Volatile organic compounds (VOCs)**

VOCs were systematically detected in all biosolids samples. Toluene and dichloromethane were, by far, the most abundant compounds, with average concentrations of 6 and 2,25 mg/kg DS, respectively. One sample exceeded the threshold limit of 100 mg/kg DS set for toluene by the Lombardy Region.

The waste water treatment plant size paradox: The results obtained by the Biosolids Observatory refute the notion that small facilities produce higher-quality biosolids. In fact, the minimum and average contaminant concentrations in biosolids are quite similar regardless of the size of the wastewater treatment plant. Only the contaminant concentrations in very large waste water treatment plants are significantly higher than those in the smallest plants.

Comparison with other biosolids analysis campaigns.

Overall, the results obtained are relatively comparable across the different analysis campaigns. However, those recorded during the FATE SEES campaign are significantly higher than those of the Biosolids Observatory for PAHs and PFAS. The concentrations of nonylphenol, phthalates, and toluene in the UTILITALIA analysis campaign are considerably lower than those of the Biosolids Observatory. The results of the analyses conducted by UKWIR differ significantly from those of the other campaigns for tributyltin, PFOS, ciprofloxacin, and triclosan. Bisphenol A is found in much higher quantities in Norwegian biosolids than in those of the Biosolids Observatory, unlike dioctyltin and tributyltin, which are present in lower concentrations.

Strategic recommendations and action plan

- **identification of priority contaminants to be monitored**

  The soil PNEC (no predicted effect concentration) and the mammalian NOAEL (no observed adverse effect dose) as well as contaminant concentrations in biosolids, were used to identify priority contaminants to be monitored which are chlorobenzene's,chlorophenol’s, organotins, PBDE's, PCB’s, PFAS and phthalates. The initial analyses of biosolids for these substances will be used to determine the analysis frequency for subsequent years.

- **Standardisation**: standardisation of analytical protocols for sample preparation, contaminant extraction and analysis are mandatory to ensure data comparability and regulatory confidence.

- **Source control**: Source control: It is essential to take advantage of EU regulatory tools such as REACH, the Industrial Emissions Directive (IED) and the recast Urban Wastewater Treatment Directive (UWWTD) to 'remove' contaminants before they reach the sewer system.

- **Risk-based action plan**: A tiered response is recommended based on analytical results compared to those of the Biosolids Observatory analysis results percentiles:

  - **<25th percentile**: Maintain status quo; monitor new industrial discharges.

  - **25th -50 th percentile**: take an inventory of industrial discharges and identify potential contaminant sources.

- **50th – 75th percentile**: Active identification of industrial discharge sources must be undertaken and the implementation of pre-treatment at the source is necessary.
- **>75th percentile**: It is urgent to implement an action plan quickly to limit contaminant concentrations in sewage sludge. Industrial discharges that compromise sludge quality must be identified, and the installation of pretreatment facilities is essential.

Detailed data can be obtained upon request by sending an email to biosolidsobservatory@gmail.com.

2 INTRODUCTION

2.1 Purpose of the Biosolids Observatory

Biosolids are defined by the International Standard Organisation (ISO) as organic-based materials from industrial or municipal wastewater sludge and their derived products, in the form solids, semi-liquid (pasty), and liquid which have been treated to meet jurisdictional standards, guidelines or requirements including the reduction of pathogens, vector attraction and contaminant criteria.

Biosolids land application is currently, and from far, the major route in the EU for sludge management [1]. This solution has many advantages such as nutrient recovery, carbon sequestration and the improvement of soil production capacity. Biosolids land application contributes to sustainable agriculture and the preservation of ecosystems, while participating in climate regulation.

Nevertheless, biosolids may contain contaminants due to domestic, commercial and industrial discharges into the sewer system [2]. Due to legitimate concerns regarding the protection of human health and the environment it is essential to have an updated overview of the biosolids quality at European Level.

The purpose of the Biosolids Observatory is to collect, package, transport and analyse representative samples of biosolids coming from various sewage treatment plants located in different EU countries. The resulting data can be used to feed risk assessment studies but also to better understand the potential impacts of the revision of national or EU regulations for biosolids land application.

Regardless of this regulatory aspect, it is important to stress that public and private entities involved in biosolids land application are committed to supply the farmers with a product that can be safely used as a fertiliser or soil improver. This liability requires a detailed knowledge of the biosolids composition. Analysis of biosolids, covering a wide range of compounds, allows to assess whether their concentrations in those compounds are close to background, average, or high levels. Based on the results obtained, it is possible to determine whether measures are necessary to reduce, at the source, the compounds that impair biosolids quality.

The Biosolids Observatory presentation brochure is presented in Annex A.

2.2 The Biosolids Observatory Steering Committee

The Steering Committee includes participants of professional associations (at national or EU level) that are willing to promote the Biosolids Observatory project..

These are:

- **Aqua Publica Europea (APE),** the European Association of Public Water Operators, unites publicly owned water and sanitation services and other stakeholders working to promote public water management at both European and international level. APE is an operator-led association that looks for efficient solutions that serve public interests rather than corporate ones.

- **EurEau,** the European Association of Water Services, represents national drinking and waste water service providers from 33 countries, from both the private and the public sectors. EurEau brings together water professionals to agree on European water industry positions regarding the management of water quality, resource efficiency and access to water and sanitation for citizens and businesses.

- **EUROPEAN FEDERATION FOR AGRICULTURAL RECYCLING (EFAR)** gathers and represents companies experienced in biosolids land application. EFAR members establish and maintain a direct link with farmers and biosolids producers. They ensure the processing and quality control of biosolids. They optimize the beneficial use of biosolids through the establishment of nutrient management plans and make sure that all health and environmental requirements are properly addressed in spreading areas. They guarantee full compliance with European, national, and local regulations.

The role of the Steering Committee is presented in the Biosolids Observatory brochure (Annex A)

2.3 Substances selection

A number of risk assessments have been conducted over the past two decades [3] [4] [5] [6] to establish the impact on human health and the environment of contaminants contained in biosolids.

The main assessments were reviewed, and their conclusions were used to classify the overall risk contribution of each contaminant according to the following classification:

- no data = 0
- low risk contribution = 1
- medium risk contribution = 2
- high risk contribution = 3

An overall score for each contaminant was calculated by summing its number of occurrences in the various publications after multiplying by the risk contribution factor.

For example, if a contaminant appears in one publication as contributing slightly to the risk, in three publications as contributing moderately to the risk, and in five publications as contributing highly to the risk, the overall score is (1*1)+(3*2)+(5*3)=22. Results for the different groups of contaminants are presented in Table 1 below.

Table 1 substances selection grid

Contaminant	High risk contribution	Medium risk contribution	Low risk contribution	Global Score
Nonylphenol, nonylphenol ethoxylates and other	8	1	1	27
Triclocarban	7	1	1	24
Triclosan	7	1		23
Brominated flame retardants (PBDE)	3	3	3	18
Pharmaceutical residues	3	3	2	17
PAH 13	2	2		10
Organotin compounds (TBT, TFT, DBT, DOT)	1	3	1	10
Perfluorinated alkyl substances (Bisphenols)	2	1	1	9
Surfactants (LAS)	2	1		8
Benzothiazoles : 2-mercaptobenzothiazole (MBTthiazole)and 2-hydroxybenzothiazole (OBT)	2	1		8
Siloxanes	1	1	2	7
Musk compounds (galaxolide, tonalide, etc.)	1	1	1	6
Alkylphenols	1	1		5

Table 1 substances selection grid

Contaminant	High risk contribution	Medium risk contribution	Low risk contribution	Global Score
Polychlorinated biphenyls (PCB)	1		2	5
Bisphenols	1	1		5
Caffeine	1	1		5
Zinc	1		1	4
Mercury		1	1	3
Copper		1	1	3
Polychlorinated naphthalenes	1			3
Polychlorinated Alkanes	1			3
Dioxin			2	2
Phthalate (DEHP and DBP)			1	1
Cadmium			1	1
Lead			1	1
Nickel			1	1
Arsenic				0
Silver				0
Organophosphorus flame retardants				0
UV substances				0
Chlorinated paraffins				0
BTEX, trichlorethylene, and other chloro-organic compounds				0
Ethinylestradiol				0

Results of the evaluation grid were submitted to the Biosolids Observatory Steering Committee for validation. Subsequently, the Steering Committee defined the three analysis packages:

- **Basic package** : Nutrients, Inorganic contaminants, microplastics, PAH, Bisphenols, PBDE, NP and NPE, Organotins, Chlorobenzenes, Chorophenols, Pharmaceuticals.
- **Additional package 1**: Bisphenols, PCB, Miscellaneous compounds (31) mostly used as solvents.
- **Additional package 2**: Phthalates, Dioxins and Furans, PCN, Siloxanes, Chloroalkanes.

3 SAMPLING AND ANALYSIS CAMPAIGN

3.1 Origin of the samples sent for analysis

3.1.1 Geographical origin

The samples analyzed come from the countries listed in Table 2.

Table 2 : Geographical origin of biosolids samples

Country	Sample number
Italy	10
Spain	8
Sweden	5
Germany	2
Slovenia	2
Austria	1
Greece	1
France	1
Total	30

3.1.2 Type of biosolids production sites

The biosolids samples are evenly splitted between the different sizes of Urban Waste Water Treatment Plant (UWWTP) as indicated in table 3.

Table 3 : Type of biosolids production sites

Type of biosolids production site	Number of sites
WWTP < 50 000 pe	7
50 000 < WWTP < 250 000 pe	9
WWTP > 250 000 pe	8
Biosolids hub treatment site	6

3.1.3 Urban waste water treatment type

In UWWTP wastewater is treated by physical, biological and chemical processes to remove minerals, organic matter, nutrients and contaminants. The different types of treatment are divided into the following four categories:

- primary treatment: solids removal
- secondary treatment: organic matter removal
- tertiary treatment: removal of nutrients
- quaternary treatment: removal of micropollutants

The wastewater treatment plants participating in the biosolids observatory systematically operate primary and secondary treatments.Two-thirds of them use tertiary treatment. Quaternary treatment is applied in a limited number of plants.

Table 4 : Wastewater treatment type

Waste water treatment	% Plants
Primary Treatment	100.00%
Secondary Treatment	100.00%
Tertiary Treatment	75.00%
Quaternary Treatment	16.67%

3.1.4 Sludge post treatment

Sludge is digested on 79% of the UWWTP.
Additionally the following treatments are applied for stabilisation and pathogens reduction.

Table 5 : Sludge post treatment

Sludge treatment	Plant number
Lime mixing	7
Composting	5
Long term storage	3
None	15

3.1.5 Biosolids yearly production

The average biosolids production per site is 21 000 tonnes wet weight with a minimum of 880 tonnes and a maximum of 166 000 tonnes. Average dry matter content is 24%.

3.1.6 Biosolids final destination

From the feedback of the Biosolids Observatory participants, land application is the main biosolids outlet. 20% of the WWTP have multi outlet solutions for their biosolids disposal (thermal treatment, landfilling) and recovery (land application).

Table 6 : Biosolids destination

Biosolids destination	Number
Land application	22
Thermal treatment	2
Multi outlet	6

3.2 Sampling methodology

Proper sampling is crucial for the quality of results. Therefore, a procedure describing in detail the sampling conditions and associated logistics was sent to the participants in the Biosolids Observatory (see Annex C).

3.2.1 Sampling equipment

Participants were requested to use sampling equipment consisting of a trowel or trowel and a bucket with a minimum capacity of 10 liters and a lid, both made of stainless steel.

It was specified that this sampling equipment must be cleaned and rinsed before the sampling session and be free of rinse water to avoid any impact on the sample (no risk of contamination or dilution).

3.2.2 Sampling methodology

It was reiterated that the sample collected and analyzed must be representative of the biosolids production and contain at least 15% dry matter to ensure the laboratories have a sufficient quantity of material to perform all the planned analyses.

Participants could choose to collect a sample with a minimum volume of 6 liters:

- continuously during biosolids production, or
- from a previously produced batch (for example, a compost or lime

treated cake stockpile).

Batch sampling was requested whenever possible. The detailed conditions for sample collection are presented hereafter.

3.2.2.1 Continuous sampling

To obtain a representative sample of the biosolids production, a minimum of 25 elementary samples must be taken evenly distributed during a predetermined period of not less than 5 days.The size of each elementary sample must be identical.

These elementary samples are placed and mixed in the 10 litre stainless steel bucket; the latter has to be kept near the sampling point with the lid on for the duration of the sampling campaign. A warning sign has to be placed on the lid to specify that the bucket is strictly and solely used for biosolids sampling and that the lid must be kept on.

3.2.2.2 Batch sampling

To obtain a representative sample of the batch to be analysed a minimum of 25 elementary samples must be taken at different points and different depths of the biosolids stockpile.
The collection of elementary samples must be evenly distributed throughout the batch at a minimum depth of 20 cm of the surface and in areas where water accumulation has not occurred.
The size of each elementary sample must be identical. These elementary samples are placed and progressively thoroughly mixed in the 10 litre stainless steel bucket.

3.2.3 Logistics

Within 10 business days of order confirmation by the participants, they were provided with a cooler containing six one-liter glass jars and ice packs,. together with the shipping label and a reminder of the sampling procedure. Halfway through the sampling campaign, participants were asked to send the laboratory a request for collection on a pre-defined date. It was requested that the glass jars be filled the day before the scheduled collection date to minimize alteration of the sample composition and reduce the risk of gas buildup. These jars were then placed in the cooler along with the pre-frozen ice packs and a copy of the order form.

3.3 Partnering laboratories

3.3.1 Laboratory selection

A consultation and selection process for partner laboratories was established. The requirements set by the steering committee focused on the candidates' ability to justify:

- their recognized expertise in the analysis of contaminants in organic matrices;
- the scope of their accreditations: ISO/IEC 17025 : 2017;
- their ability to guarantee traceability of the analytical chain, from sampling to the final result;
- their capacity to collect biosolids samples at production sites located all over EU Member States and their rapid transportation to the laboratory;
- the achievement of the lowest possible limits of detection and quantification.

3.3.2 LANAE

LANAE is a French independent laboratory specializing in high value-added environmental analyses. It supports public and private entities in the context of technical, regulatory, and standards-related monitoring of organic waste and products that are spread on land. LANAE has developed significant skills to test organic contaminants in biosolids.

3.3.3 EUROLAB WHITE LAB

White Lab is the largest Italian network of laboratories. Their activities are dedicated to the protection of consumer health thanks to a wide range of tests and consultancy services for companies in the food, environmental, medical, consumer goods, engineering and automotive industries. White Lab has a specific expertise in testing organic contaminants and pharmaceuticals in biosolids.

3.4 Analysis methods

Details of analysis methods are provided in Annex D.

4 ANALYSIS RESULTS

For the calculation of the values presented below (minimum, maximum, percentiles, mean), and when the results were below the limits of quantification, the latter was used, which is a very conservative position.

4.1 Parameters of agronomic interest

4.1.1 General data on parameters of agronomic interest

4.1.1.1 Organic matter

Organic matter, which is the major component in biosolids, is primarily composed of excess microorganisms produced by the treatment of wastewater. This organic matter consists of a wide variety of substances, including proteins, carbohydrates, humic substances, lipids, lignins, and organic acids. Depending on the type of treatment applied to the biosolids, a greater or lesser proportion of this organic matter will contribute to the formation of stable humus in the soils.

Humic acids are essential components of organic matter and play a crucial role in soil health and plant growth. By binding mineral and organic particles, they improve soil structure and facilitate water and air retention. This also increases the permeability of clay and sandy soils, while optimizing water and nutrient management. Furthermore, their ability to increase the soil's cation exchange capacity (CEC) facilitates the release and availability of essential elements for plants, making them more accessible to roots. By stimulating root growth, humic acids enhance nutrient absorption, improve plant resistance to water stress, and increase their defenses against diseases and pests such as nematodes. Furthermore, they play a protective role by reducing the bioavailability of heavy metals and organic pollutants, thus limiting the contamination of crops and food chains.

Humic acids also have the capability to bind trace elements and organic substances and, therefore, play a key role in soil and water remediation. In soils, they reduce the mobility and toxicity of these substances while facilitating their natural degradation.

The organic matter in biosolids directly supports soil biological health by stimulating microbial communities and acting as a biostimulant for plants. The carbon-rich organic material serves as an energy source for soil microbes, enhancing microbial biomass, enzyme activity, and overall microbial diversity.

Regular spreading of digested biosolids can increase soil organic carbon of 180 kg C ha−1y−1tds−1 [7]. A significant contribution from sequestered carbon also comes from increased soil biological activity and the stimulation of their native biomass (microflora, microfauna, microorganisms). Depending on the characteristics of Biosolids and of the application site the carbon retention coefficients can vary from 10 to 50% [8].

4.1.1.2 Major nutrients

Nitrogen

Nitrogen is an essential element for plants because it is a component of chlorophyll molecules. It is also a constituent of vitamins and amino acids, and participates in the formation of proteins, a quality criterion for many agricultural products.

The majority (50 to 90%) of the nitrogen contained in biosolids is in organic form and must be mineralized into inorganic nitrogen that plants can assimilate before it can be absorbed by crops. Microorganisms decompose organic matter, releasing inorganic nitrogen into the soil.

Depending on the characteristics of the biosolids and the receiving soils, the amount of nitrogen available to crops can vary from 10 to 70% [9] [10] [11].

Phosphorus

Phosphorus promotes root development and early seedling growth. Plants growth factors, such as stalk and stem strength, flower formation and seed production, are associated with phosphorus supply.

Wastewater treatment processes influence phosphorus solubility. The fraction of plant available phosphorus in biosolids captured via biological removal is higher than in biosolids from chemical precipitation where iron or aluminium salts have been used.
However phosphorus in biosolids is present primarily (70 to 80%) in mineral form, often in chemical forms similar to those of mineral fertilizers [12]. Therefore, the availability of phosphorus from biosolids to plants is relatively independent of the mineralization rate of organic forms of phosphorus in the soil. It is essentially determined, as with phosphorus from mineral fertilizers, by the physicochemical conditions of the environment (particularly soil pH), which regulate the mineral form of phosphorus, its solubility (higher in acidic environments), its adsorption and desorption onto soil particles, etc.

Potassium

Potassium stimulates the activity of numerous enzymes involved in photosynthesis. It contributes to cellulose synthesis and facilitates the transport of sugars and starch. Potassium has a significant impact on crop quality factors such as size, shape, color, taste, shelf life, and fiber content. Potassium also promotes root growth and improves drought tolerance.

In biosolids, potassium is entirely present in soluble mineral form, making its availability equivalent to that of mineral fertilizers.

4.1.1.3 Secondary nutrients

Calcium

Calcium indirectly contributes to increased yields by improving root growth conditions and stimulating microbial activity, as well as the availability and absorption of other nutrients. It also has a significant impact on important physical soil properties such as aggregate stability and porosity. Therefore, calcium promotes root development, improves permeability, and limits the formation of surface crusts.

Biosolids can be a significant source of calcium, particularly when stabilized with lime. These biosolids can be used to correct the pH of acidic soils, with a liming capacity equivalent to 30 to 70% of pure agricultural lime (based on calcium equivalent) [13] [14] [15].

Sulphur

Sulfur is an essential element for the synthesis of certain amino acids (cysteine and methionine) and proteins. It also plays an important role in photosynthesis and the resistance of crops to winter cold. Sulfur promotes seed production.

Organic sulfur must be mineralized by heterotrophic microorganisms before it can be assimilated by plants.

The availability of sulfur in biosolids varies depending on the wastewater and sludge treatment processes. The highest plant-available sulfur content is found in biosolids stabilized in an alkaline environment (52%), followed by anaerobically digested biosolids (40%), lagoon sludge (36%), aerobically digested biosolids (15%), and composted biosolids (4%). The pH of biosolids, the organic C/S ratio, and the concentrations of total Ca, total S, and SO_4-S are strongly correlated with the rate of sulfur release, the plant-assimilable sulfur content, or both [16] [17].

Magnesium

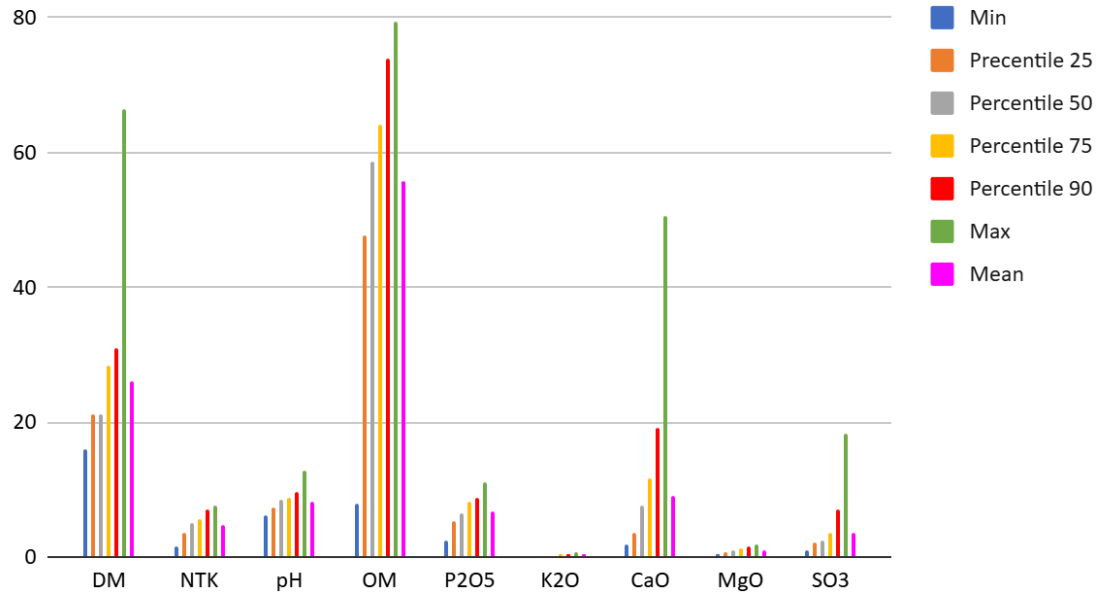
Magnesium has a diverse and critical role in various plant functions. These functions, including photosynthesis, transportation of carbohydrates to different growing tissues (roots, shoots and reproductive organs), and stress-tolerance mechanisms. Magnesium availability in biosolids depends on its chemical form, treatment process, and soil conditions after land application. The short-term availability of magnesium from biosolids to the plant varies from 20 to 50% of the total magnesium for the year following application. Long-term availability, on the other hand, increases over time as the organic magnesium is mineralized [18].

4.1.1.4 Micronutrients

Biosolids are a long-term source of micronutrients in agriculture, particularly for zinc and copper. Micronutrients supplied in biosolids are naturally chelated by organic matter and less subject to leaching. Micronutrients availability ranking in biosolids is generally as follows : Zn ≈ Cu > Mn > Fe > B > Mo, considering that the first year after land application 10–30% of Zn and Cu may be plant-available [19] [20].

4.1.2 Biosolids observatory results for parameters of agronomic interest

Figure 1 : Nutrients content in % on DS



The average dry matter content is close to 25%, and this contains approximately 55% organic matter.
The average levels of major nutrients are 6.6% for phosphorus (expressed as P_2O_5) and 4.7% for nitrogen. Unsurprisingly, potassium concentrations are much lower, representing only 0.3% of the dry matter (expressed as K_2O). Calcium concentrations and pH vary considerably, with the highest values corresponding to lime-treated sludge. Sulfur levels are far from negligible, reaching 3% of the dry matter (expressed as SO_3).

4.2 Inorganic trace elements

4.2.1 General data on inorganic trace elements

Some of the chemical elements in soil are referred to as trace elements because of their occurrence at concentrations less than 100 mg/kg. Part of these elements, such

as manganese (Mn), iron (Fe), zinc (Zn), boron (B), chromium (Cr), Nickel (Ni) copper (Cu) and molybdenum (Mo) are micronutrients and are essential for plants and/or animals including human beings. Other elements like lead (Pb), cadmium (Cd); mercury (Hg) have to be considered as contaminants in the strict sense. Most of these elements can represent a threat for the environment and for human health if their concentrations in soils surpass certain thresholds.

4.2.2 Inorganic trace elements in the environment

Trace elements are naturally present in soils due to the nature of their parent material (rocks) [21] [22] [23]. Their concentrations vary significantly as presented in Table 7.

Table 7 : Soils background content in inorganic trace elements

	Cu	Cr	Cd	Pb	Zn	Ni	Hg
Min	0.2	0.3	0.01	2.2	0.5	0.1	0.01
Max	107	690	6.3	3000	3820	478	0.1
Median	13	66	0.2	34	80	31	0.04

Trace elements can enter the soil by a number of pathways, and their behaviour and fate differ according to their source and species. The most important anthropogenic sources of trace elements are [24]:

- Atmospheric deposition, arising from coal and petroleum base products, metal mining, smelting and manufacturing, waste incineration, production of phosphate fertilizers and cement, and wood combustion;
- Land application of biosolids, animal manure and other organic wastes and by-products from agriculture and food industries;
- Fertilizers, lime, pesticides and irrigation water used for food crop production.

From an overall quantitative perspective, animal manure, mineral fertilizers, plant protection treatments and atmospheric deposition are the major sources of trace metal inputs [25].

Animal manure plays a significant role in the input of trace elements into agricultural soils. Significant flows of copper and zinc originate from animal feeds which are generally supplemented with these elements, resulting in their excretion in manure. 40% to 50% of total annual Cu inputs to EU agricultural soils come from animal manure. In regions with intensive pig farming (e.g., parts of the Netherlands, Belgium, and Denmark), this figure can exceed 80%. Furthermore, the use of copper as a fungicide, particularly in organic farming, can represent up to 34% of the input of this element into agricultural soils. Similarly to copper, 35% to 50% of total Zn input to soil comes from pig and poultry manure [26].

Phosphate fertilizers represent a significant source (between 45 and 55% on average) of global cadmium inputs to agricultural soils [27], [28].

Calcium and magnesium amendments play a minor role in the input of trace elements into soils (on the order of 1 to 5%).

Atmospheric deposition also represents a significant source of diffuse contamination, representing 3% for Cu and Zn to 33% for Hg and Pb of total emissions, depending on the trace elements, and most often in the range of 10-15%. Sources of trace elements in the atmosphere can be natural (wind erosion, volcanic activity, sea spray, and forest fires) or anthropogenic (energy production, industrial processes, incineration plants, road traffic, etc.).

Biosolids and compost account for a relatively small share of the total amount of trace elements entering agricultural soils (around 4-8%), except for mercury (17%) and lead (20%) [29].

Trace elements in biosolids come from:

- Households connection to the sewer containing human faeces (average metal content of the latter, in mg/Kg DS : Zn = 250; Cu = 68; Pb = 11; Ni = 4.7; Cd = 2), cosmetic, medical and cleaning products;
- Rainwater runoff on roofs, roads, and generally on all impermeable surfaces in the city, leads to the enrichment of the effluent with lead, zinc, and nickel;
- Commercial activities such as garages, laundries and dry cleaners, restaurants, medical laboratories, hospitals and industrial activities that have the permit to discharge (with or without pretreatment) their effluent into the sewer.

The overall data mentioned above relates to global trace element fluxes at EU or Member States levels. At the plot of land level, the values obtained are significantly different. This is why some national regulations have set maximum inputs of inorganic trace elements in g/ha/year associated with biosolids land application.

4.2.3 Legislative framework

The Directive 86/278/EEC on the protection of the environment, and in particular of the soil, when sewage sludge is used in agriculture, sets rules on how farmers can use sewage as a fertiliser to prevent it from harming the environment and human health, by ensuring that the nutrient needs of the plants are considered and that the quality of the soil and of the surface and groundwater is not impaired. To this end, it sets limit values on the concentrations allowed in biosolids and soils for trace elements.

Table 8 : Limit values for inorganic trace elements in biosolids in mg/kg DS [30].

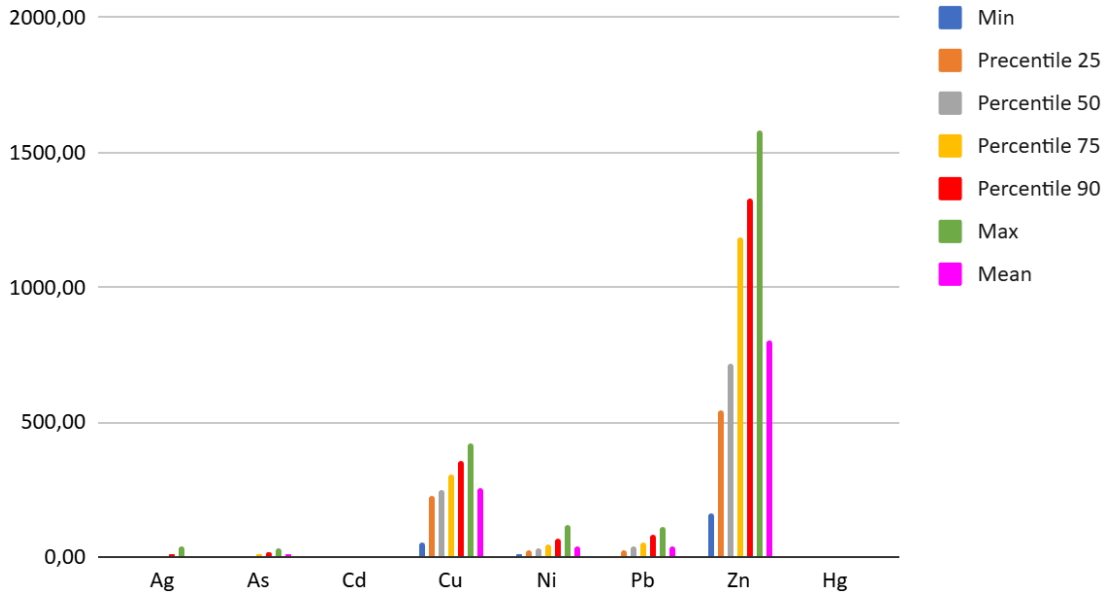
Legislation	Trace Elements							
	Cd	Cr	Cu	Hg	Ni	Pb	Zn	As
Directive 86/278/EEC	20–40	-	1000–1750	16–25	300–400	750–1200	2500–4000	-
Germany	1.5	1 (Cr VI)	900	1	200	150	4000	40
United Kingdom	-	-	-	-	-	-	-	2
Spain	20–40	1500	1000–1750	16–25	300–400	750–1200	2500–4000	-
France	20	1000	1000	10	200	800	3000	-
Italy	20	200	1000	10	300	750	2500	20
The Netherlands	1.25	75	75	0.75	30	100	300	15
Austria	2–10	50–500	70–500	0.4–10	25–100	45–500	200–2000	20
Sweden	0.75	40	300	1.5	25	25	600	-
Portugal	20	1000	1000	16	300	750	2500	-
Finland	1.5	300	600	1	100	100	1500	25
Denmark	0.8	100	1000	0.8	30	120	4000	25
Ireland	20	-	1000	16	300	750	2500	-
Greece	20–40	500	1000–1750	16–25	300–400	750–1200	2500–4000	-
Belgium	6–10	100–150	600–800	1–1.6	100	300–500	1500–2000	20–150
Luxembourg	2.5	100	700	1.6	80	200	3000	-
Poland	20	500	1000	16	300	750	2500	-
Hungary	10	1000	1000	10	200	750	2500	75
Czech Republic	5	200	500	4	100	200	2500	30
Romania	10	500	500	5	100	300	2000	10
Lithuania	1.5–20	140–400	75–1000	1–8	50–300	140–750	300–2500	-
Slovakia	10	1000	1000	10	300	750	2500	20
Bulgaria	30	500	1600	16	350	800	3000	25
Estonia	20	1000	1000	16	300	750	2500	-
Cyprus	20–40	-	1000–1750	16–25	300–400	750–1200	2500–4000	-
Latvia	2–10	100–600	400–800	3–10	50–200	150–500	800–2500	-
Slovenia	1.5	200	300	1.5	75	250	1200	-
Malta	5	800	800	5	200	500	2000	-
Croatia	5	500	600	5	80	500	2000	-

Table 9 : Limit values for inorganic trace elements in soils in mg/kg DS [30].

Legislation	Cd	Cr	Cu	Hg	Ni	Pb	Zn	As
Directive 86/278/EEC	1–3	-	50–140	1–1.5	30–75	50–300	150–300	-
Germany	0.4–1.5	30–100	20–60	0.1–1	15–70	40–100	60–200	-
United Kingdom	3	400	80–200	1	50–110	300	200–300	50
Spain	1–3	100–150	50–210	1–1.5	30–112	50–300	150–450	-
France	2	150	100	1	50	100	300	-
Italy	1.5	-	100	1	75	100	300	-
The Netherlands	0.8	10	36	0.3	30	35	140	-
Austria	0.5–2	50–100	40–100	0.2–1.5	30–70	50–100	100–300	-
Sweden	0.4	60	40	0.3	30	40	100	-
Portugal	1–4	50–300	50–200	1–2	30–110	50–450	150–450	-
Finland	0.5	200	100	0.2	60	60	150	-
Denmark	0.5	30	40	0.5	15	40	100	-
Ireland	1	-	50	1	30	50	150	-
Greece	1–3	-	50–140	1–1.5	30–75	50–300	150–300	-
Belgium	1.2–1.5	91–100	50–72	1–1.5	20–56	50–120	200	22
Luxembourg	2	150	100	1.5	75	200	300	-
Poland	1–3	50–100	25–75	0.8–1.5	20–50	40–80	80–180	-
Hungary	1	75 (CrVI)	75	0.5	40	100	200	15
Czech Republic	0.4–0.5	55–90	45–60	0.3	45–50	55–60	105–120	15–20
Romania	3	100	100	1	50	50	300	-
Lithuania	1–1.5	50–80	50–80	0.6–1	50–60	50–80	160–260	-
Slovakia	1	60	50	0.5	50	70	150	25
Bulgaria	1.5–3	200	80–200	1.5	75–110	60–120	200–300	25
Estonia	3	100	50	1.5	50	100	300	-
Cyprus	1–3	-	50–140	1–1.5	30–75	50–300	150–300	-
Latvia	0.5–0.9	40–90	15–70	0.1–0.5	15–70	20–40	50–100	-
Slovenia	1	100	60	0.8	50	85	200	-
Malta	0.5–1.5	30–100	20–100	0.1–1	15–70	70–100	60–200	-
Croatia	0.5 – 1.5	50–100	40–100	0.2–1	30–70	50–100	100–200	-

4.2.2 Biosolids Observatory results for inorganic trace elements

Figure 2 : Inorganic trace elements concentrations in mg/kg DS



Copper and zinc are the most abundant trace elements, with average concentrations of 250 and 800 mg/kg DS respectively. Cadmium and mercury levels, on the other hand, are less than 1 mg/kg DS.

The following table compares the maximum values obtained within the framework of the Biosolids Observatory with the limit values set by the Directive 86/278/EEC.

Table 10: Comparison of maximum concentrations of inorganic trace elements in samples analyzed within the framework of the Biosolids Observatory with the limit values set by Directive 86/278

	Biosolids Observatory Max in mg/kg DS	Limit value Directive 86/278/EEC in mg/kg DS	Ratio
Cd	2,20	20,00	11,00%
Cu	424,00	1000,00	42,40%
Ni	122,00	300,00	40,67%
Pb	113,00	750,00	15,07%
Zn	1580,00	2500,00	63,20%
Hg	1,90	16,00	11,88%

All the limit values set by the Directive 86/278/EEC are met by far.Furthermore, regarding cadmium, the European fertiliser regulation has set a maximum flux of 3.6 g per hectare for an application of 60 kg of P2O5. Based on average cadmium values recorded by the biosolids observatory, the cadmium flux associated with an application of 60 kg of P2O5 per hectare through biosolids land application is only 0.75 g.

4.3 Organic substances

4.3.1 Alkanes

4.3.1.1 General data on Alkanes

Chlorinated paraffins (CP) are mixtures of polychlorinated n-alkanes formed through the chlorination of paraffin fractions under conditions of high temperature and/or ultraviolet irradiation. These mixtures contain congeners that differ based on carbon chain length, chlorination level, chlorine position on the chain, and carbon atom stereochemistry. The number of possible isomers is greater than 10,000. Chlorinated paraffins are generally divided into three groups based on their carbon chain lengths: short-chain (SCCPs, C10–13), medium-chain (MCCPs, C14–17), and long-chain (LCCPs, C≥18).

Short-chain (SCCPs) and medium-chain chlorinated paraffins (MCCPs) are used as flame retardants in the clothing and footwear industry, and also in plasticizers in plastics, rubbers, inks, paints, adhesives, and surface coatings. In addition, these compounds are used as additives in metalworking, and as lubricants or coolants in metal cutting and forming. While the use of SCCP is strictly limited, MCCP is widely used in PVC production.

4.3.1.2 Alkanes in the environment

There are no known natural sources of polychlorinated alkanes. Polychlorinated alkanes can enter into soils via atmospheric deposition, biosolids land application and wastewater irrigation. Their presence in biosolids can be attributed to the discharge in the sewer system of landfill leachates or of effluent originating from industrial sites producing or using products containing polychlorinated alkanes in their process.

A limited number of analysis campaigns have been undertaken to determine polychlorinated alkanes soil background concentrations in Europe. The limited existing data suggests that MCCP and SCCP concentrations are in the same range of circa 10 ng/g [31]. The predicted overall persistence of polychlorinated alkanes ranged from 503 to 519 days in soils and sediments [32].

4.3.1.3 Legislative framework

In 2017, SCCPs with a chlorine content greater than 48% by weight were listed under the Annex A to the Stockholm Convention on POPs. The Stockholm

Convention requires the control, reduction and, ultimately, the elimination of POP production, commercialization, use and release. Additionally, the content of SCCPs (C10–C13 > 48% Cl) in other CP mixtures was limited to 1% by weight, meaning that CPs with a SCCP content ≥ 1% by weight are also considered POPs and need to be treated accordingly. Subsequently, the Basel Convention on controlling transboundary movements of hazardous wastes and their disposal, proposed two tentative low POP content (LPC) limits (provisional limits of 100 mg kg−1 and 10 000 mg kg−1) for the classification of products that shall be destroyed or irreversibly transformed in an environmentally sound manner. However, SCCPs benefit from several specific exemptions and can still be used in certain products.

More recently, MCCPs have been proposed for inclusion in the Stockholm Convention on Persistent Organic Pollutants. However, the current restrictions on the use of SCCPs are expected to result mainly in a replacement by MCCPs in many applications.

At EU level, the Commission Regulation (EU) 2015/2030 included SCCP Annex I to Regulation (EC) No 850/2004. This means that SCCPs, production, placing on the market and use, whether on their own, in preparations or as constituents of articles, shall be prohibited, with the exception of a few types of use. Furthermore, Regulation (EU) 2019/1021 on persistent organic pollutants, introduced new specifications for the treatment of waste containing SCCPs.

In response to these limitations, chlorine-free paraffin alternatives such as alkylphosphates and sulfonated fatty acid esters are used for specific applications. Natural animal, vegetable, and mineral oils are used as substitutes in leather production. Polyacrylic esters and phosphates are used in paint and coating applications, while aluminum hydroxide, antimony trioxide, acrylic polymers, and phosphate-containing compounds are used as flame-retardant alternatives.

There are currently no regulations setting limits for alkanes in biosolids at EU level. Nevertheless, some countries have set limits at national level: Hungary has set a limit of 4000 mg kg/DS for C5-C40 alkanes, while Italy has set a limit for these compounds of 1000 mg/kg WW.

4.3.1.4 Biosolids Observatory results for alkanes

SCCP and MCCP have been analysed in 23 biosolids samples. All the results are under the quantification limit of 500 µg/kg DS.

4.3.2 Bisphenols

4.3.2.1 General data on bisphenols

Bisphenols are compounds used in the industrial manufacture of plastics, either as monomers in polycarbonate or as additives in epoxy resins. They form a large family of substances with similar chemical structures and uses. Among the best known are bisphenol A (BPA) and bisphenol S (BPS).

Polycarbonates are used to manufacture a wide range of everyday objects (laptops, cell phones, greenhouse enclosures, automobile headlights, outdoor fixtures, eyeglasses, plastic bottles, food containers and medical industry applications. Polycarbonates are used in the composition of epoxy resins that line the interior of cans and tins, and in the manufacture of dental amalgams. They help preserve the taste of food and protect it from microbiological contamination. Bisphenols are also used as additives in the manufacture of heat-sensitive papers.

Global production in 2022 was estimated to be in the region of 10 million tonnes [33].

BPA's largest single application is as a co-monomer in the production of polycarbonates, accounting for 65–70% of all BPA production. The manufacturing of epoxy resins and vinyl ester resins account for 25–30% of BPA use. The remaining 5% is used as a major component of several high-performance plastics, and as a minor additive in polyvinyl chloride (PVC), polyurethane, thermal paper, and several other materials.

4.3.2.2 Bisphenols in the environment

Environmental sources of BPA can be classified as preconsumer and postconsumer products. Preconsumer sources include those attributed to the production of BPA and BPA-containing products, where the first source of BPA release is from effluent discharge of manufacturing plants. Processing of BPA-containing products are additional sources for preconsumer release.
Postconsumer sources include those associated with disposal or waste including effluent discharge from municipal wastewater treatment plants, leaching from landfills, combustion of domestic waste, and degradation of plastics in the environment. Primary sources of BPA to soils include the land application of biosolids, irrigation with wastewater effluent, discharge of landfill leachate, disposal and recycling of electronic waste or use of pesticides.

Due to the low volatility of BPA, low air emissions, and rapid photo-oxidation half-life (<7 hours), BPA is expected to have a negligible presence in the atmosphere. BPA also dissipates quickly in soil, with a half-life of less than 5 days [34].

4.3.3.3 Legislative framework

The European Food Safety Authority (EFSA) completed a re-evaluation of the risks of BPA in 2023, concluding that its tolerable daily intake should be greatly reduced. This led the European Union to ban BPA in all the food contact materials, including plastic and coated packaging, in December 2024. The ban will come into force after an implementation period of up to three years.
Although Bisphenol A is not completely banned, there are several regulations that either restrict or ban its use in several categories of products:

- On 19 December 2016, the Member State Committee (MSC) unanimously agreed on the identification of bisphenol A as Substance of very high concern (SVHC) under REACH Regulation.
- ECHA has added Bisphenol A to Annex XVII to REACH, restricting by 2 January 2020 the concentration of Bisphenol A in thermal paper to 0.02% by weight.
- Regulation (EU) 2024/3190 prohibits the use of BPA and other bisphenols and bisphenol derivatives in certain materials and articles intended to come into contact with food.
- Directive 2014/81/EU o amended the Toy Safety Directive 2009/48/EC, setting migration limits in toys for children up to three years and for toys meant for placement in a child's mouth.
- The Cosmetic Products Regulation 1223/2009 banned the use of bisphenol A in cosmetic products.

There are currently no regulations setting limits for BPA in biosolids, neither at the EU nor at Member State levels.

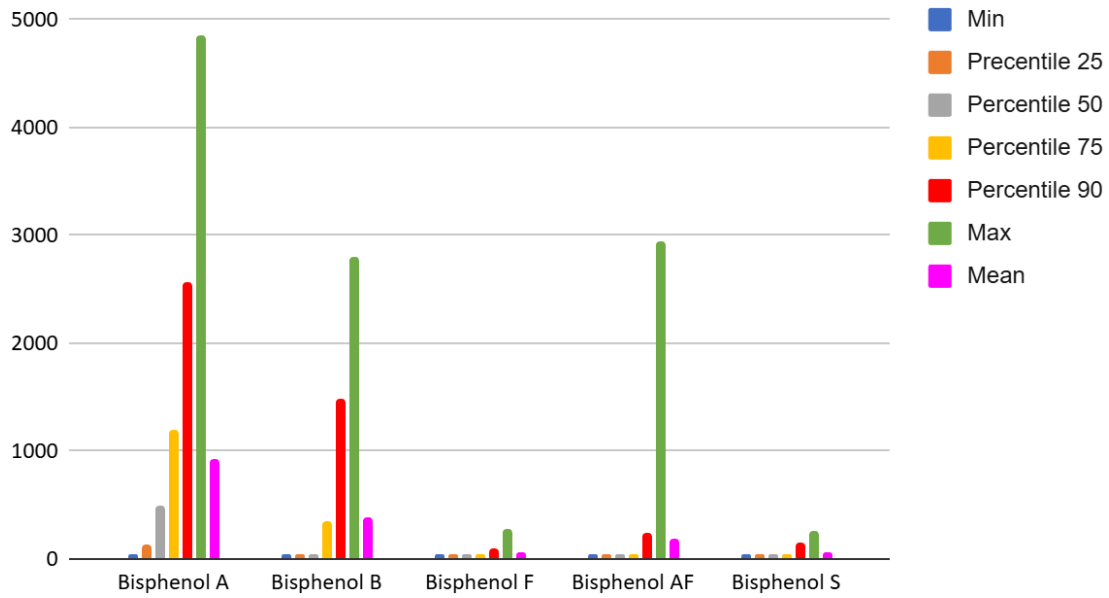
4.3.2.4 Biosolids Observatory results for bisphenols

The 5 different bisphenols that have been analysed in 25 biosolids samples are detailed in Table 11. The results for all of them had at least one result above the quantification limit.

Table 11 : Quantification limits and detection frequency for bisphenols

Substance	Quantification Limit (µg/kg DS)	Detection Frequency
Bisphenol A	50	100%
Bisphenol B	50	100%
Bisphenol F	50	100%
Bisphenol AF	50	100%
Bisphenol S	50	100%

Figure 3 : Bisphenols concentrations in µg/kg DS



All bisphenol compounds were systematically detected. Bisphenol A and B were by far the most prevalent, with average values of 920 and 380 µg/kg DS, respectively. Some relatively high values for bisphenol AF were observed, raising the average to 180 µg/kg DS.

4.3.3 Chlorobenzenes

4.3.3.1 General data on chlorobenzenes

Chlorobenzenes (CB) are a group of aryl chlorides/halobenzenes consisting of one or more chlorine atoms as substituents on a benzene core. Water solubility of chlorobenzene compounds is low, decreasing with increased chlorination.

Penta-CB was used as an intermediate in the production of pesticides, particularly the fungicide pentachloronitrobenzene. In addition, it was also used in a mixture of chlorobenzenes added to products containing polychlorinated biphenyls in order to reduce their viscosity or as a fire retardant.

Hexachlorobenzene (HCB) was commonly used as a seed treatment for prevention of fungal growth on crops such as wheat, barley, oats, and rye. Hexachlorobenzene is currently found as an unintentional by-product in several high-volume chlorinated solvents (carbon tetrachloride, trichloroethylene, and perchloroethylene) and in various pesticides, including pentachloronitrobenzene,

chlorothalonil, dimethyl 2,3,5,6-tetrachlorotereohthalate (DCPA), picloram, and pentachlorophenol.

4.3.3.2 Chlorobenzenes in the environment

Natural sources of chlorobenzenes in the environment have not been identified. The release of chlorobenzenes into the environment occurs primarily during manufacturing processes but also through the variety of their uses.Chlorobenzenes are removed from the environment principally by biological, and, to a lesser extent by non-biological, mechanisms; however, they are considered moderately persistent in water, air, and sediments.

Chlorobenzenes were released to the environment from sites where they were either manufactured or used as intermediates for the production of other chemicals. Other uses of chlorobenzenes, including as deodorizers, fumigants, degreasers, insecticides or herbicides have also resulted in indirect releases into the environment.

Part of chlorobenzenes released into the aquatic environment have evaporated to the atmosphere, the preferential distribution from water to air decreasing with increasing chlorination.

Many microorganisms from sediments and sewage sludge can degrade chlorobenzenes. Higher chlorinated compounds are less readily degraded, and such degradation occurs only under aerobic conditions. Under anaerobic conditions in soil and groundwater, Dichlorobenzene (DCB), Trichlorobenzenes (TCBs), and Pentachlorobenzenes (PeCBs) are usually resistant to microbial degradation. Retention of chlorobenzenes in soil increases with the organic content of the soil; there is a positive correlation between the degree of chlorination of the compound and its adsorption on organic matter. Soil half life of CB ranged from 13 to 219 days [35] except for HCB for which half life reaches 9 years [36].

4.3.3.3 Legislative framework

PeCB and HCB are persistent organic pollutants banned under the 2001 Stockholm Convention on Persistent Organic Pollutants. In the European Union, the ban is implemented through Regulation (EU) 2019/1021, on persistent organic pollutants (the POP Regulation). Substances included in Annex I of the Regulation can not be placed in the market or used, except under specified conditions. The Regulation (EU) 2022/22911 includes an unintentional trace contaminant (UTC) limit value of less than 10 mg/kg for substances, mixtures and articles to hexachlorobenzene (HCB) under POPs Regulation Annex I, which is applicable from 13 December 2022.

There is no limit set for chlorobenzenes in biosolids at European or Member State level. However, the German Sewage Sludge Ordinance (AbfKlärV) sets an AOX (adsorbable organic halogens, which chlorobenzenes are part of) limit to 500 mg/kg of dry matter.

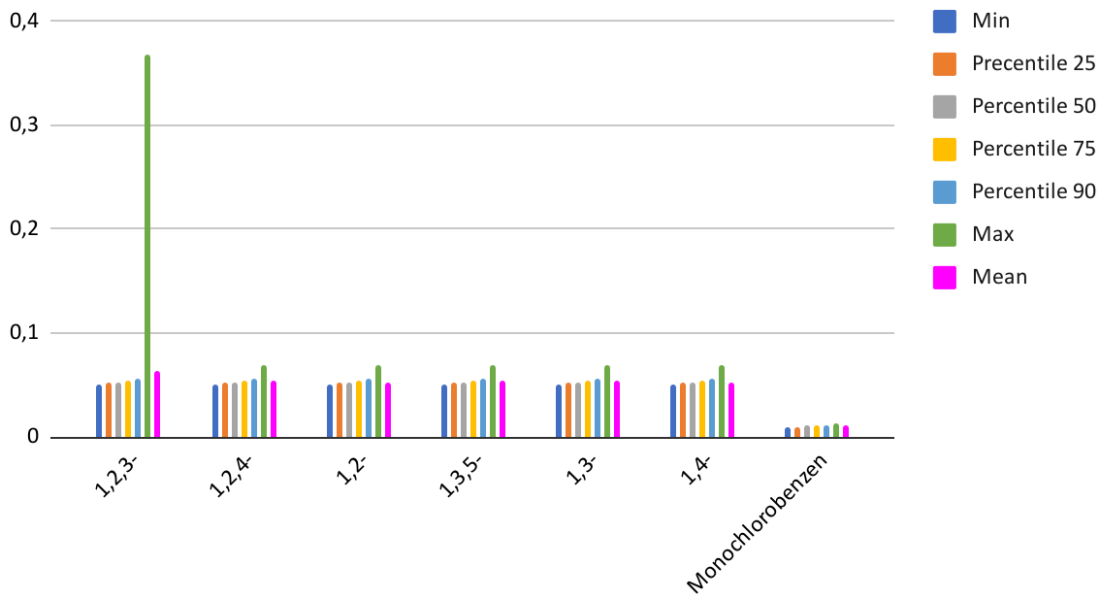
4.3.3.4 Biosolids Observatory results for chlorobenzenes

The 7 different chlorobenzenes that have been analysed in 29 biosolids samples are included in Table 12. Only hexachlorobenzene and pentachlorobenzene are in concentrations under the quantification limit. The other chlorobenzenes are systematically detected.

Table 12 : Quantification limits and detection frequency for chlorobenzenes

Substance	Quantification Limit (mg/kg DS)	Detection Frequency
Hexachlorobenzene	0,1	0 %
Pentachlorobenzene	0.05	0 %
1,2,3-trichlorobenzene	0,05	100%
1,2,4-trichlorobenzene	0,05	100%
1,2-dichlorobenzene	0,05	100%
1,3,5-trichlorobenzene	0,05	100%
1,3-dichlorobenzene	0,05	100%
1,4-dichlorobenzene	0,05	100%
Monochlorobenzene	0,01	100%

Figure 4 : Chlorobenzenes concentrations in mg/kg DS



All chlorobenzenes compounds are systematically detected in the biosolids samples. The concentrations of monochlorobenzenes are all equivalent to, or very close to, the limit of quantification set at 0.01 mg/kg DS. For the other chlorobenzenes, the average concentrations are all very close to 0.05 mg/kg DS except for one sample which has a concentration of 0.368 mg/kg DS for 1,2,3-trichlorobenzene.

4.3.3.5 Biosolids Observatory results for adsorbable organic halogens

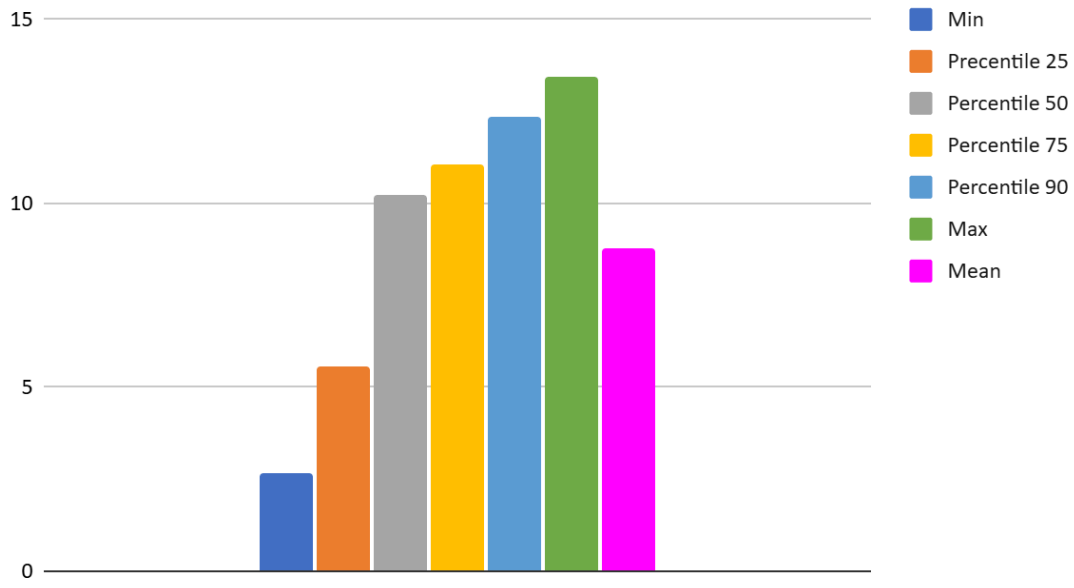
As mentioned in § 4.3.3.3 Chlorobenzenes are part of the AOX family for which in Germany a limit of 500 mg /kg DS is set in the German regulation for Biosolids that are spread on land. AOX is not a single chemical, but the sum of organic compounds that contain a halogen bond (chlorine, bromine, or iodine). AOX compounds are used in different industrial processes or for disinfection such as:

- Pulp and paper industry: historically, this has been the biggest source due to chlorine bleaching of wood pulp.
- Water disinfection: when chlorine is added to drinking water or swimming pools, it reacts with natural organic matter to form AOX (like chloroform).
- Chemicals' manufacturing: production of solvents dichloromethane (DCM), trichloroethylene (TCE), pesticides, and pharmaceuticals.

- Textile & leather: used in flame retardants, dyes, and tanning processes.

AOX index was calculated by summing up the chlorinated, iodinated, brominated and fluorinated compounds. The latter, which notably include PFAS, are also classified as adsorbable organic fluorine (AOF).

Figure 4 bis : AOX concentrations in mg/kg DS



With mean values of less than 10 mg/kg DS and max values of less than 15 mg/kg DS, AOX concentrations are all far away from the German limit of 400 mg/kg DS.

4.3.4 Chlorophenols

4.3.4.1 General data on chlorophenols

Chlorophenols are a group of chemicals that are produced by adding chlorines to phenol, an aromatic compound derived from benzene. Most chlorophenols are solid at room temperature.

Tri-, tetra- and penta- chlorophenols are used to surface-treat fresh-cut logs and lumber. Large quantities of lower chlorophenols serve as intermediates in the production of pesticides. Lesser amounts of chlorophenols are used as wood preservatives in agricultural and domestic applications. Chlorophenols, particularly tetra-, and to a lesser extent, trichlorophenols, have been used as bactericides, algicides, molluscicides, acaricides, fungicities, and mould inhibitors, and for less specific uses, such as general antiseptics and disinfectants.

4.3.4.2 Chlorophenols in the environment

Chlorophenol compounds and their derivatives are ubiquitous contaminants in the environment. Chlorophenols get into the environment from a variety of sources, such as industrial waste, pesticides, and insecticides, or by degradation of complex chlorinated hydrocarbons.

While chlorophenols are considered to be primarily water and soil contaminants, atmospheric deposition also occurs. Measurable quantities of chlorophenols have been detected in air, rainfall, and snow, sometimes far from emission points. Furthermore, significant quantities of chlorophenols can be released by incinerators' flue gas. Chlorophenols are also generated by human activity via several indirect routes such as paper or textile bleaching, water disinfection or waste incineration. No estimates are available of the quantities of chlorophenols released as a result of the disinfection of wastewater with chlorine, volatilization, or domestic uses of products containing these substances.

Chlorophenols are strongly absorbed on acidic soils, and those with a high organic content. Leaching is more significant in basic and mineral soils. Chlorophenol residues are removed from the environment by both biological and non-biological degradation. Although chlorophenols are quite toxic for microorganisms in general, they are readily metabolized by a large number of microorganisms that occur in soils, natural water, sediments, and sewage sludge.
Estimates of soil half-life for pentachlorophenol in an aerobic unacclimated environment were reported to range from 23 to 178 days. Increased content of organic matter in the soil has also been reported to increase the half-life of pentachlorophenol in soil by decreasing the bioavailability of the compound. Flooded soils and paddy soils have half-lives on the order of 10 to 120 days [37].

4.3.4.3 Legislative framework

The Stockholm Convention on Persistent Organic Pollutants listed pentachlorophenol and its salts/esters in its Annex A in 2015, designating them for elimination with limited exemptions for utility pole treatment.

In the European Union, production of pentachlorophenol and its sodium salt ceased in 1992, with subsequent bans on use implemented under national legislation and harmonized through the Biocides Directive (98/8/EC), prohibiting active substance approval for preservatives. Member States maintain zero tolerance limits for pentachlorophenol residues in food and drinking water, enforced via Regulation (EC) 396/2005. Chlorophenols such as

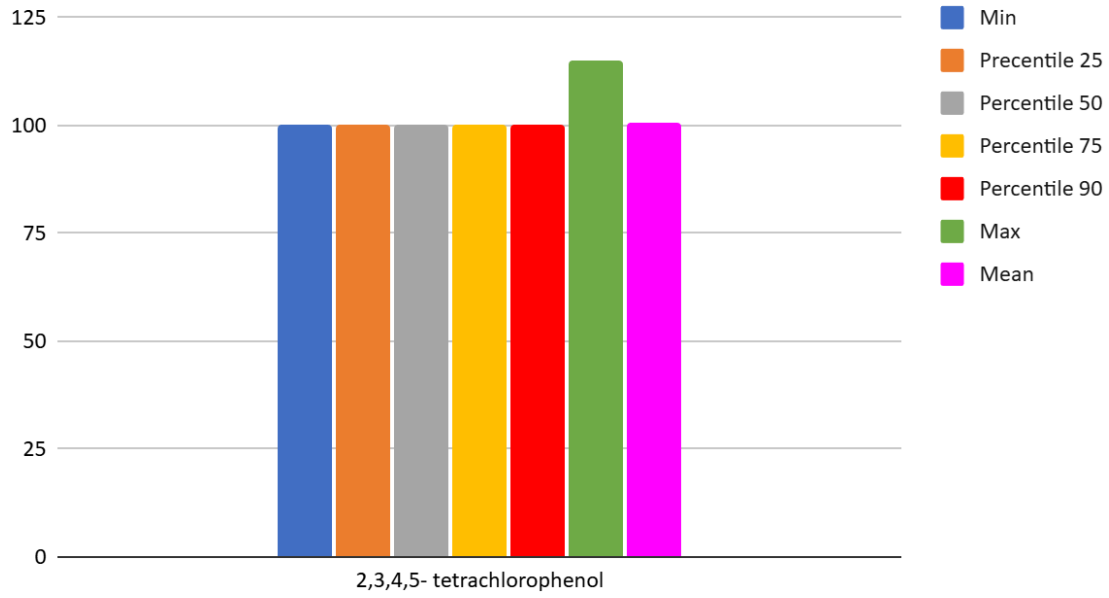
pentachlorophenol and its salts/esters are also regulated under the Persistent Organic Pollutants (POPs) Regulation (EC) 850/2004, integrated with REACH, classifying them as POPs requiring strict controls on production, use, and unintentional releases, with a maximum allowable concentration of 5 mg/kg in substances and preparations since 2021. Dichlorophenols, including 2,4-dichlorophenol, face limitations in biocidal applications under the Biocidal Products Regulation (EU) 528/2012, where active substances like chlorophene (a dichlorophenol derivative) were not approved for product-types such as disinfectants due to inadequate efficacy and risk data.

There is no limit set for chlorophenols in biosolids at European or Member State level.

4.3.4.4 Biosolids Observatory results for chlorophenols

Twenty different chlorophenols (list in Annex D) were analysed in 29 biosolids samples. Among these, only 2,3,4,5- tetrachlorophenol had at least one result above the quantification limit.

Figure 5 : Chlorophenols concentrations in µg/kg DS



2,3,4,5- tetrachlorophenol was the only chlorophenol compound detected in 2 biosolids samples (out of 29), with concentrations of 103 and 115 µg/kg DS which is very close to the quantification limit of 100 µg/kg DS.

4.3.5 Dioxins and furanes

4.3.5.1 General data on dioxins and furanes

Dioxins and dioxin-like compounds refer to a group of chlorinated organic chemicals characterized by shared structural features and toxicological properties, primarily polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and select polychlorinated biphenyls (PCBs). These compounds are persistent environmental pollutants known for their stability and bioaccumulative nature.

4.3.5.2 Dioxins and furanes in the environment

Dioxins are naturally emitted from volcanic eruptions, forest fires and natural combustion.

However, anthropogenic activities are the predominant sources of PCDDs, PCDFs, and dioxin-like polychlorinated biphenyls (dl-PCBs), formed primarily as unintentional by-products during incomplete combustion or high-temperature processes involving organic matter, chlorine, and oxygen. Waste incineration represents a major emission pathway, particularly from municipal solid waste (MSW), hazardous waste, and medical waste facilities.

Metallurgical processes, such as secondary copper and aluminum smelting, iron sintering, and electric arc furnaces, generate PCDD/F through chlorine-containing feedstocks and fly ash interactions. Other contributors include power generation from coal or biomass, crematories, and vehicle exhaust, though these are minor compared to incineration and metallurgy.

The main sources for emissions of dioxins to air in the EU are [38] [39]:

- Residential combustion: 35 to 45%,
- Iron and steel industry: 25 to 35%,
- Energy supply: 10 to 15%,
- Waste incineration: < 5%,
- Others (transport, outside burning, accidental fires): 10 to 15%.

Atmospheric transport is a major dispersal mechanism for dioxin-like compounds (DLCs) in the environment. The presence of DLCs in remote nonindustrial locations suggests that long-range atmospheric transport of the compounds occurs. DLCs are removed from the atmosphere by either dry or wet deposition.

Dioxins and dioxin-like compounds exhibit high environmental persistence due to their chemical stability, as well as resistance to natural degradation processes, such as biodegradation, hydrolysis, and photolysis, under typical

environmental conditions. In soil, the half-life of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD), the most studied congeners, ranges from 5 to 10 years on average, with surface soils showing 9 to 15 years and subsurface soils extending from 25 to 100 years, depending on factors like organic content and microbial activity.

The dioxin levels in rural and agricultural surface soil are typically 0.1-5 ng I-TEQ/kg dry weight, and about ten times higher levels are found in urban soil. The mean dioxin levels in United Kingdom's and Norwegian grass- and woodland soil (0-5 cm depth) were 4-30 ng I-TEQ/kg dry weight. In Danish topsoil (0-10 cm depth) the levels in rural areas were all <1 ng I-TEQ/kg dry weight. The highest toxic equivalent (TEQ) concentrations were found in soils from a park in Copenhagen (15 ng I-TEQ/kg) and a residential area downtown (20 ng I-TEQ/kg) [40].

4.3.5.3 Legislative framework

The Stockholm Convention on Persistent Organic Pollutants designates polychlorinated dibenzo-p-dioxins (PCDDs) and dibenzofurans (PCDFs) as unintentional persistent organic pollutants (POPs), requiring the 186 parties to minimize releases through application of best available techniques (BAT) and best environmental practices (BEP), aiming at continual reduction and ultimate elimination where feasible.

In 1989, for the first time, the EU adopted legislation to reduce dioxin emissions from municipal waste incineration by setting up so-called operational conditions, leading to a significant reduction of dioxin emissions. The Directive 2000/76/EC on the incineration of waste sets an air emission limit value of 0.1 ng I-TEQ/Nm^3 for all waste incinerators and for stationary sources with co-incineration of waste, including cement kilns. This Directive also sets a discharge limit value of 0.5 ng I-TEQ/l or 150 ng I-TEQ/tonne of waste.

Directive 96/61/EC concerning integrated pollution prevention and control covers many relevant industrial sectors that generate dioxins, and Best Available Technique Reference Documents (BREFs) address dioxins explicitly, giving clear indications on achievable emission values.

Directive 2000/76/EC on incineration sets emission limits for dioxins at 0.1 ng TEQ/Nm^3 for large combustion plants, to curb releases from waste treatment and energy production.

Dioxins emissions in the environment are also regulated through the waste and waste incineration Regulation 850/2004 on persistent organic pollutants and amending Directive 79/117/EC.

Germany, Luxembourg, Croatia, Portugal and Austria have set a limit of 100 ng/kg DS TEQ in biosolids that are applied on land. Italy has set the most stringent limit in the EU to date: 25 ng/kg DS.

According to standards from some countries (e.g., Germany, Japan, United States, The Netherlands) the acceptable dioxin concentrations in soil for residential areas is of 1000 pg/g TEQ (Toxic Equivalents); and between 3.9 pg/g and 4.3 pg/g TCDD for differing zones within the United States. Other countries have set lower guideline levels, e.g., Finland (500 pg/g TEQ), Canada (350 pg/g TEQ) and Sweden (10 pg/g TEQ). For agricultural soil, the standard/guideline values are less than or equal to10 pg/g, with the German standard being 5 pg/g; and the standard in England, Columbia, Canada, and The Netherlands being 10 pg/g TEQ [41].

The French Food Safety Agency considers that tolerable soil contamination levels to avoid the risk of non-conformities in meat and/or milk from animals having grazed in the contaminated area lies between 2 and 15 pg TEQ/g dry weight, depending on the different categories of investigated animals [42].

In 1992 German authorities set a preliminary reference (limit) value of 5 ng TEQ/kg dry weight for soil, where no sanitary measures need to be carried out. If levels are 5-40 ng TEQ/kg dry weight, monitoring should be carried out and soil with more than 40 ng TEQ/kg dry weight should not be used for agriculture. It was recommended that soil with ≥100 TEQ ng/kg dry weights should be removed from children playgrounds, soil with ≥1000 ng/kg dry weight should be removed from residential areas and soil with ≥10 000 ng TEQ/kg dry weight from all areas.

4.3.5.4 Biosolids Observatory results for dioxins and furanes

The 17 different dioxins and furanes compounds that have been analysed in 20 Biosolids samples are in Table 13. The concentration in biosolids for all of them was above the quantification limits.

Table 13 : Quantification limits and detection frequency for dioxins and furanes

Substance	Quantification Limit (ng/kg DS)	Detection Frequency
1,2,3,7,8-Pentachlorodibenzofuran	0,1	100%
2,3,4,7,8-Pentachlorodibenzofuran	0,1	100%
1,2,3,4,7,8-Hexachlorodibenzofuran	0,1	100%
1,2,3,6,7,8-Hexachlorodibenzofuran	0,1	100%
2,3,4,6,7,8-Hexachlorodibenzofuran	0,1	100%
1,2,3,7,8,9-Hexachlorodibenzofuran	0,1	25%
1,2,3,4,6,7,8-Heptachlorodibenzofuran	0,1	100%

1,2,3,4,7,8,9-Heptachlorodibenzofuran	0,3	100%
Octachlorodibenzofuran	4	90%
2,3,7,8-Tetrachlorodibenzodioxin	0,1	70%
1,2,3,7,8-Pentachlorodibenzodioxin	0,1	100%
1,2,3,4,7,8-Hexachlorodibenzodioxin	0,1	100%
1,2,3,6,7,8-Hexachlorodibenzodioxin	0,1	100%
1,2,3,7,8,9-Hexachlorodibenzodioxin	0,1	100%
1,2,3,4,6,7,8-Heptachlorodibenzodioxin	0,4	100%
Octachlorodibenzodioxin	4	100%
2,3,7,8-Tetrachlorodibenzofuran	0,2	100%

Figure 6 : Dioxins and Furanes concentration in ng/kg DS chart 1/2

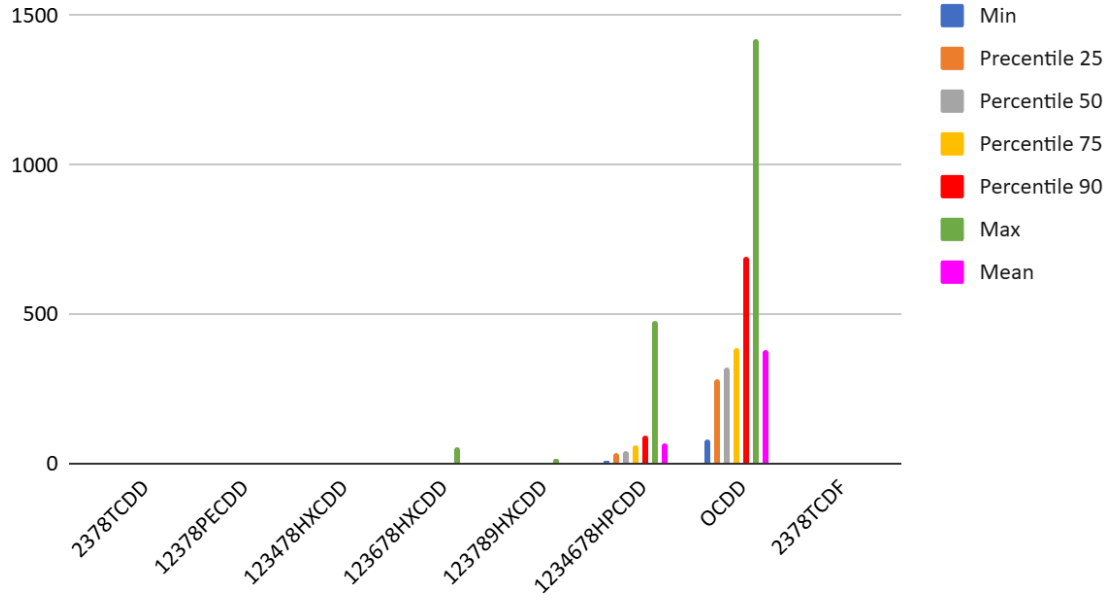
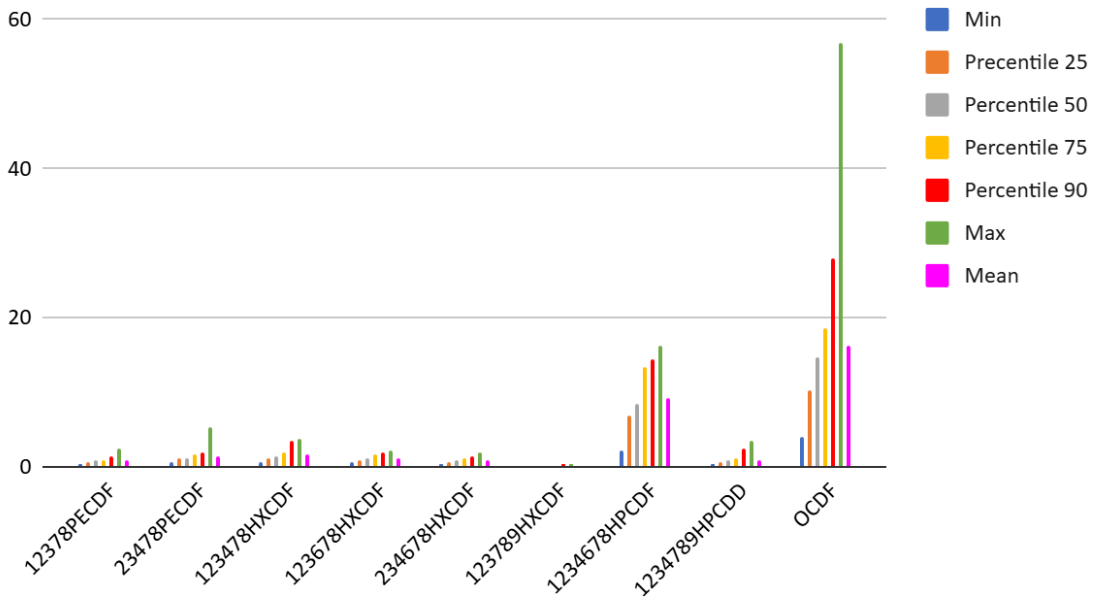


Figure 7 : Dioxins and Furanes concentration in ng/kg DS chart 2/2



All dioxins and furans have concentrations exceeding the quantification limits. Octachlorodibenzodioxin is by far the dioxin compound with a higher presence, with an average value of 378 ng/kg followed by 1,2,3,4,6,7,8-heptachlorodibenzodioxin 66.3 ng/kg and octachlorodibenzofuran 16.3 ng/kg. All the other dioxin compounds' concentrations do not exceed 10 ng/kg DS.

Toxicity Equivalent Factor (TEF) is 0.0003 ng/kg DS for OCDD and OCDF and 0.01 ng/kg DS for 1,2,3,4,6,7,8-Heptachlorodibenzodioxin. The average total TEF is 3.3 ng/kg DS and the maximum value is 14 ng/kg DS , which is lower than the strictest limit set by one of the EU Member States, in this case Italy with 25 ng/kg DS.

4.3.6 Nonylphenol and nonylphenol ethoxylates

4.3.6.1 General data on nonylphenol and nonylphenol ethoxylates

Nonylphenol (NP) is a synthetic alkylphenol compound, characterized by a phenol ring substituted with a nine-carbon alkyl chain. It is used primarily as an intermediate in the production of nonylphenol ethoxylates (NPEs) which are nonionic surfactants widely employed in detergents, industrial cleaners, emulsifiers, and agrochemical formulations due to their wetting, emulsifying, and detergent properties.

NPEs exhibit strong emulsifying, wetting, and dispersing capabilities, essential for industrial cleaning formulations. These properties enable NPEs to effectively solubilize oils, greases, and particulate soils in detergents, making them suitable for heavy-duty applications, such as metalworking fluids, industrial laundry, and surface preparation processes. In textile processing, NPEs serve as scouring agents to remove impurities from fabrics prior to dyeing, improving processing efficiency and product quality. Their chemical stability across a wide pH range and temperature tolerance further supports their utility in demanding industrial environments, such as pulp and paper production and leather tanning.

Nonylphenol serves as an intermediate in the production of phenolic resins, epoxy resins, and phenolic oximes, which are utilized in coatings, adhesives, and composites. It functions as an antioxidant in rubber and plastics, enhancing material stability against oxidation and degradation. Additionally, nonylphenol is incorporated as a stabilizer and plasticizer in polyvinyl chloride (PVC) formulations to improve flexibility and longevity. In the petroleum sector, it acts as a lubricating oil additive and demulsifier to prevent emulsion formation in crude oil processing. Other niche uses include fungicides and emulsifiers in industrial formulations.

4.3.6.2 Nonylphenol and nonylphenol ethoxylates in the environment

Industrial point sources, such as textile factories and chemical production sites, discharge nonylphenol-laden effluents directly or following post-treatment. Domestic diffuse releases occur through household laundry detergents and personal care products, reaching municipal sewage systems. Nonylphenol enters the environment mainly as a degradation product of NPEs following urban wastewater treatment exhibiting moderate solubility in water and high lipophilicity that promotes bioaccumulation in soils and sediments.

Biosolids represent a secondary terrestrial pathway, followed by agricultural inputs, including pesticide formulation. Atmospheric pathways are minor, involving volatilization from contaminated waters and aerosols enabling long-range transport and wet deposition. Landfill leachates and direct product disposal provide additional, localized sources, though less quantified compared to aqueous discharges

Nonylphenol demonstrates variable persistence in environmental compartments, influenced by redox conditions and isomer composition. In aerobic sediments, degradation half-life ranges from 0.9 to 13.2 days, while anaerobic conditions extend persistence with half-live from 23.9 to 69.3 days. NPEs degrade more rapidly in soils than nonylphenol itself, with an initial half-life of 0.3 to 5 days often yielding NP as metabolite [43].

4.3.6.3 Legislative framework

In the late 1990s, regulatory scrutiny of nonylphenol and NPEs intensified due to emerging evidence of their persistence, bioaccumulation, and endocrine-disrupting effects in aquatic environments.

The Directive 2003/53/EC, which amended Directive 76/769/EEC to restrict the marketing and use of nonylphenol and NPEs at concentrations of 0.1% or greater by weight in products supplied after specified transition periods, aiming to limit environmental releases from industrial and household applications. This directive set the first comprehensive EU-wide prohibition on these substances in non-pesticidal contexts. By 2004, the Directive's implementation extended restrictions to textile processing within the EU, banning NP/NPE use to prevent aquatic contamination from wastewater.

Nonylphenol and NPE are also restricted under entry 46a of REACH Annex XVII, prohibiting their intentional addition to mixtures or articles intended for supply to the general public or for uses likely to result in direct or indirect release into the aquatic environment, with phased implementation resulting in full restrictions for textile processing by February 3, 2021. Related branched and linear nonylphenols variants remain on the REACH SVHC candidate list, with ECHA updating entries for substances like tris(4-nonylphenyl, phosphorous acid) in January 2025. No further expansions to Annex XVII for NP/NPE occurred in 2025, though enforcement under REACH intensified with updated penalties announced in May 2025.

As no limits have been set for the concentration of NP/NPE in imported textiles, these substances may occur in clothing and textile products imported from countries outside the EU where there are no limits on the use of NPE and NP in the manufacturing process.

The Danish and Swedish environmental agencies have set a limit of 10 and 50 mg/kg DS of NP and NPEs in biosolids, while Portugal has set a maximum of 450 mg/kg DS and theRegion Lombardy (IT) has set a limit of 50 mg/kg DS.

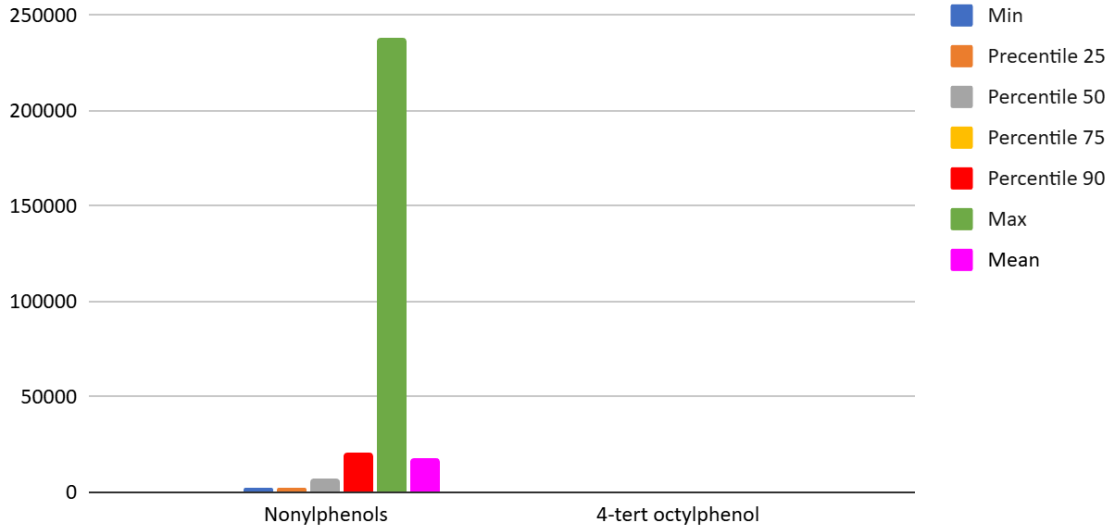
4.3.6.4 Biosolids Observatory results for nonylphenol and nonylphenol ethoxylates

Eight different nonylphenol and nonylphenol ethoxylates have been analysed in 29 biosolids (see Annex D), amongst which only nonylphenol and 4-tert octylphenol had at least one result above the quantification limit.

Table 14 : Quantification limits and detection frequency for nonylphenol and 4-tert octylphenol

Substance	Quantification Limit (µg/kg DS)	Detection Frequency
nonylphenol	100	100 %
4-tert octylphenol	100	35 %

Figure 8 : Nonylphenol and Nonylphenol Ethoxylates concentrations in µg/kg DS



Nonylphenol concentrations outweigh by far those of 4-tert-octylphenol, with average values of 18,300 µg/kg DS versus 187 µg/kg DS. Furthermore, there is significant heterogeneity in the nonylphenol content of the analyzed samples, with the maximum value representing more than 10 times the average values. Three biosolids samples exceeded the strictest limit of 10 mg/kg DS set by the Danish EPA.

4.3.7 Organotins

4.3.7.1 General data on organotins

Organotins are organometallic compounds containing tin–carbon bonds.

Di-organotin compounds, particularly dioctyltin and dimethyltin derivatives such as dioctyltin maleate and dimethyltin thioglycolate, function as heat and light stabilizers in polyvinyl chloride (PVC) processing. Organotin-stabilized PVC

demonstrates extended dynamic thermal stability. Beyond stabilization, organotin compounds catalyze key polymerization steps in polyurethane and silicone manufacturing. In silicone production, organotins facilitate condensation reactions between silanols and alkoxysilanes, promoting efficient network formation for sealants and elastomers. Organotin compounds, especially trisubstituted derivatives like tributyltin (TBT), were introduced as biocides in marine antifouling paints in the late 1950s. These paints released TBT ions to deter settlement and growth of fouling organisms such as barnacles, algae, and tubeworms on ship hulls, outperforming prior copper-based alternatives by maintaining cleaner surfaces over extended periods.
In agriculture, triphenyltin (TPT) compounds, including triphenyltin hydroxide (TPTH) and triphenyltin acetate, functioned as contact fungicides from the 1960s through the 1990s, targeting foliar diseases in crops like sugar beets, potatoes, rice, and pecans.

4.3.7.2 Organotins in the environment

Environmental transport of organotins involves leaching from antifouling paints and industrial sources into surface waters. To date, there is a lack of knowledge of other organotin sources and on the contamination of other environmental compartments. In soil, the half-life for triphenyltin has been determined to be approximately 140 days, while the mean half-life of TPTH Triphenyltin hydroxide is about 26 days [44] .

4.3.7.3 Legislative framework

Annex XVII of the REACH Regulation (EC) No 1907/2006 imposes restrictions on organotin compounds, limiting tri-substituted organotins to no more than 0.1% by weight (as tin) in articles such as textiles, gloves, and footwear since 1 July 2010, with earlier preparatory impact assessments completed in 2007 evaluating risks from consumer and industrial uses. These rules were extended to forbid certain dioctyltin and monooctyltin stabilizers in food-contact materials and toys, reflecting phased implementation to curb non-essential applications while allowing controlled industrial exemptions. There is no limit set for organotins in biosolids neither at European nor at Member State level.

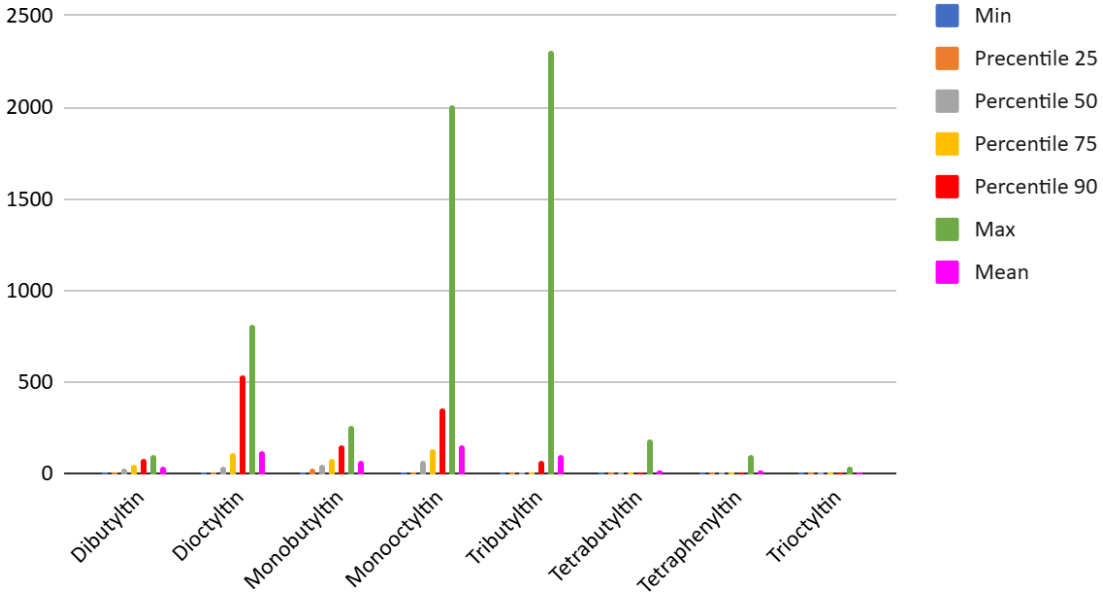
4.3.7.4 Biosolids Observatory results for organotins

Twelve different organotins have been analysed in 29 biosolids samples (see Annex D). Dibutyltin, dioctyltin, monobutyltin, monooctyltin, tributyltin, tetrabutyltin, tetraphenyltin and trioctyltin, had at least one result above the quantification limit.

Table 15 : Quantification limits and detection frequency for organotins

Substance	Quantification Limit (µg/kg DS)	Detection Frequency
Dibutyltin	10	62 %
Dioctyltin	10	65 %
Monobutyltin	10	89 %
Monooctyltin	10	65 %
Tributyltin	10	17 %
Tetrabutyltin	10	7 %
Tetraphenyltin	10	3 %
Trioctyltin	10	3 %

Figure 9 : Organotins concentrations in µg/kg DS



Monooctyltin, dioctyltin, and tributyltin are the organotins with the highest average concentrations, with 158, 123, and 101 µg/kg DS, respectively. For these three compounds, the maximum values represent between 10 and 20 times the average values.

4.3.8 Polycyclic aromatic hydrocarbons (PAHs)

4.3.8.1 General data on PAHs

Polycyclic aromatic hydrocarbons (PAHs) are organic compounds composed of two or more fused aromatic rings of carbon and hydrogen atoms.

PAHs are formed naturally through high-temperature pyrolysis and incomplete combustion of organic matter, primarily in wildfires where biomass, such as wood, leaves, and grasses, undergoes thermal decomposition under oxygen-limited conditions. Volcanic activity also contributes to a lesser extent to PAHs generation via magmatic processes and eruptive emissions.

4.3.8.2 PAHs in the environment

Anthropogenic sources dominate the release of PAHs into the environment, primarily through incomplete combustion of fossil fuels, biomass, and organic materials, accounting for the majority of global emissions. Stationary combustion sources, including residential heating, power plants, and industrial boilers, contribute substantially, particularly in regions reliant on coal or wood for energy. Industrial stationary sources, such as coke ovens in steel production and carbon black manufacturing, emit PAHs through high-temperature pyrolysis, with historical data indicating coke production alone as a leading point source in heavy industry. Other industrial activities, including aluminum smelting, asphalt production, and coal tar distillation, generate PAHs as byproducts or impurities, often volatilizing during processing or application. Waste incineration and open biomass burning, such as agricultural residue disposal, add to diffuse emissions, with municipal solid waste combustion releasing a mix of low- and high-ring PAHs depending on incinerator efficiency;

Mobile sources, chiefly internal combustion engines in vehicles, account for significant urban PAH burdens, with diesel exhaust producing particle-bound PAHs that enhance atmospheric persistence. Lower molecular weight PAHs like phenanthrene and pyrene predominate in the gas phase, while higher weight compounds such as benzopyrene bind to fine particulate matter. Deposition mechanisms include dry processes, such as gravitational settling and wet deposition by rain or snow. Deposition fluxes vary seasonally, averaging 229 ng m^{-2} day^{-1} (Sum of 13 PAH) in coastal urban settings, with winter peaks up to 523.8 ng m^{-2} day^{-1}. Higher molecular weight (HMW) PAHs, being less volatile, predominate in soil profiles due to their strong sorption to organic matter and clay minerals, which limits leaching and promotes persistence.

PAHs present in biosolids come mainly from impermeable surfaces runoff(roofs, roads, etc.). PAHs persistence is mainly determined by compound molecular weight. Half-lives go from < 2 years for naphthalene to > 16 years for coronene. Biological degradation is the most important loss mechanism for PAHs in soil [45].

4.3.8.3 Legislative framework

EU regulation addresses the PAHs content in drinking water (Directive 2020/2184) and foodstuff (Regulation 835/2011), or in specific items such as plastic or rubber components of consumer products that come into contact with the human body (Regulation 1272/2013) or in clay targets (Regulation 2025/660).

Regulation 2021/1199 also limits PAHs in granules or mulches used as infill material in synthetic turf pitches, or in loose form on playgrounds or in sport applications.

Directive 2010/75/EU on industrial emissions and Directive 2015/2193 on the limitation of emissions from medium combustion plants do not set any limit for PAHs. The Euro 6 standard, which sets emission limits for gasoline and diesel vehicles does not include PAH.

The decrease in industrial pollutant emissions to air can be partly attributed to European regulations, such as the EU Emissions Trading System and the Industrial Emissions Directive, and the switch away from fossil fuels towards renewable energy sources. In addition, the decreasing trends could be partly explained by the relocation of various heavy-polluting and energy-intensive manufacturing industries — such as textile or metal production — outside Europe.

No limits are set for PAHs in biosolids at EU level, but some Member States impose maximum values that can not be exceeded for the sum of a list of PAHs (with a content that varies considerably):

- Austria, Denmark and Sweden have set limits varying from 3 to 6 mg/kg DS for benzo(b+j+k)fluoranthene, benzo(a)pyrene, benzo(ghi) perylene and indeno (1,2,3-c,d) pyrene.
- Germany and Luxembourg have set limit values varying from 10 to 20 mg/kg DS for 16 US EPA HAPs (naphthalene, acenaphthylene, acenaphthene, fluorene, fenanthren, anthracene, fluoranthene, pyrene, benz(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, dibenzo (a,h) anthracene, indeno (1,2,3-c,d) pyrene and benzo(ghi)perylene);
- France has set limits for specific HAPs: 5 mg/kg DS for Fluoranthene, 2.5 mg/kg DS for benzo(b)fluoranthene and 2 mg/kg DS for benzo(a)pyrene.
- Italy has set a limit of 6 mg/kg DS for the sum of 19 HAPs.

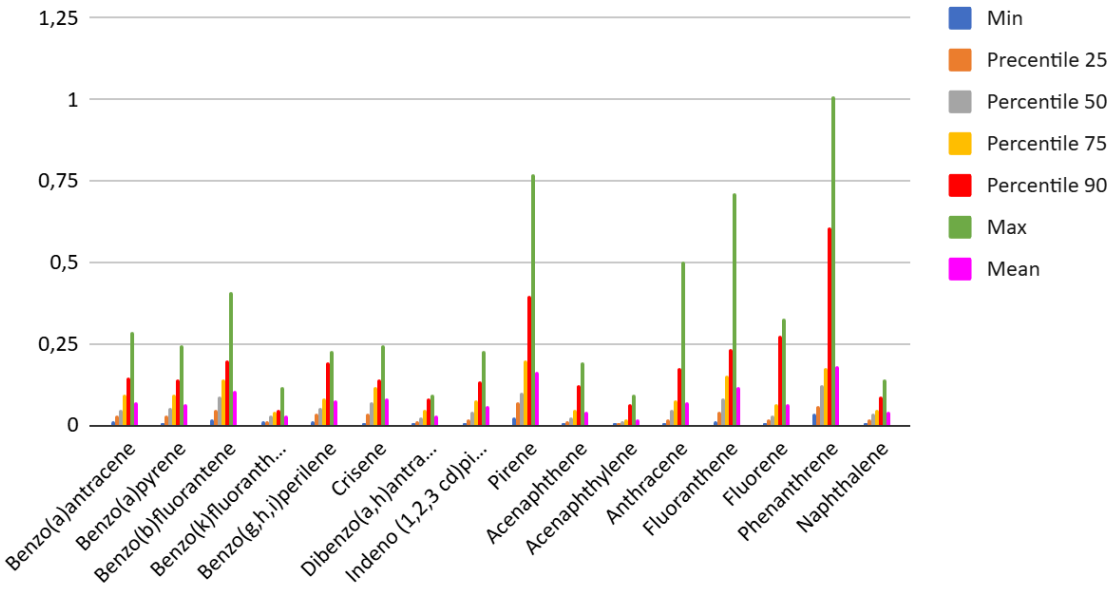
4.3.8.4 Biosolids Observatory results for PAH

Sixteen different PAHs have been analysed in 30 biosolids samples (shown in Table 16). All the results were above the quantification limit.

Table 16 : Quantification limits and detection frequency for PAHs

Substance	Quantification Limit (mg/kg DS)	Detection Frequency
Benzo(a)antracene	0,005	100 %
Benzo(a)pyrene	0,005	100 %
Benzo(b)fluorantene	0,005	97 %
Benzo(k)fluoranthene	0,005	83 %
Benzo(g,h,i)perilene	0,005	93 %
Crisene	0,005	100 %
Dibenzo(a,h)antracene	0,005	73 %
Indeno (1,2,3 cd)pyrene	0,005	83 %
Pyrene	0,005	100 %
Acenaphthene	0,005	77 %
Acenaphthylene	0,005	53 %
Anthracene	0,005	97 %
Fluoranthene	0,005	97 %
Fluorene	0,005	83 %
Phenanthrene	0,005	100 %
Naphthalene	0,005	83 %

Figure 10 : PAH Concentrations in mg/kg DS



The PAHs with the highest average concentrations are phenanthren (0.180 mg/kg DS), pyrene (0.165 mg/kg DS), fluoranthene (0.118 mg/kg DS), and

anthracene (0.071 mg/kg DS). For these compounds, the maximum observed values range from 0.5 to 1 mg/kg DS. Among the 30 samples tested, none was above the strictest limit set for 16 PAHs by the German EPA. Even if benzo(j)fluoranthene was not tested, the sum for benzo(b+k)fluoranthene, benzo(a)pyrene, benzo(ghi) perylene and indeno (1,2,3-c,d) pyrene was far away from the limit values set by Austria, Denmark and Sweden. Limit values set by the French EPA are also fully complied with.

4.3.9 Polybrominated diphenyl ethers (PBDEs)

4.3.9.1 General data on PBDEs

Polybrominated diphenyl ethers (PBDEs) are a class of synthetic organobromine chemicals comprising two phenyl rings connected by an oxygen atom, substituted with two to ten bromine atoms across 209 possible congeners, commercially formulated into mixtures such as pentaBDE, octaBDE, and decaBDE for use as additive flame retardants. These compounds are incorporated into polymers, polyurethane foams, textiles, and electronic casings to inhibit ignition and slow flame spread, thereby enhancing fire safety in consumer and industrial products. Unlike reactive flame retardants that chemically bond to host materials, PBDEs' additive application results in gradual leaching, contributing to their widespread environmental dispersal. PBDEs exhibit strong persistence in the environment, are resistant to degradation and are subject to bioaccumulation.

4.3.9.2 PBDEs in the environment

Transport mechanisms for PBDEs primarily involve atmospheric pathways, enabled by their semi-volatility and gas-particle partitioning, which facilitate long-range atmospheric transport of lower congeners. Soil deposition from atmospheric inputs further contributes to terrestrial persistence, with minimal leaching into groundwater owing to strong adsorption.

Contamination patterns are influenced by proximity to emission sources, including urban areas, electronic waste recycling sites, and landfills, where levels exceed background concentrations by orders of magnitude.

PBDEs are not chemically bound to the material in which they are incorporated. This means that at the production stage they were physically combined with the material being treated rather than chemically combined. Therefore, there is potential for them to leach out of products to household dust particles. High levels of PBDEs have been found in dust samples from homes, offices, classrooms and, in particular, car cabins. Dust particles settle on indoor floors or adhere to clothing and are released during cleaning operations, thus reaching

the urban wastewater system. PBDE 209 is the compound produced and marketed in the largest quantities.
PBDE 209 is mostly used in high-impact polystyrene plastic used to produce housings for televisions, computers, stereos and other small electronics as well as in upholstery textiles.

PBDEs congeners prevailing in soil are 47, 99, 100 and 183. Due to their lipophilic and hydrophobic properties PBDEs bind firmly to organic matter and remain in soils having a half-life as long as 28 years[46] .

4.3.9.3 Legislative framework

The Stockholm Convention on Persistent Organic Pollutants, designates certain PBDEs as POPs, requiring global elimination due to their persistence, bioaccumulation, and potential toxicity. In 2009, at the fourth Conference of the Parties in Geneva, commercial pentabromodiphenyl ether (containing tetra-, penta-, and hexaBDE congeners) and commercial octabromodiphenyl ether (containing hexa-, hepta-, and some nonaBDE congeners) were added to Annex A of the Stockholm Convention, mandating the elimination of their production and use, with limited exemptions for specific applications, like transport vehicles, until 2013.

Decabromodiphenyl ether (decaBDE), the dominant commercial formulation historically used in plastics and textiles, was listed in Annex A of the Stockholm Convention in 2017, at the eighth Conference of the Parties, with a phase-out deadline set for 2022–2023 depending on the application, though specific exemptions persist for items like aircraft parts and electrical components, to allow time for alternatives.

PentaBDE and octaBDE formulations were prohibited in 2004 under the Directive 2003/11/EC, which mandated Member States to ban their marketing and use in concentrations exceeding 0.1% by weight, driven by concerns over persistence and bioaccumulation identified in risk assessments. DecaBDE faced restrictions as a substance of very high concern under REACH since 2008, with further amendments to the POPs Regulation adopted on August 7, 2025, tightening unintentional trace contaminant limits for all PBDEs to 50 mg/kg in mixtures and articles to reduce emissions in the environment.

Delegated Regulation (EU) 2025/1482 amending Regulation (EU) 2022/2400, reduces the concentration limit in waste for the sum of tetraBDE, pentaBDE, hexaBDE, heptaBDE and decaBDE to 500 mg/kg as of 10 June 2023, to 350 mg/kg from 30 December 2025 and to 200 mg/kg from 30 December 2027.

There is no limit set for PBDE in biosolids at European or Member State level. Nevertheless, as PBDEs are listed under the POP Regulation ((EU) 2019/1021), they are subject to "Low POP Content" limits.
Therefore, the limits for the sum of regulated PBDEs (tetra-, penta-, hexa-, hepta-, and decaBDE) in biosolids are as follows:

- Current limit (2026): 350 mg/kg (dry matter).
- Future tightening: this limit is scheduled to drop to 200 mg/kg on December 30, 2027.

If biosolids contain (or will contain) PBDEs above these concentrations, they are (or will be) classified as "POP waste" and cannot (or will not) be applied to agricultural land.

4.3.9.4 Biosolids Observatory results for PBDEs

Eighteen different PBDEs were analysed in 29 biosolids samples (see Annex D). The ones in Table 17 had at least one result above the quantification limit.

Table 17 : Quantification limits and detection frequency for PBDEs

Substance	Quantification Limit (ng/kg DS)	Detection Frequency
Penta BDE 100	100	96 %
Hexa BDE 138	20	86 %
Hexa BDE 153	20	100 %
Hexa BDE 154	20	100 %
Tri BDE 17	20	93 %
Hepta BDE 181	20	69 %
Hepta BDE 183	20	100 %
Hepta BDE 190	20	79 %
Octa BDE 203	50	100 %
Deca BDE 209	20000	97 %
Tri BDE 28	20	100 %
Tetra BDE 47	1000	93 %
Tetra BDE 66	20	97 %
Tetra BDE 71	20	79 %
Penta BDE 85	20	93 %
Penta BDE 99	200	97 %

Figure 11 PBDEs concentrations in ng/kg DS (except PBDE 209)

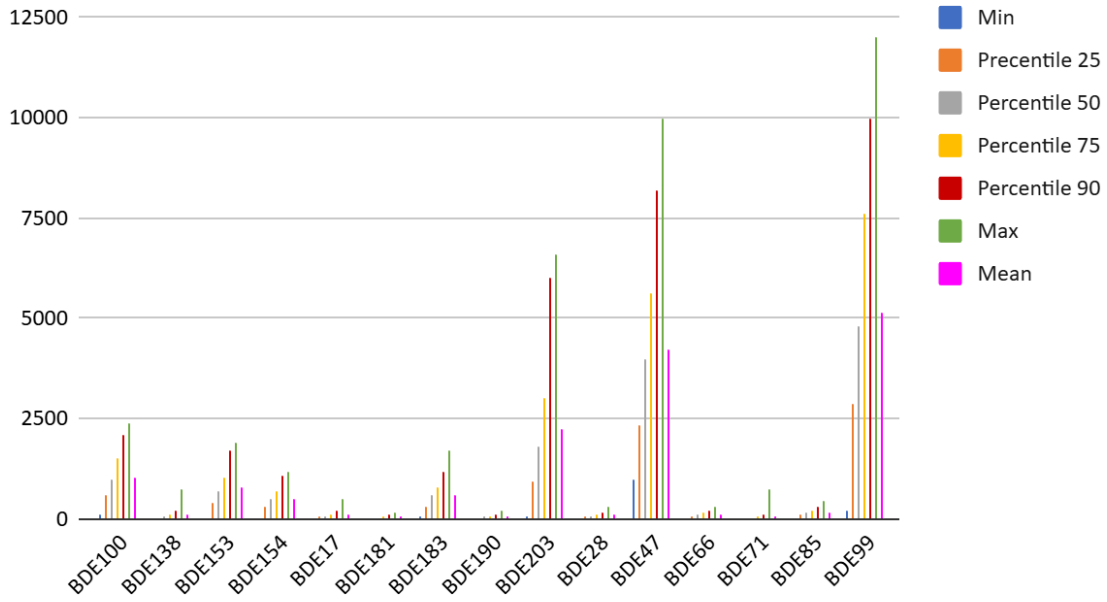
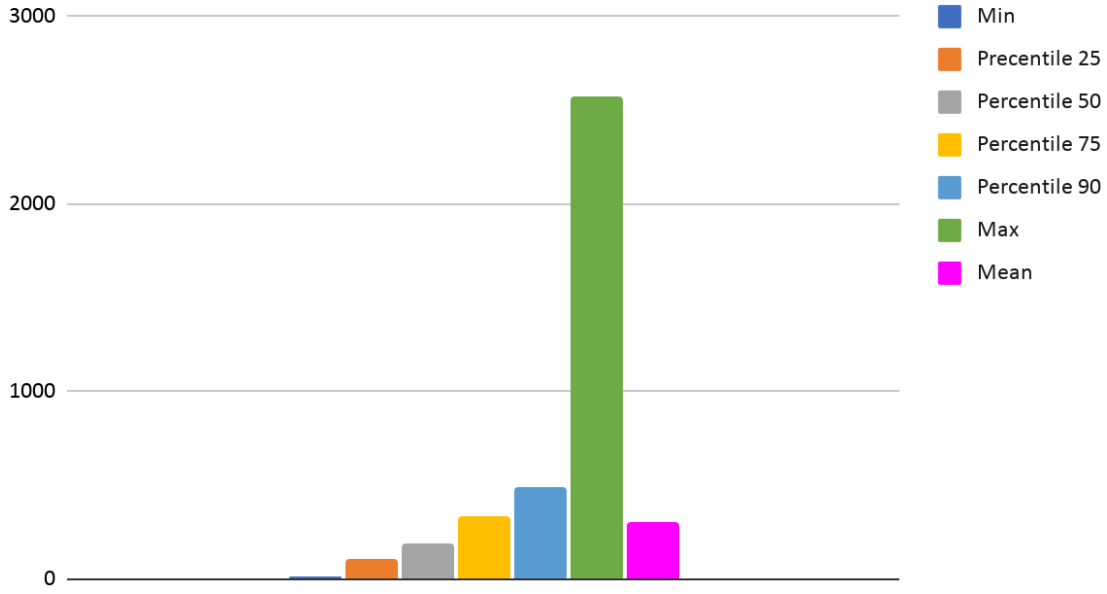


Figure 12 PBDE 209 concentrations in µg/kg DS



PBDEs with the highest average concentrations are PBDE 209 (305,841 ng/kg DS), PBDE 99 (5,120 ng/kg DS), PBDE 47 (4,217 ng/kg DS), and PBDE 203 (2,231 ng/kg DS). For the last three compounds, the maximum observed values range from 6,000 to 12,000 ng/kg DS , while they reach 2,570,000 ng/kg DS for PBDE 209. With a max 2.6 mg/kg DS of regulated PBDEs, biosolids tested are far away from the limit to be considered as "POP waste".

4.3.10 Polychlorinated biphenyls (PCBs)

4.3.10.1 General data on PCBs

Polychlorinated biphenyls (PCBs) are a group of 209 synthetic organochlorine compounds formed by adding chlorine atoms to biphenyl, resulting in highly stable, lipophilic molecules resistant to biological and chemical degradation. PCBs show very low water solubility, generally below 1 mg/L at 25°C, decreasing further with their chlorination level. PCBs were extensively used as dielectric insulating and coolant fluids in electrical transformers and capacitors due to their high dielectric strength, chemical stability, non-flammability, and ability to withstand high temperatures without degrading. Beyond core electrical insulation, PCBs were also used in cable wraps and insulation for power transmission lines, providing resistance to moisture and fire hazards. In other equipment, they functioned as hydraulic and heat transfer fluids in industrial machinery, such as presses, pumps, and heat exchangers, where their low viscosity and thermal stability offered advantages over flammable alternatives. PCBs also acted as lubricants and coolants in compressors, vacuum pumps, and gas turbines, enhancing performance in high-stress mechanical environments. Unintentionally produced PCBs may arise as by-products in various chemical manufacturing processes involving carbon, chlorine, and high temperatures. This includes the production of organic pigments used in inks, dyes, paints, and coatings. Additional ongoing sources include thermal industrial processes such as waste incineration, coal combustion, steel and pig iron production, and cement manufacturing

4.3.10.2 PCBs in the environment

PCBs exhibit semi-volatile properties that facilitate their transfer across environmental compartments, primarily through partitioning between gas and particle phases, volatilization, adsorption, and deposition processes. Lower chlorinated congeners, with higher vapor pressures, predominate in the gas phase and undergo long-range atmospheric transport, while higher chlorinated congeners preferentially sorb to aerosols or surfaces, limiting their mobility. In air, PCBs enter via volatilization from contaminated soils, waters, or surfaces resulting in their dispersion over thousands of kilometers.

In water, PCBs display low aqueous solubility (typically <0.01 mg/L for most congeners), resulting in strong partitioning to suspended particulates, sediments, and organic matter via hydrophobic interactions.

In soil, PCBs are strongly linked to organic matter and clays, evidenced by soil organic carbon-water partition coefficients (Koc) exceeding 5000 L/kg, which

restrict vertical and lateral migration. Surface soils act as sinks for atmospherically deposited PCBs, with limited downward percolation. PCBs pose a low groundwater threat due to their sorption dominance, though erosion can export particulates to adjacent waters [47].

Lower-chlorinated PCBs have a half-life in soil of 3–5 years while the higher-chlorinated ones (like PCB-180) often exceed 20–40 years [48].

4.3.10.3 Legislative framework

The Stockholm Convention on Persistent Organic Pollutants classified PCBs among the initial 12 persistent organic pollutants, obliging 185 ratifying parties to cease production and new uses immediately, conduct national inventories, and achieve environmentally sound disposal of in-use equipment (concentrations ≥0.005%) by 2025.

Different EU directives progressively restricted PCBs' marketing and use. In 1985 the Directive 85/467/EEC banned their application as raw materials or chemical intermediates, followed in 1996 by Directive 96/59/EC mandating inventory, collection, and disposal of PCB-containing equipment by December 31, 2010. Provisions under Regulation (EU) 2019/1021 aim to set limits for unintentional PCB contaminants in products.

No limits are set for PCBs in biosolids at EU level, but some Member States impose maximum values that must not be exceeded. Austria, France, Belgium, Sweden, Czech Republic, Luxembourg, Romania and Hungary have set limits varying from 0.2 to 1 mg/kg DS for the sum of 7 congeners: PCB 28, 52, 101, 118, 138, 153, 180; Croatia has set a limit of 0.2 mg/kg DS for PCBs 28, 52, 101, 141, 180; and Germany has set the same limit for any congener. Italy has set a limit of 0,8 mg/kg DS for the sum of all congeners.

4.3.10.4 Biosolids Observatory results for PCB

Thirty onePCBs were analysed in 25 biosolids samples (see Annex D). Among these, 18 PCBs, included in figures 13, 14 and 15, had at least one result above the quantification limit of 0.005 mg/kg DS.

Figure 13 : PCBs concentrations in mg/kg DS (1/2)

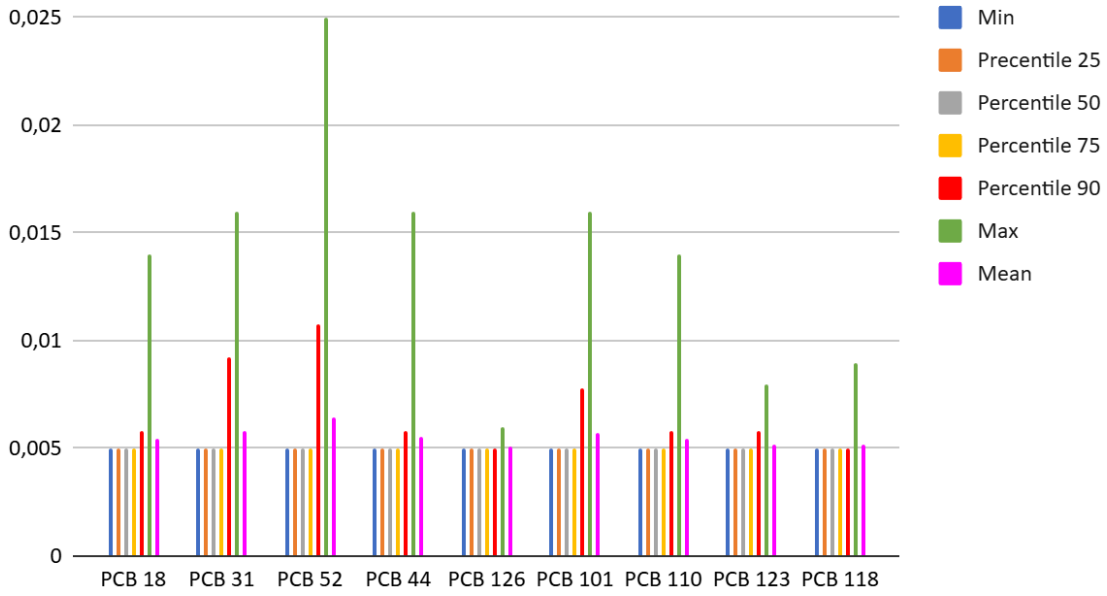


Figure 14 : PCBs concentrations in mg/kg DS (2/2)

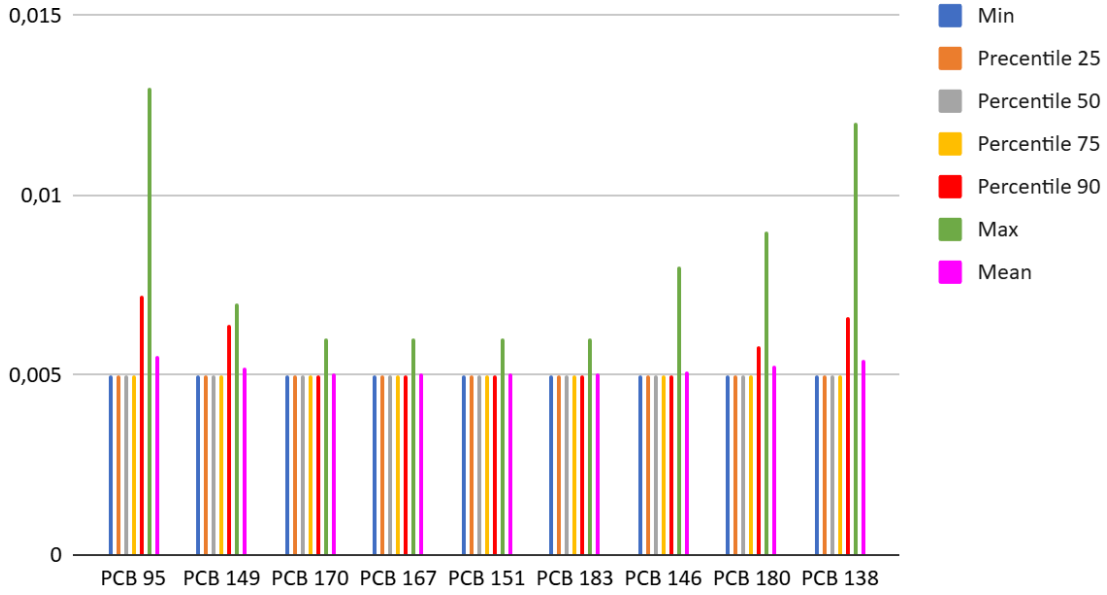
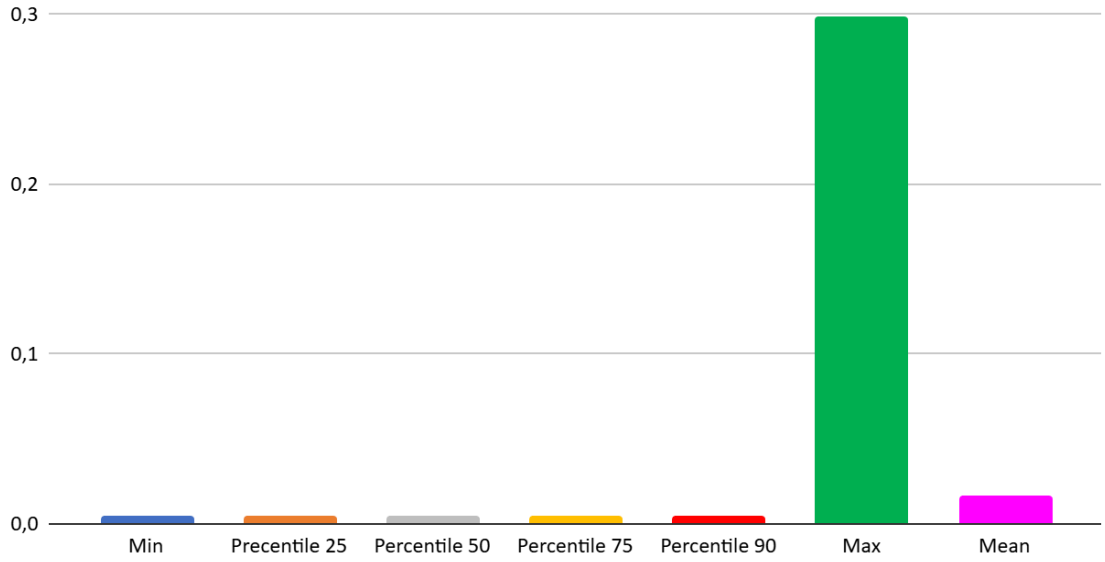


Figure 15 : PCB 169 concentrations in mg/kg DS



Most of the recorded values were close to the limit of quantification. Three samples alone account for the maximum recorded values. One sample shows relatively high concentrations of PCB 169 (0.3 mg/kg DS). Highest sum for PCB 28, 52, 101, 118, 138, 153, 180 is 0.068 mg/kg DS which is significantly lower than the most stringent regulatory limit of 0.2 mg/kg DS.

4.3.11 Polychlorinated naphthalenes (PCNs)

4.3.11.1 General data on PCNs

Polychlorinated naphthalenes (PCNs) are a group of theoretically 75 possible substances, containing one to eight chlorine atoms bound to the naphthalene di-benzene ring . PCNs have physical and chemical properties largely similar to those of PCBs. They are hydrophobic, have high chemical and thermal stability, good weather resistance, good electrical insulating properties,low flammability and a medium to low volatility, and they are compatible with other materials.. Higher chlorinated PCNs are potential candidates for long-range airborne transport due to their increasing affinity to escape from water.

In the past PCNs have been widely used primarily in:

- cable insulation
- wood preservatives
- engine oil additives
- feedstock for dye production and dye carriers
- flame proofing
- moisture proofing sealant

PCNs can also be found as contaminants in PCB mixtures, as they are formed during similar manufacturing practices.

4.3.11.2 PCNs in the environment

PCNs are formed in thermal heating and burning processes, of which waste incineration is the most important. Sixty eight of the 70 isomers of PCN have been measured in flue gas samples from municipal solid waste incinerators.

Reclamation and smelting of aluminium and copper may result in PCNs emissions. The major current sources of PCNs are likely to be emissions from municipal and special waste incinerators and disposal of items containing PCNs to landfill.

Half-life of PCNs in soils vary from 5 to 10 years [49].

4.3.11.3 Legislative framework

PCNs are listed in Annex A (elimination) of the Stockholm Convention on POPs, with specific exemptions for production of those chemicals as intermediates in production of polyfluorinated naphthalenes, including octafluoronaphthalene.

Regulation (EU) 2019/1021 on persistent organic pollutants added PCNs to its Annex III, thus obliging Member States to take measures to reduce their use and limit their dispersion in the environment.

There is no limit set for PCN's in biosolids at European or Member State level.

4.3.11.4 Biosolids Observatory results for PCNs

The three PCNs (1-chloronaftalene, 2-cloronaftalene, 1,2,3,4-tetracloronaftalene) analysed in 23 biosolids samples were all under the quantification limit of 0.2 mg/kg DS.

4.3.12 Per- and polyfluoroalkyl substances (PFAS)

4.3.12.1 General data on PFAS

Per- and polyfluoroalkyl substances (PFAS), the so-called forever chemicals, are a group of thousands of manufactured chemicals that have been used in industry and consumer products for their physical and chemical properties.

Perfluorooctanoic Acid (PFOA) and Perfluorooctane Sulfonate (PFOS) are two of the most widely used and studied chemicals in the PFAS group.

PFAS are widely used as oil or water repellents due to their surfactant properties but also in firefighting foam to extinguish petroleum-based fires. Major industry sectors are using PFAS such as automotive, aviation, textiles, leather and clothing, construction and household products, electronics, printing, adhesives and tapes, fire-fighting foams, food processing, and medical articles.

4.3.12.2 PFAS in the environment

Industrial production and use of PFAS have been the main sources of air, water and soil PFAS contamination. Firefighting foams have been identified as a significant source of PFAS but these compounds can also be found in consumer products, such as textiles, furniture, polishing and cleaning agents and creams, as well as in pharmaceuticals and pesticides.

PFAS migrate from air to soil through dry and wet deposition. Dry deposition involves settling of particle matters on which ionic PFAS are attached. The PFAS from dry deposition can be taken up by vegetation, surface water, and soil. Wet deposition of PFAS usually takes place with precipitation, such as rainfall and snowfall. PFAS occurrence in wet deposition occurs worldwide, and is a major pathway for PFAS removal from the atmosphere and its transport to the surface environment, such as topsoil.

Soils with no known PFAS point sources have average background levels of PFOS and PFOA of 2.5 µg/kg [49]. This means that the global quantities of PFOS and PFOA per hectare are of circa 10 grams. On the other hand, nearby PFAS manufacturing sites, aqueous film forming foam or fire testing areas, the maximum reported concentrations for PFOS range from 0.4 to 460,000 µg/kg, with a median value of 8,722 µg/kg. The maximum reported concentrations for PFOA range from 2 to >50,000 µg/kg, with a median value of 83 µg/kg. For soil indirectly exposed to PFAS, such as locations at which soil improvers are applied, and/or sites at which surface water, groundwater, or treated urban wastewater are used for irrigation, the maximum reported concentrations for PFOS range from 0.4 to 5,500 µg/kg, with a median value of 680 µg/kg, while the maximum reported concentrations for PFOA range from 0.8 to 2,531 µg/kg, with a median value of 38 µg/kg [51].

PFAS compounds are found in urban wastewater and some of these PFAS have been shown to accumulate in sewage sludge. High concentrations of PFAS occur especially when UWWTPs receive effluent from PFAS manufacturers, PFAS-using industries, storm water from airports and fire-fighting sites or landfill leachates. Other sources are of a more diffuse character, reflecting the occurrence of PFAS in domestic products used in households. Sewage sludge from these sources usually presents low levels of PFAS.

Various experiments aimed at determining the half-life of PFAS in soils give results ranging from several tens to several hundreds of years [52].

4.3.12.3 Legislative framework

In September 2024, the European Commission adopted new measures under the REACH Regulation to protect human health and the environment by restricting the use of undecafluorohexanoic acid (‘PFHxA') and PFHxA -related substances. These sub-groups of per- and polyfluoroalkyl substances (‘PFAS') are very persistent and mobile in water, and their use in certain products poses an unacceptable risk to human health and the environment.

The PFHxA restriction adopted is focusing on uses where the risk is not adequately controlled, alternatives are available, and socio-economic costs will be limited in comparison to the human health and environmental benefits.

The restriction will ban the sale and use of PFHxA in consumer textiles, such as rain jackets; food packaging, like pizza boxes; consumer mixtures such as waterproofing sprays; cosmetics like skin care products; and in some firefighting foam applications like for training and testing, without compromising safety. It does not affect other applications of PFHxA, for example in semiconductors, batteries or fuel cells for green hydrogen.

The consolidated version of the Regulation 2019/1021 – POPs legislation – has set the limit of 0,025 mg/kg for products (Annex 1) both for PFAS and PFOA. The same regulation, in its Annex IV (presence of substances in the waste), sets a limit of 50 mg/kg for PFOS, and 1mg/kg for PFOA and its salts, and 40 mg/Kg for the sum of PFOA-related compounds.

Additionally, the national authorities of Denmark, Germany, the Netherlands, Norway and Sweden have submitted a joint Annex XV PFAS restriction dossier to ECHA. The 14th edition of the Background Document was released by ECHA in August 2025.

Biosolids

In Denmark, an indicative threshold concentration of 15 µg/kg DS was proposed in March 2023 for the sum of 4 PFAS (PFOA, PFOS, PFNA and PFHxS).

In Germany, the Sewage Sludge Ordinance (Klärschlammverordnung) of October 2017 indirectly stipulates a threshold value for PFASs for the soil-related recovery of sewage sludge by referencing the Fertilisers Ordinance (Düngemittelverordnung) of May 2015. The latter generally sets a threshold value of 100 µg/kg total PFOS plus PFOA for both source materials of fertilisers as well as the fertilisers themselves; where the concentration exceeds 50µg/kg this must be indicated on the label.

In Austria, there is a limit value of 100µg PFOS+PFOA /kg DS in sewage sludge used on agricultural soils.

In Norway a limit value has been set to 40µg/kg DS for the sum of PFOS+PFOA.

Soils

In Sweden, developed guideline values take into account the risks posed by a contaminated land area for people staying in the area (health risks), as well as the risks that the pollution means for the environment within the area (soil environment) and downstream the area (groundwater and surface water).

In Norway, a revised report made for the national Environmental Protection Agency (EPA) suggests normative values for contaminated soils, lowering the value for PFOS from 0.1 to 0.002mg/kg and establishing a new one 0.011 mg/kg for PFOA.

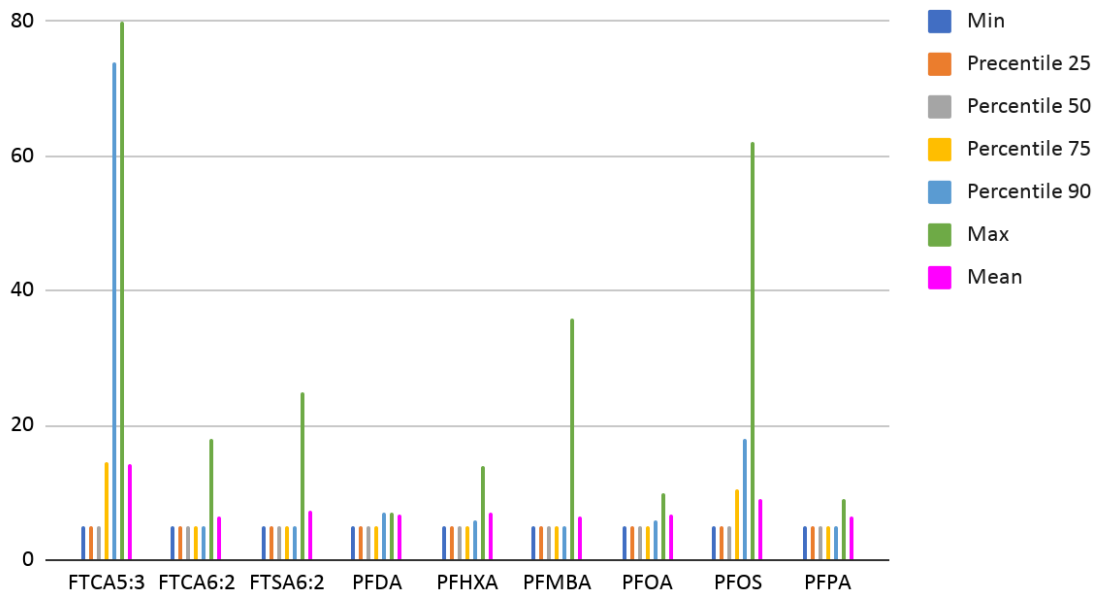
4.3.12.4 Biosolids Observatory results for PFAS

Forty different PFAS were analysed (see Annex D). Among these, the ones listed in Table 18 had at least one result above the quantification limit.

Table 18 : Quantification limits and detection frequency for PFAs

Substance	Quantification Limit (µg/kg DS)	Detection Frequency
FTCA5:3	5	66 %
FTCA6:2	5	7 %
FTSA6:2	5	3 %
PFDA	5	21 %
PFHXA	5	10, %
PFMBA	5	3 %
PFOA	5	17 %
PFOS	5	55 %
PFPA	5	7 %

Figure 16 : PFAS concentration in µg/kg DS



FTCA5:3 was the most often detected PFAS but also the one with the highest mean and max concentrations, respectively 80 and 14.2 µg/kg DS. PFOS was detected in half of the biosolids samples with an average concentration of 9.1 µg/kg DS while PFOA was detected in less than 20% of the samples with an average concentration of 6.8 µg/kg DS.

The threshold limit value of 100 µg/kg DS for PFOS + PFOA set by Germany and Austria was systematically complied with for the different biosolids samples. The limit of 40 µg/kg for the same compounds set by Norway was exceeded by only one sample. It was not possible to determine compliance with the Danish indicative threshold concentration of 15 µg/kg DS for PFAS4 due to the limits of quantification (5 µg/kg DS).

4.3.13 Pharmaceuticals

4.3.13.1 General data on Pharmaceuticals

Numerous medicinal products are used in human and veterinary medicine. Several thousand of active substances are produced by the pharmaceutical industry and then used in the composition of more than ten thousand marketed products.
Over the last decade, increasing amounts of pharmaceutical residues have been measured, primarily in surface waters but also in ground or drinking water and soils.

Pharmaceutical substances discharged into wastewater are primarily present in dissolved form and are only weakly absorbed by the particulate phase.

Consequently, their migration into sludge remains relatively limited. Their removal varies during wastewater treatment. Measured removal efficiencies show increasing efficiency in primary, secondary, and tertiary processes. Primary processes generally achieve only low removal of pharmaceutical substances, with efficiencies below 30%. Secondary biological treatments, such as extended aeration activated sludge, achieve efficiencies exceeding 70% for half of the active substances considered [53].

The hydrophobicity of pharmaceuticals is a key factor that determines their affinity for the aqueous phase of wastewater or for their migration into biosolids. Pharmaceuticals with log Kow values less than 0.4 are present at higher concentrations in effluent and thus are released to the aqueous environment, whereas those with a log Kow greater than 1.0 are primarily found in biosolids and thus can reach the terrestrial environment.

4.3.13.2 Pharmaceuticals in the environment

The various pathways of pharmaceutical products and their fate into the environment are presented below.

Pharmaceuticals production sites

Pharmaceutical substances and drug production sites can be the first point of entry of these contaminants into the environment due to the discharge of their wastewater into the urban sewage system.

This industry is divided into two sectors most of the time located in different production sites:

- The primary industry, responsible for the production of the active pharmaceutical substance.

- The manufacturing industry, more specifically, responsible for the preparation and packaging of the drug; one that may contain several active substances and adjuvants.

For the primary phase industry, the pharmaceutical substances' discharge comes primarily from process water. This consists mostly of cleaning water from manufacturing processes which may, in some cases, include production of liquid substrates. In the manufacturing sector, only the production of liquid medicines is likely to produce wastewater.

Overall, in the pharmaceutical industry, discharges of pharmaceutical substances residues into wastewater are very low due to the very high cost of

the active ingredient. As this sector is highly regulated it is unlikely that any liquid or solid substance enters directly in the environment.

Excretion after drug administration to patients

The excretion of pharmaceutical residues by human beings is the main source of releases of these substances into the environment, far ahead of the inappropriate disposal of unused medicines and the discharge of effluents from pharmaceutical industries.

After administration, the active substance is transported to the target organs in order to produce the desired therapeutic effect.The elimination of the active substance or its metabolites takes place mainly in the liver and kidneys before ending up in the urine or feces. Ultimately, it is estimated that 30 to 90% of ingested doses of active substances are excreted, either in their original form or as active metabolites. This excretion rate depends on the properties of the active substance, its method and dose of administration, as well as the patient's characteristics.

Hospitals concentrate a large patient population, leading to the use of vast quantities of medicines of extremely varied types. Hospital effluents, although more concentrated in drug residues than urban wastewater, represent on average only 15 to 20% of the total load of these substances entering urban wastewater treatment plants. Only liquid or solid waste resulting from the use of radioactive cancer drugs is considered hazardous waste. Due to their environmental impact, their disposal is strictly regulated.

Animal manure and biosolids land application

Another source of release of pharmaceutical active substances into the environment, classified as a dispersed source, is the land application of organic material such as biosolids or animal manure.

The release of pharmaceutical active substances into the environment due to the use of veterinary medicines, considering the considerable quantity of substances used, is deemed to be significantly higher than the one resulting from biosolids. Animal manure is not treated in any way, and as a result, all the pharmaceutical substances it may contain would end up in the soil if animal manure is applied to it.

Water reuse

Reuse of treated wastewater, in particular for food crop production, is becoming a more extended practice due to climate change and water scarcity, and is promoted through the recast UWWTD and the European Water Resilience Strategy . This practice offers many benefits, such as reducing the pressure on freshwater resources and increasing food production. However, it may result in

the release of pharmaceutical substances into the environment, and more specifically onto soils.

Fate and behaviour of pharmaceuticals in soils

Pharmaceuticals' fate in soils vary widely, with half-lives varying from days to years. These differences are due to differences in soil properties (moisture content, OC and clay content, pH, and soil bioactivity), soil temperature, and the physicochemical properties of the substance itself (lipophilicity and the degree of dissociation). Pharmaceuticals can either be retained in the topsoil or leached into groundwater and flow towards surface water. Pharmaceuticals migration into the soil depends on the intensity of their sorption on the soil solid phase particles. The sorption intensity affects the bioavailability of these substances and their persistence in the soil environment.

There are three phases involved in the disappearance of a pharmaceuticals in soil: the lag phase, which is a short period of time during which the substance is maintained at a certain concentration; the dissipation phase, which is relatively rapid in terms of disappearing from the soil; and the persistence phase, which is responsible for the persistence of the pharmaceuticals and is expressed in hours, days, weeks, months, and even years. Carbamazepine has the greatest persistence in soil (>40 days), followed by diclofenac, triclosan sulfamethazine (<20days) [54].

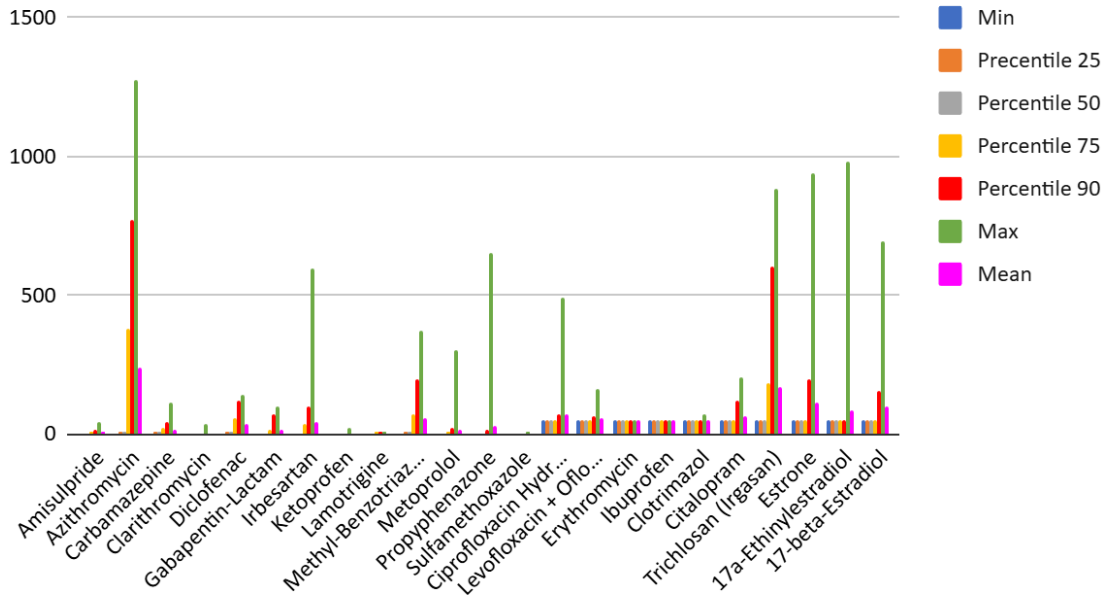
4.3.13.3 Legislative framework

Directive 2013/39/EU identified a list of priority substances, the emissions of which have to be reduced or phased out. As a result, compounds such as some pharmaceuticals (macrolide antibiotics, diclofenac, sulfamethoxazole, and venlafaxine) have been included in various pieces of EU legislation (such as the Implementing Decisions (EU) 2015/495 and (EU)2020/1161, or the Implementing Regulation (EU) 2018/840) due to their prevalence, persistence, and lack of regulation, aiming to meet goals such as the zero emissions target announced in the European Green Deal, and linked to the Sustainable Chemicals Strategy. In the context of this initiative, the Strategic Approach on Pharmaceuticals in the Environment and the Pharmaceuticals Strategy for Europe, highlight the environmental and potential health impacts of pollution from pharmaceutical residues and list a number of actions to address these challenges. In October 2025 the Council presidency and the European Parliament’s representatives reached a provisional political agreement on a proposed directive to review and update the lists of pollutants affecting surface waters and groundwater and corresponding regulatory standards. This agreement introduces pharmaceuticals into the priority lists.

4.3.13.4 Biosolids Observatory results for pharmaceuticals

Twenty three different pharmaceuticals were analysed in 30 biosolids samples (see Annex D), for all of which the detection limit was exceeded in all the biosolids samples.

Figure 17 : Pharmaceuticals concentrations in µg/kg DS



Families of pharmaceutical compounds with the highest average concentrations include antibiotics (azithromycin 239 µg/kg DS, ciprofloxacin 68 µg/kg DS, levofloxacin + ofloxacin 56 µg/kg DS, erythromycin 50 µg/kg DS), antibacterials (triclosan 166 µg/kg DS, clotrimazole 51 µg/kg DS), hormones (estrone 114 µg/kg DS, 17-beta-estradiol 98 µg/kg DS, 17a-ethinylestradiol 82 µg/kg DS) and antidepressants (citalopram 63 µg/kg DS). Some pharmaceuticals such as clarithromycin, irbesartan, ketoprofen, metoprolol, propyphenazone and 17a-ethinylestradiol have maximum values 10 to 20 times higher than average values.

4.3.14 Phthalates

4.3.14.1 General data on phthalates

Phthalates are commonly added to plastics to increase their flexibility and transparency; they are used to soften polyvinyl chloride (PVC). Phthalates are also used in industrial applications as well as in cosmetics, children's toys and feeding bottles, pharmaceutical applications, and medical devices. Most widely used phthalates are di(2-ethylhylhexyl) phthalate (DEHP), benzyl butyl phthalate (BBP), diethyl phthalate (DEP) and di-n-butyl phthalate (DBP). Phthalates are grouped into: high-molecular-weight (HMW) with 7 to 13 carbon chains and

low-molecular-weight (LMW) with 3 to 6 carbon chains. DEHP ($C_{24}H_{38}O_4$) belongs to the HMW whereas; DMP ($C_{10}H_{10}O_4$), DEP ($C_{12}H_{14}O_4$), and DBP ($C_{16}H_{22}O_4$) belong to the LMW. Phthalalates' physicochemical properties depend on their chemical structure and alkyl chain length.These properties strongly influence their behavior, fate/transport, and as well as their degradation in the environment.

4.3.14.2 Phthalates in the environment

Phthalates are not chemically bonded to the plastics or PVC and can be easily released in the environment via volatilization or leaching. High concentrations of phthalates have been observed in indoor air and house dust. Many phthalates, especially DEHP and BBP, are ubiquitously present in air, water, soil and biosolids.

The main sources of phthalates emissions in the atmosphere are anthropogenic activities including manufacturing, distribution, consumption, and discharge, which are resulting in long-term transport in the atmosphere. DnBP and DEHP are the most common phthalates found in the urban atmosphere. DEHP is abundant in the dust phase, whereas DIBP and DBP are predominant in the gas phase. Atmospheric pathways, though secondary, occur through volatilization from open-use products and incineration incomplete combustion, facilitating deposition onto remote ecosystems via wet and dry fallout.

Phthalates enter ecosystems primarily through leaching from plastic products, as these compounds are physically entrapped rather than covalently bonded to the polymer matrix, enabling gradual migration under environmental stresses such as abrasion, hydrolysis, and UV exposure. This process is exacerbated in polyvinyl chloride (PVC) materials, where phthalates constitute 20-50% by weight, leading to sustained release over years. Industrial manufacturing and processing represent key point sources, with emissions occurring via volatilization, wastewater effluents, and solid waste disposal; untreated or partially treated industrial discharges introduce phthalates directly into rivers and coastal waters, where concentrations can exceed 10 µg/L in heavily industrialized areas. Municipal wastewater treatment plants are receiving phthalates from household products like flooring, cosmetics, and packaging leach during use and enter sewage systems. Significant phthalates concentrations can be found in surface runoff and in domestic wastewater with concentrations varying from 1 µg L^{-1} to 100 µg L^{-1}, with DEHP being the compound detected at the highest concentration. Due to their low water solubility phthalates are not easily mineralized in WWTPs and are absorbed on suspended organic matter.

High concentration of phthalates can also be found in landfill leachate following their release by various kinds of waste. Landfills amplify releases via leachate

generation from buried plastics and consumer waste, where phthalates migrate into groundwater and surface waters at rates influenced by moisture and anaerobic conditions, often necessitating specialized liners to mitigate off-site transport

In soils, phthalates are discharged from agricultural activities (e.g., plastic film mulching and plastic waste) and different inputs (e.g., fertilizers, compost and biosolids). DEHP, DEP, and DMP are the dominant compounds in contaminated soil [55].

Aerobic soils with active microbial communities can degrade many phthalates relatively rapidly, with estimated half-lives ranging from about 1 to 75 days. A mid-range estimate might be around 30 days [56].

4.3.14.3 Legislative framework

In the European Union, restrictions on phthalates were initially implemented through an emergency ban in 1999 on DEHP, DINP, DIDP, and DNOP in PVC toys for children under three years, due to concerns over potential migration and exposure risks to young children, followed by a permanent ban on DEHP, DBP, and BBP in toys and childcare products exceeding 0.1% by weight under the Toy Safety Directive 2009/48/EC. These measures were extended under REACH Regulation (EC) No 1907/2006, which in 2018 added restrictions on DIBP alongside the prior three, and further expanded in 2020 to limit four additional phthalates (DIHP, DCHP, DnHP, DIBP) to 0.1% in articles like consumer plastics, reflecting a precautionary approach to mitigate suspected reproductive and developmental toxicity based primarily on rodent studies showing anti-androgenic effects at high doses.

Regulation (EU) 2023/2482 deferred the DEHP sunset date in medical devices to May 27, 2025, while allowing authorization applications until January 1, 2029, and postponing the effective ban to July 1, 2030, unless exemptions are granted.

Denmark is the only Member State that has set a limit value of 50 mg/kg DS for DEHP in Biosolids. In addition, the Lombardy Region (IT) has set a limit value of 100 mg/kg DS for DEHP.

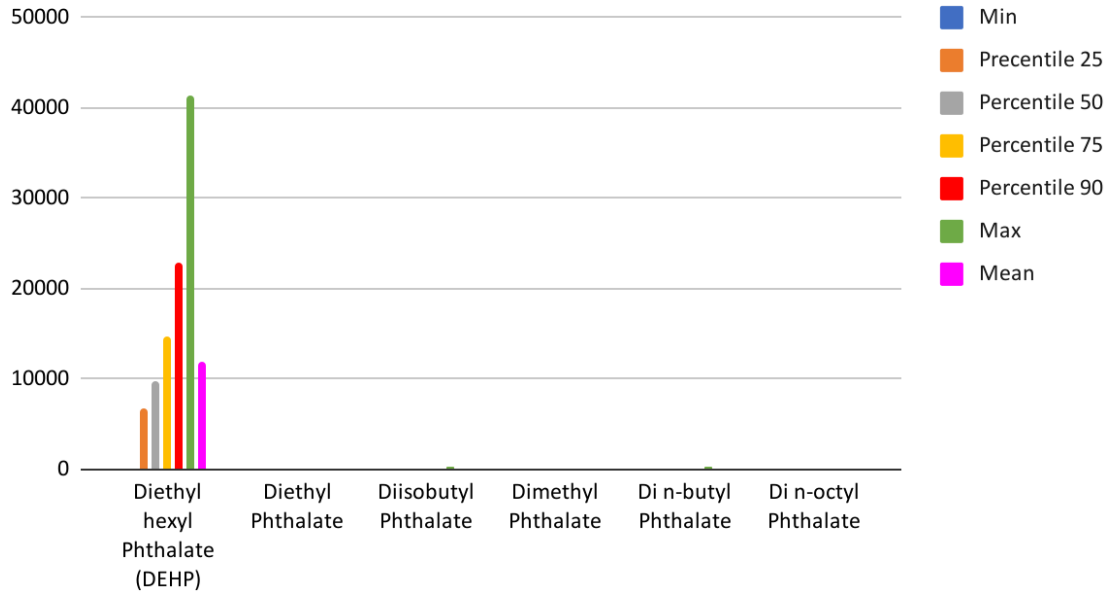
4.3.14.4 Biosolids Observatory results for phthalates

Seven phthalates were analysed in 20 biosolids samples)see Annex D).. Table 19 includes those that had at least one result above the quantification limit.

Table 19 : Quantification limits and detection frequency for Phthalates

Substance	Quantification Limit (µg/kg DS)	Detection Frequency
Diethyl hexyl Phthalate (DEHP)	25	100 %
Diethyl Phthalate	25	15 %
Diisobutyl Phthalate	50	15 %
Dimethyl Phthalate	25	5 %
Di n-butyl Phthalate	25	20 %
Di n-octyl Phthalate	25	5,00%

Figure 18 : Phthalates concentrations in µg/kg DS



DEHP is the only phthalate present in significant concentrations with an average value of 11 982 µg/kg DS and a maximum value of 41 355 µg/kg DS. All samples are compliant with the most stringent limit of 50 mg/kg DS set by the Danish regulation.

4.3.15 Siloxanes

4.3.15.1 General data on siloxanes

Siloxanes constitute a class of organosilicon compounds featuring a backbone composed of alternating silicon and oxygen atoms connected by Si-O-Si linkages.

Polysiloxanes, particularly polydimethylsiloxanes (PDMS), are integral to sealants and adhesives in construction and automotive engineering, offering elasticity, adhesion to diverse substrates, and resistance to weathering and chemicals. In building applications, they seal joints in facades, windows, and sanitary installations, maintaining integrity under thermal expansion and UV exposure. Automotive uses include gaskets, hoses, and body adhesives that endure engine heat up to 260°C and vibrational stress.

Polysiloxanes serve as antifoaming agents in chemical processing, oil refining, and wastewater treatment, where their low surface tension disrupts foam stability without altering process chemistry. In coatings for marine and industrial structures, hybrid polysiloxane systems enhance corrosion resistance and durability through covalent bonding and hydrophobic surfaces. Electrical insulation applications in cables and potting compounds exploit their thermal conductivity and flexibility.

Siloxanes, especially volatile methylsiloxanes (VMS) like cyclotetrasiloxane (D4), cyclopentasiloxane (D5), and cyclohexasiloxane (D6), serve as solvents, carriers, and emollients in personal care products such as shampoos, conditioners, antiperspirants, and skin creams. In hair care formulations, non-volatile siloxanes such as dimethicone act as conditioning agents. Cosmetic foundations and lotions incorporate cyclomethicone and dimethicone crosspolymers to create breathable barriers, enhance wear resistance, and provide a lightweight, non-comedogenic feel on skin. Approximately 16% of analyzed European cosmetics contain cyclic VMS, predominantly D5 [57].

4.3.15.2 Siloxanes in the environment

Volatile methylsiloxanes (VMS), particularly cyclic variants such as octamethylcyclotetrasiloxane (D4), decamethylcyclopentasiloxane (D5), and dodecamethylcyclohexasiloxane (D6), exhibit high environmental persistence due to their chemical stability, with atmospheric half-lives ranging from approximately 7 to 9 days for D4 and D5 via reaction with hydroxyl radicals, and longer for D6. In aquatic and soil compartments, persistence is greater, as biodegradation rates are negligible under aerobic conditions, with probabilities approaching zero for D5 in water or soil, and hydrolysis half-lives exceeding months to years at neutral pH. Mobility is dominated by volatilization, as >90% of VMS emissions partition to air due to high Henry's law constants (e.g., air-water partition for D5 ≈ 0.3-0.5 dimensionless), facilitating long-range atmospheric transport before deposition. In soils, sorption to organic matter is strong (log Koc >5 for cyclic VMS), reducing leaching potential.

4.3.15.3 Legislative framework

In the European Union, D4 was designated a substance of very high concern (SVHC) in 2018 for PBT characteristics, leading to a restriction in wash-off cosmetic products effective January 31, 2020, limiting concentrations to below 0.1% by weight to mitigate aquatic releases.

Regulation (EU) 2024/1328 amends REACH Annex XVII to broadly restrict D4, D5, and D6, prohibiting their placement on the market in mixtures or articles at concentrations of 0.1% or more for consumer and professional uses starting June 6, 2026. This includes bans in leave-in cosmetics from June 6, 2027, and medical devices from June 6, 2031, with derogations for specific industrial applications such as silicone polymer production and non-metal surface treatments, aiming to reduce environmental emissions by up to 90%. There is no limit set for siloxanes in biosolids at European or Member State level.

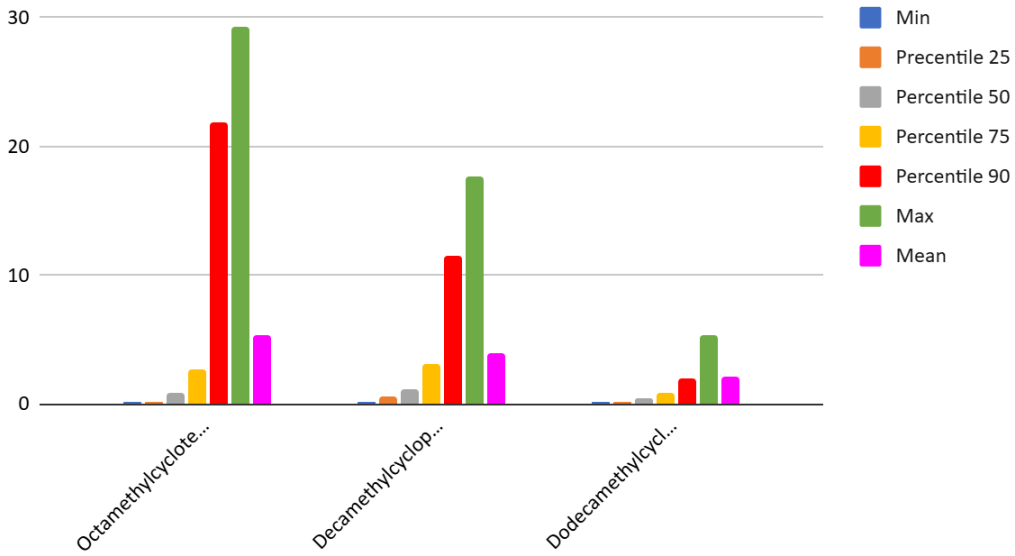
4.3.15.4 Biosolids Observatory results for siloxanes

All three siloxanes analysed in 23 biosolids samples had at least one result above the quantification limit.

Table 20 : Quantification limits and detection frequency for Siloxanes

Substance	Quantification Limit (mg/kg DS)	Detection Frequency
Octamethylcyclotetrasiloxane D4	0,2	65 %
Decamethylcyclopentasiloxane D5	0,2	87 %
Dodecamethylcyclohexasiloxane D6	0,2	70 %

Figure 19 : Siloxanes concentrations in mg/kg DS



Siloxanes were detected in more than two thirds of biosolids samples at average concentrations varying from 2 to 5 mg/kg DS.

4.3.16 Volatile organic compounds (VOCs)

4.3.16.1 General data on VOCs

Volatile organic compounds (VOCs) are compounds that have a high vapor pressure and low water solubility. Many VOCs are human-made chemicals that are used and produced in the manufacture of paints, pharmaceuticals, and refrigerants. VOCs typically are industrial solvents, such as trichloroethylene; fuel oxygenates, such as methyl tert-butyl ether (MTBE); or by-products produced by chlorination in water treatment, such as chloroform. VOCs are often components of petroleum fuels, hydraulic fluids, paint thinners, and dry cleaning agents. VOCs are common groundwater contaminants.

4.3.16.2 VOCs in the environment

VOCs have high volatility, mobility and strong resistance to degradation that allows them to be transported over long distances in the environment once released.

VOCs generated by anthropogenic activities (both domestic and industrial processes) include metal surface degreasing, textile cleaning, food extraction, fertilizers and pesticide application, septic system, chlorination, fumigation, traffic, hydrocarbon fuel evaporation, petroleum storage, distribution and storage, landfill, printing, building. BTEX accounted for 95% of the total VOCs emitted by automotive engines. BTEX were reported to be the most abundant VOCs found in the urban atmosphere, surface water and industrial settings. Petrochemical industrial sources account for 21–51% of the total VOC emissions followed by vehicular traffic (15–34%), and biogenic emissions [58].

VOCs enter the soil surface from the atmosphere as vapors, liquids and aqueous solution. Then, move throughout the soil profile due to diffusion, convection and advection under the effect of gravity. The amount of VOCs adsorbed on the surface of soil particles depends on the nature of the soil, including permeability, porosity, surface area and inorganic and organic contents of soil. However, the presence of water may actively reduce the adsorption capacity. Soils with higher organic content will tend to adsorb higher amount of VOCs on their surface

Only a small fraction of VOCs released into the environment is sequestered in soil and sediment compartments [59]. Overall, VOCs are ubiquitous in the

environment with the atmosphere being the major sink for these chemicals. Inhalation is the major source of human exposure to VOCs and exposure in industrial and occupational settings can be orders of magnitude higher than ambient exposures. Indoor air concentrations of VOCs in residential settings were in the order of several tens to hundreds of µg/m3 whereas those in occupational settings were on the order of tens of mg/m3. Drinking water and food are minor sources of human exposure to VOCs.

4.3.16.3 Legislative framework

The primary tool for controlling VOCs from industrial sources is the EU Directive 2024/1785, also known as IED 2.0.There is no limit set for VOCs in biosolids at European or Member State level. The Lombardy Region (IT) has a limit value of 100 mg/kg for toluene in biosolids.

4.3.16.4 Biosolids Observatory results for VOCs

All of the 18 t VOCs that were analysed in 23 biosolids samples (listed in Table 21) had at least one result above the quantification limit.

Table 21 : Quantification limits and detection frequency for VOC's

Substance	Quantification Limit (mg/kg DS)	Detection Frequency
Cis 1,3-dichloropropylene (Z)	0,1	96 %
Carbone tetrachloride	0,01	100 %
Chloroform	0,01	100 %
Vinyl chloride	0,05	100 %
Dibromochloromethane	0,01	96 %
Dichlorobromomethane	0,01	100 %
Dichloromethane	0,5	100 %
Ethylbenzene	0,01	100 %
Hexachloroethane	0,01	96 %
Isopropylbenzene (cumene)	0,01	100 %
MTBE (methyl tertiobutyl ether)	0,01	100,00%
n-butylbenzene	0,01	96 %
Styrene	0,05	100 %
Trans 1,2-dichloroethylene (E)	0,01	100 %
Trans 1,3-dichloropropylene (E)	0,1	100 %
Trichlorofluoromethane	0,1	100 %
Tert butylbenzene	0,01	100 %
Tetrachlorethylene (perchlorethylene)	0,01	100 %
Trichlorethylene	0,01	100 %
Xylene o	0,01	100 %

Table 21 (continue) : Quantification limits and detection frequency for VOC's

Substance	Quantification Limit (mg/kg DS)	Detection Frequency
Xylenes m + p	0,02	100 %
Hexachlorobutadiene	0,01	100 %
1,1,1,2-tetrachloroethane	0,01	100 %
1,1,1-trichloroethane	0,01	96 %
1,1,2-trichloroethane	0,01	100 %
1,1,2-trichlorotrifluoroethane	0,1	100 %
1,1-dichloroethane	0,01	100 %
1,1-dichloroethylene	0,01	100 %
1,2,3-trimethylbenzene	0,01	96 %
1,2,4-trimethylbenzene	0,01	96 %
1,2-dibromoethane	0,01	100 %
1,2-dichloroethane	0,05	100 %
1,2-dichloropropane	0,01	100 %
1,3,5-trimethylbenzene (mesityene)	0,01	96 %
2,3-dichloropropylene	0,01	100 %
2-chlorotoluene	0,05	100 %
3-chloropropene	0,1	100 %
3-chlorotoluene	0,05	100 %
4-chlorotoluene	0,01	100 %
Benzene	0,01	100 %
Bromochloromethane	0,01	100 %
Bromoform	0,01	100 %
Cis 1,2-dichloroethylene (Z)	0,01	100 %
4-isopropyl toluene (p cymene)	0,01	100 %
Toluene	0,05	100 %

Figure 20 : VOCs concentrations in mg/kg (1/2) DS

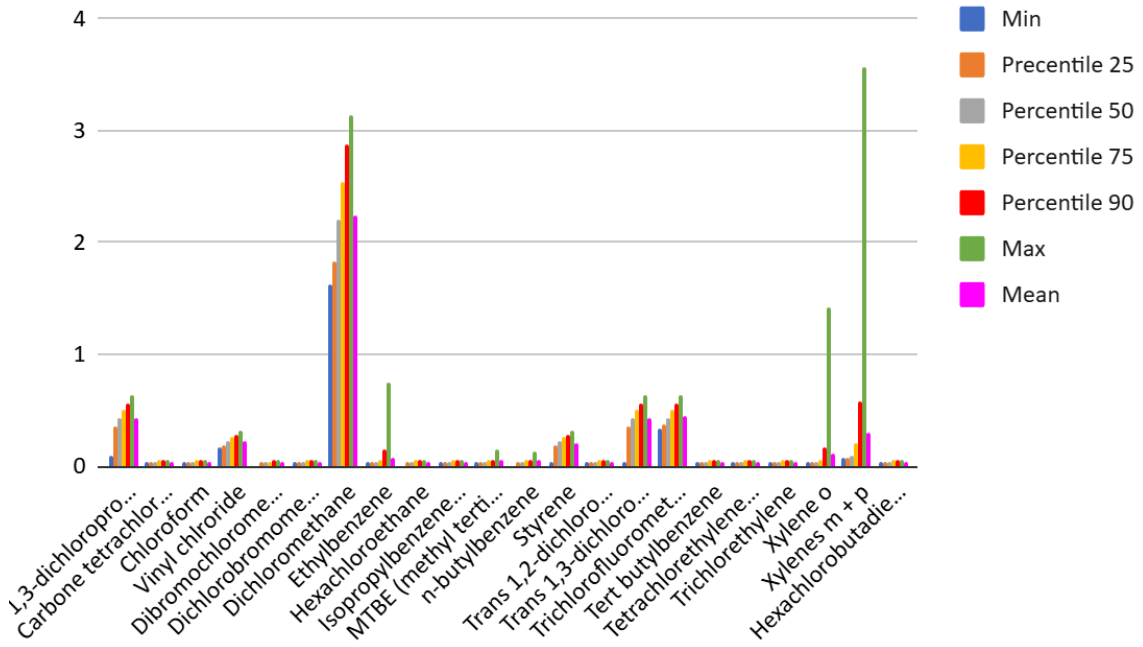


Figure 21 : VOCs concentrations in mg/kg (2/2) DS

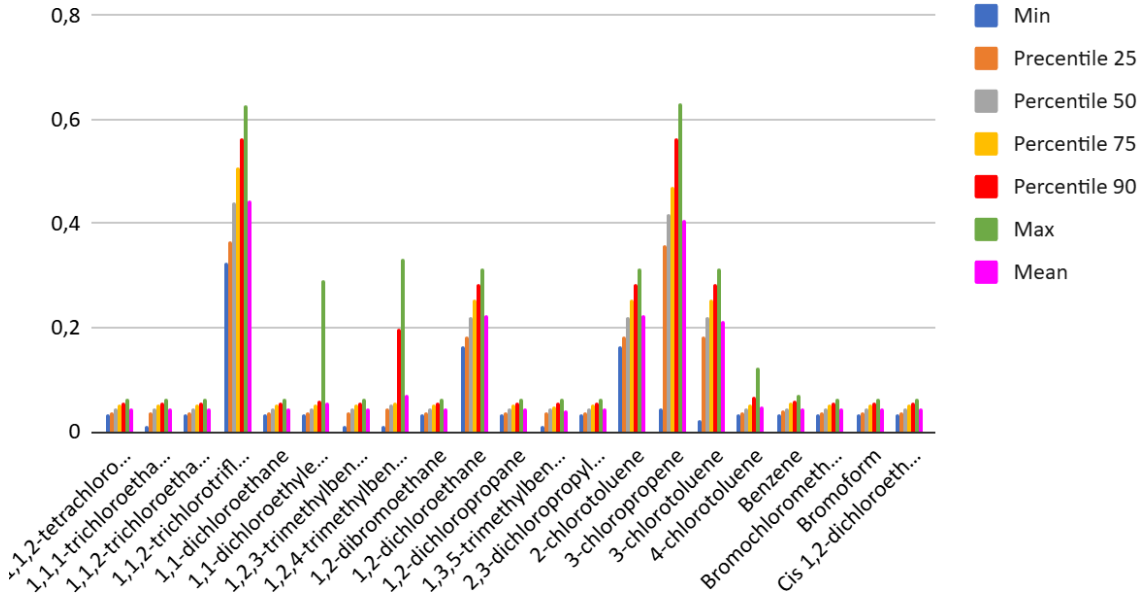
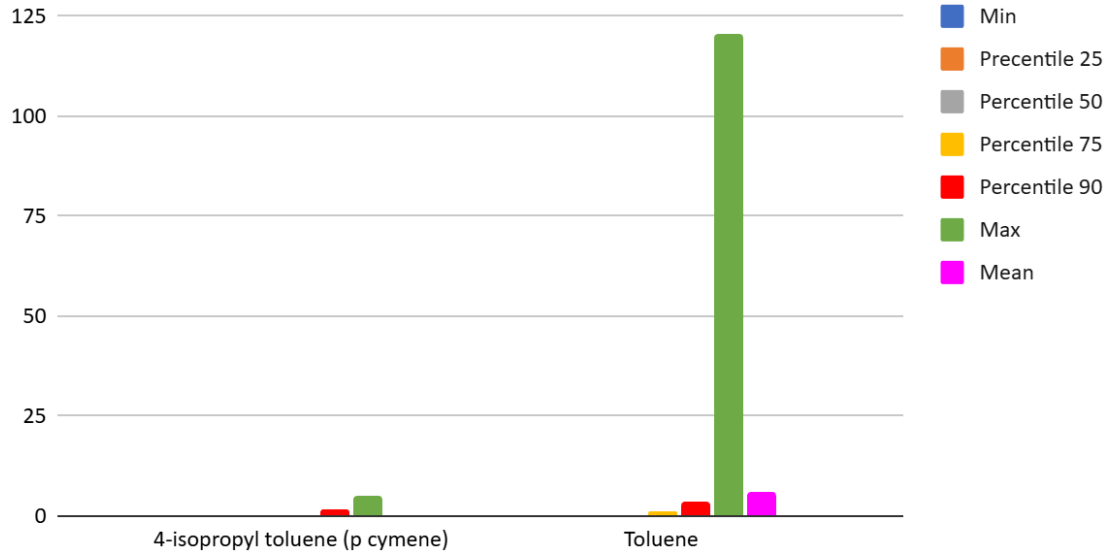


Figure 22 : 4-Isopropyl toluene and Toluene concentrations in mg/kg DS



VOCs were systematically detected in all biosolids samples. Toluene and dichloromethane were, by far, the most abundant compounds, with average concentrations of 6 and 2,25 mg/kg DS, respectively. Next were cis-1,3-dichloropropylene, trans-1,3-dichloropropylene, trichlorofluoromethane, and 3-chloropropene, with average concentrations close to 0.4 mg/kg DS. All other compounds were present in trace amounts. Two significant peaks were recorded for xylenes O and M+P, with maximum values of 1.42 and 3.6 mg/kg DS, respectively. One sample exceeded the threshold limit set for toluene by the Lombardy Region.

5 OTHER BIOSOLIDS TESTING CAMPAIGNS

The results obtained within the framework of the Biosolids Observatory were compared, whenever possible, with those of previous analytical campaigns carried out by other entities or organisations. The most relevant ones are briefly presented below.

5.1 FATE SEES: Occurrence and levels of selected compounds in European Sewage Sludge Samples [60]

This analytical campaign, the results of which were published in 2012, was one of the first of its kind. It allowed to identify and prioritize the relevant compounds to be considered in European regulations concerning sewage sludge. It synthesizes the results of analyses of representative sewage sludge samples from across Europe, focusing on the presence and concentrations of compounds of concern, many of

which had never been assessed on a pan-European scale. In total, 63 samples from 15 countries were analyzed for 22 minor and trace elements and 92 organic compounds, including ingredients in personal care and pharmaceutical products.

5.2 Norwegian Water analysis campaign [61]

A number of substances, such as organic micropollutants, arsenic, silver and medicine residues, were analysed in sludge samples taken from 17 municipal wastewater treatment plants/biogas plants during the winter season 2022/2023. Five monthly mixed samples were collected from all the facilities. This is the same type of survey that has been carried out approximately every five years since 1996/97.

The substances analysed were PAH16, brominated flame retardants (BFH), phthalates (including DEHP), nonylphenol/ethoxylates and other alkylphenols, surfactants (LAS), perfluorinated alkyl substances, bisphenols, polychlorinated biphenyls (PCB7), triclosan, siloxanes , musk compounds (galaxolide, tonalide etc.), arsenic, silver, bisphenols, organophosphorus flame retardants, UV substances, chlorinated paraffins (MCCP, SCCP, LCCP), drug residues, organotin compounds (TBT, TFT, DBT, DOT) and quaternary ammonium compounds (QAC). Total organic carbon (TOC) and the nutrients nitrogen and phosphorus were also analysed.

5.3 Utilitalia analysis campaign [62]

Sludge quality data was collected from plants operated by UTILITALIA members, which manage the integrated water service in Italy. Unlike previous surveys, plants with a capacity of less than 10,000 PE were also included. The survey covers 2023, although some members also provided data for 2022.

Each water company provided, for each plant they manage, the data from the quality certificates for the sludge produced. These certificates, along with the forms, accompany all sludge loads destined for final recovery/disposal sites. The quality data requested covered the following parameters:

- Concentration of total and volatile organic compounds;
- Concentration of organic carbon, nitrogen, and phosphorus;
- Number of salmonellae, Escherichia coli, and fecal coliforms;
- Concentration of metals (As, Be, Cd, total Cr, Cr VI, Cu, Hg, Ni, Pb, Se, Zn);
- Concentration of C10-C40 hydrocarbons, ΣPAHs, ΣPCBs, PCDD/F TE, toluene, AOX, DEHP, NPE, PFOS

5.4 UKWIR analysis campaign [63]

The United Kingdom National Chemical Investigations Programme (CIP) is intended as a means of gaining a better understanding of the occurrence, behaviour, and management of trace contaminants in the environment. One area of interest throughout the CIP has been removal of substances from wastewater and the

potential for treatment processes to partition substances into sludge (untreated material produced from treatment of wastewater) and end up in biosolids (treated sludge ready for application to the land).

The substances analysed are: benzotriazole, bis(2-ethylhexyl) phthalate (DEHP), ciprofloxacin, cypermethrin, decabromodiphenyl ether (BDE) 28, decabromodiphenyl ether (BDE) 47, decabromodiphenyl ether (BDE) 99, hexabromocyclododecane (HBCDD), 4-nonylphenol, perfluorobutane sulfonic acid (PFBS), perfluorooctanoic acid (PFOA), perfluorooctanesulfonoic acid (PFOS), tributyltin (TBT), triclosan.

5.5 Analysis results comparison

Table 22 : Comparison of the mean values obtained within the framework of the Biosolids Observatory with those of other analysis campaigns and scientific publications
* as per the list of references

Substance group	Parameter	Unit	Scientific publication review*	Fate Sees	Utilitalia	Norwegian Water	UKWIR	Biosolids Observatory
Inorganic trace elements	Cu	mg/kg DS	200 - 600	257	255	180	-	253
	Zn	mg/kg DS	500 - 1800	700	690	460	-	806
	Cd	mg/kg DS	1 - 5	0.9	0.9	0.7	-	0.84
	Hg	mg/kg DS	0.1 - 4	0.4	0.6	0.4	-	0.58
Bisphenol	Bisphenol A	µ/kg DS	100 - 300	-	-	1605	-	921
Nonylphenol and nonylphenol ethoxylates	Nonylphenol	mg/kg DS	10 - 450	-	1.2	-	-	18.3
	Nonylphenol Ethoxylates	mg/kg DS	20 - 780	-	2.4	1.7	-	<0.05
	4 n Nonylphenol	mg/kg DS	0.1 - 5	-	-	< 0. 02	20.7	< 0.1
Organotins	Dibutyltin	µ/kg DS	100 - 800	-	-	27.9	-	32.9
	Dioctyltin	µ/kg DS	10 - 140	-	-	32.6	-	123.1
	Tributyltin	µ/kg DS	5 - 150	-	-	2.7	29	101.2
PAHs	PAH 16	mg/kg DS	2 - 15	-	-	0.94	-	1.21
	Phenanthrene	mg/kg DS	0.2 - 1.2	0.644	-	0.283	-	0.18
	Pyrene	mg/kg DS	0.3 - 2.5	0.698	-	0.282	-	0.165
	Fluoranthene	mg/kg DS	0.5 - 3.5	0.814	-	0.244	-	0.118
	Anthracene	mg/kg DS	0.05 - 0.25	0.724	-	0.051	-	0.071

Substance group	Parameter	Unit	Scientific publication review*	Fate Sees	Utilitalia	Norwegian Water	UKWIR	Biosolids Observatory
PBDEs	PBDE 47	µ/kg DS	10 - 250	-	-	5.6	< 0.5	4.2
	PBDE 99	µ/kg DS	10 - 250	-	-	4.5	< 0.5	5.1
	PBDE 203	µ/kg DS	0.5 - 15	-	-	-	-	2.2
	PBDE 209	µ/kg DS	200 - 3500	-	-	352	-	305.8
PCBs	∑PCB (7)	µ/kg DS	50 - 560	-	-	16	-	38
	PCB 31	µ/kg DS	5 - 25	-	-	-	-	5.8
	PCB 52	µ/kg DS	2 - 40	-	-	1.96	-	6.4
	PCB 101	µ/kg DS	1.5 - 35	-	-	2.14	-	5.7
	PCB 169	µ/kg DS	0.1 E-3 - 5 E-3	-	-	-	-	16.8
PFAs	PFOA	µ/kg DS	2 - 15	6.7		0.76	< LOQ	5.4
	PFOS	µ/kg DS	5 -50	66.4	-	13.97	46	10
	PFHxS	µ/kg DS	0.5 - 10	-	-	8.46	-	< 5
	PFNA	µ/kg DS	0.5 - 5	4.6	-	-	-	< 5
Pharmaceuticals	Ciprofloxacin	µ/kg DS	300 - 2500	-	-	-	6596	68
	Trichlosan	µ/kg DS	500 - 1600	-	-	51	4700	166
	Carbamazepine	µ/kg DS	50 - 260	-	-	66	-	16.7
	Azithromycin	µ/kg DS	20 - 1500	-	-	187	-	239
Phathalates	DEHP	mg/kg DS	18 - 150	-	3.4	27.3	25.8	11.9
Siloxanes	D4	mg/kg DS	0.3 - 17	0.49	-	0.89	-	4.29
	D5	mg/kg DS	2.5 - 35	10.82	-	5.1	-	3.02
	D6	mg/kg DS	0.01 - 6	2.82	-	2.86	-	0.83
VOCs	Toluene	mg/kg DS	0,0002 - 2.83	-	1.2	-	-	5.9

Overall, the results obtained are relatively comparable across the different analysis campaigns. However, those recorded during the FATE SEES campaign are significantly higher than those of the Biosolids Observatory for PAHs and PFAS. The concentrations of nonylphenol, phthalates, and toluene in the UTILITALIA analysis campaign are considerably lower than those of the Biosolids Observatory. The results of the analyses conducted by UKWIR differ significantly from those of the other campaigns for tributyltin, PFOS, ciprofloxacin, and triclosan. Bisphenol A is found in much higher

quantities in Norwegian biosolids than in those of the Biosolids Observatory, unlike dioctyltin and tributyltin, which are present in lower concentrations.

6 DISCUSSION

6.1 WWTP size impact

Even though the number of samples analyzed remains relatively limited and does not allow for efficient statistical processing, it is worthwhile to assess whether the contaminant concentrations in biosolids are related to the size of the wastewater treatment plants from which they originate. Table 23 below shows the percentage of cases where the minimum, average, and maximum contaminant concentrations in wastewater treatment plants with a capacity of less than 50,000 p.e. exceed those in wastewater treatment plants with a capacity between 50,000 and 250,000 p.e. and those with a capacity exceeding 250,000 p.e. Parameters of agronomic interest are not included in the calculation, nor are those for which the concentrations are below the limit of quantification (LOQ) for each of the categories compared.

Table 23 : Percentage for which minimum, average, and maximum contaminant concentrations in WWTP of less than 50,000 p.e. are lower than those recorded for larger WWTP treatment plants.

	% **Min** contaminant concentrations for WWTP < 50 000 p.e. lower than those specified in column 1	% **Mean** contaminant concentrations for WWTP < 50 000 p.e. lower than those specified in column 1	% **Max** contaminant concentrations for WWTP < 50 000 p.e. lower than those specified in column 1
Min contaminant concentrations for WWTP of 50 000 to 250 000 p.e.	**52,3%**	1,2%	0%
Min contaminant concentrations for WWTP > 250 000 p.e.	**39,5%**	8,2%	3,8%
Mean contaminant concentrations for WWTP of 50 000 to 250 000 p.e.	95,5%	**51,9%**	25,3%
Mean contaminant concentrations for WWTP > 250 000 p.e.	100%	**54,4%**	34,8%
Max contaminant concentrations for WWTP of 50 000 to 250 000 p.e.	100%	83,8%	**51,50%**
Max contaminant concentrations for WWTP > 250 000 p.e.	100%	85,4%	**68,6%**

Analysis of the collected data leads to the conclusion that for the various contaminants analyzed:

- Minimum concentrations are not consistently found in biosolids produced by small WWTP (< 50,000 p.e.); this is true in only slightly more than 50% of cases.

- The same applies to maximum concentrations. These are higher in WWTP with a capacity of less than 50,000 p.e. in 48% of cases when compared to WWTP with a capacity between 50,000 and 250,000 p.e., and in slightly more than 30% of cases for those with a capacity of more than 250,000 p.e.

Comparing average values also provides interesting information, and this across a larger dataset. For example, it should be noted that contaminant concentrations in biosolids produced in WWTP with a capacity of less than 50,000 p.e. are higher than those in WWTP with a capacity between 50,000 and 250,000 p.e. in 49% of the cases, and in 45% of the cases when compared to those on plants with a capacity greater than 250,000 p.e.

The graphs hereafter illustrate that it is not possible to establish a systematic correlation between the size of wastewater treatment plants and contaminant concentrations in sewage sludge. These concentrations depend on various parameters, such as the characteristics of non-domestic discharges into the sewage system and the treatment applied to the wastewater and sludge. Therefore, contaminant limits set by regulations are the most effective way to ensure the safe use of sewage sludge in soils, regardless of the size of the treatment plant.

Figure 23 : PFAS Mean concentrations in µg/kg DS

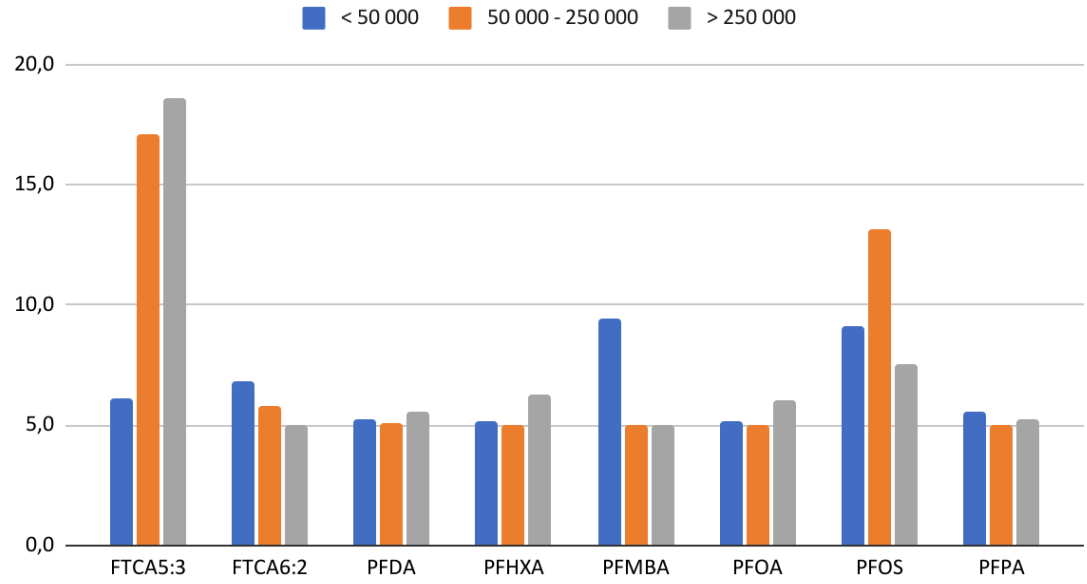


Figure 24 : PFAS Max concentrations in µg/kg DS

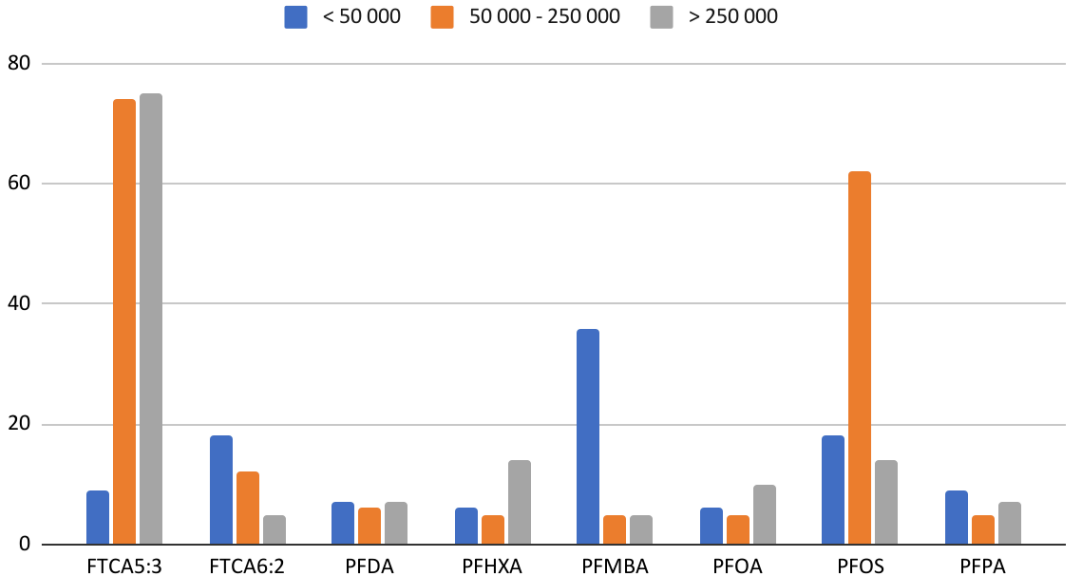


Figure 25 : PBDE mean concentrations in ng/kg DS

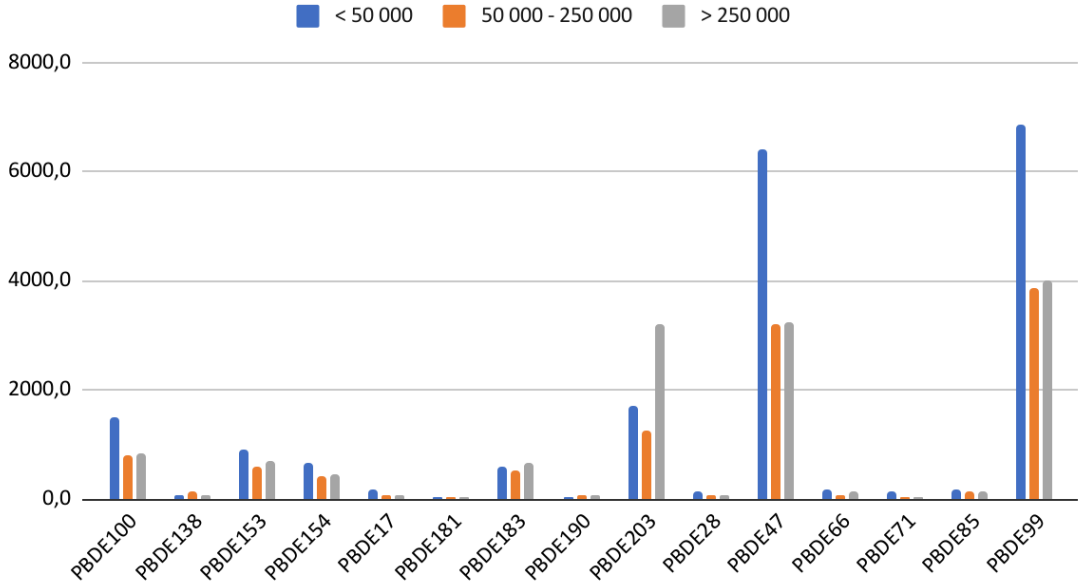


Figure 26 : PBDE max concentrations in ng/kg DS

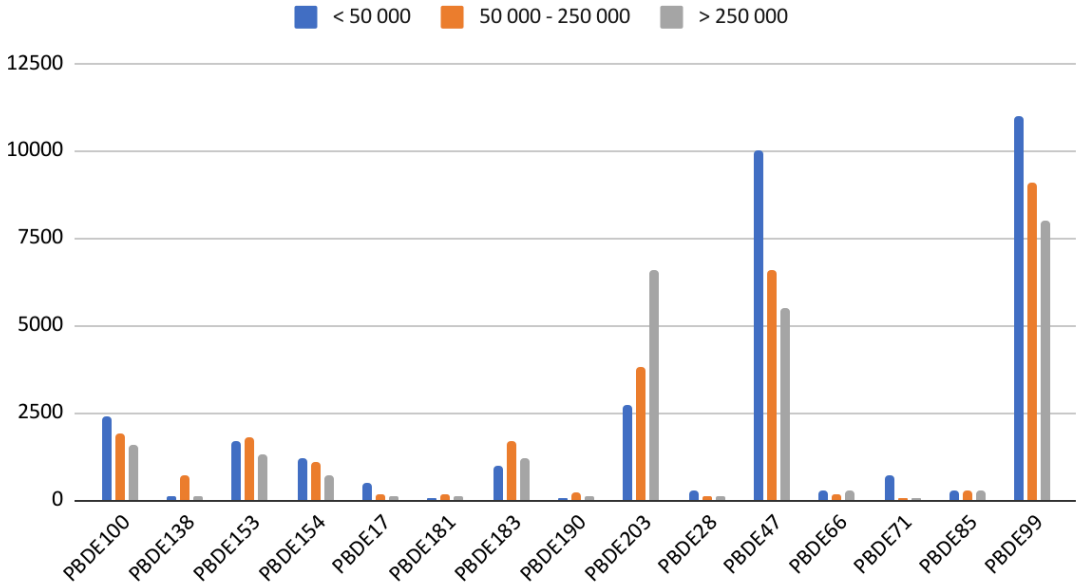


Figure 27 PCB mean concentrations in µg/kg DS

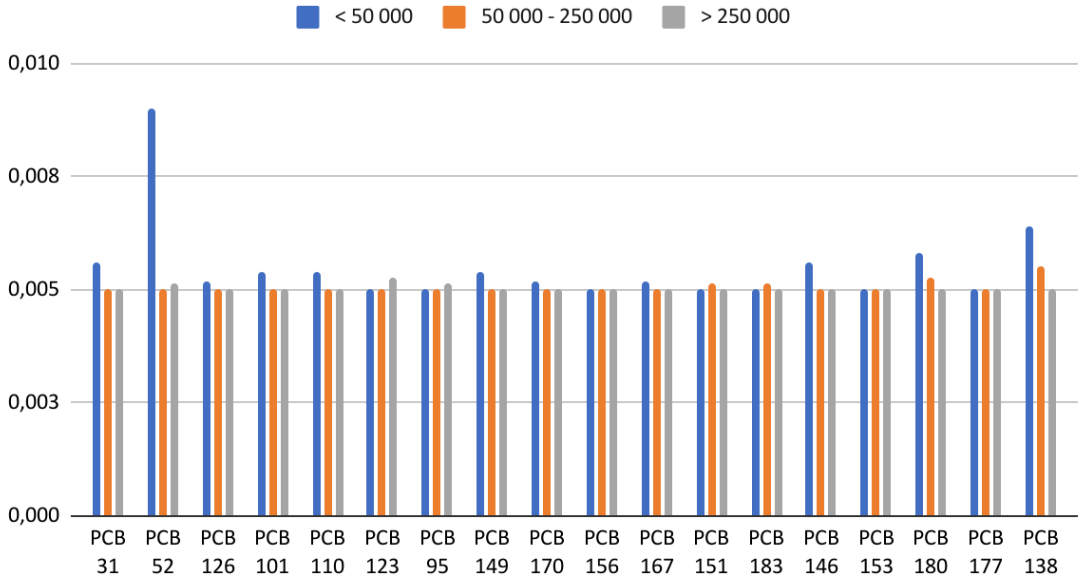
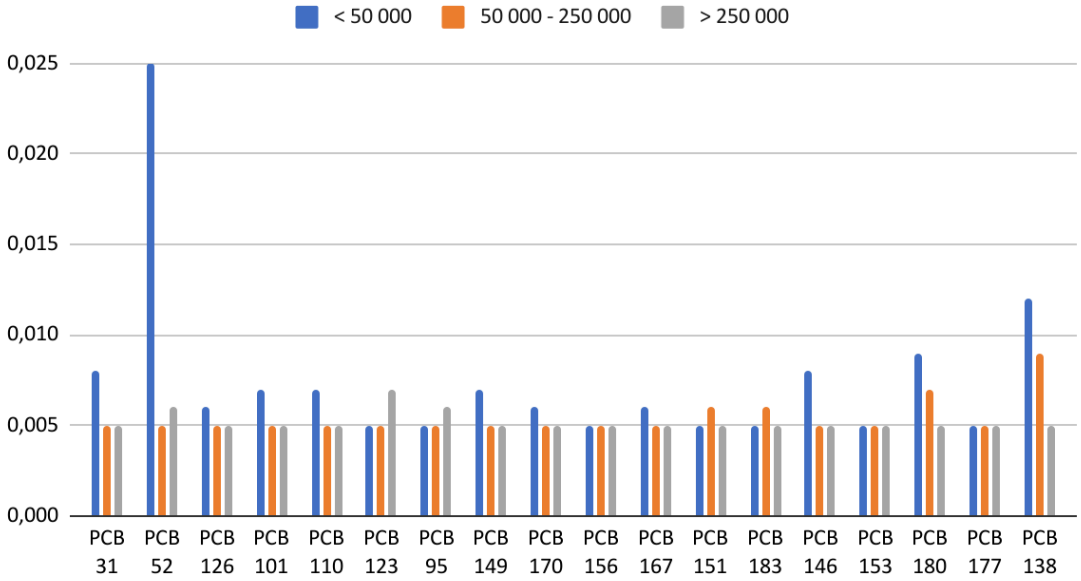


Figure 28 : PCB Max concentrations in µg/kg DS



6.2 Substances of interest to be monitored

The data collected by the biosolids observatory can be used to develop a monitoring strategy to control the risks associated with their use on soils. Identifying the substances that should be regularly analyzed is therefore essential.

The data that have been used to identify the substances that need to be monitored are outlined below:

- Soil Predicted No-Effect Concentration (PNEC) in mg/kg,
- Mammalian NOAEL No-Observed-Adverse-Effect Level (NOAEL) in mg/kg/bw/day,
- Soil half life in days,
- Mean and max concentrations in the biosolids.

The following substances were not taken into account in the development of this approach:

- Substances that are indirectly discharged into the sewer system due to the fact that they are mostly released into the air: dioxins and PAH.
- Substances for which the analysis results are all below the quantification limits: alkanes, poly-chlorinated naphthalenes.

For other substances contaminants with the highest concentrations and toxicities were selected and ranked based on the calculation of the ratios of average and maximum

concentrations in biosolids divided by PNEC soil and mammal NOAEL. The results obtained are presented in the Table 24, applying the following color code:

Purple: contaminant of limited interest to be monitored due to high soil PNEC, high mammalian NOAEL and to their concentrations in biosolids leading to Soil PNEC (mg/kg) / biosolids concentration (mg/kg) >1 and Mammalian NOAEL (mg/kg BW/day) / biosolids concentration (mg/kg) >30.

Light brown: contaminant of significant interest to be monitored due to medium soil PNEC, medium mammalian NOAEL and to their concentrations in biosolids leading to 1< Soil PNEC (mg/kg) / biosolids concentration (mg/kg) > 0.1 and 30 < Mammalian NOAEL (mg/kg BW/day) / biosolids concentration (mg/kg) > 1.

Flashy blue: contaminant of high interest to be monitored due to low soil PNEC, low mammalian NOAEL and to their concentrations in biosolids leading to Soil PNEC (mg/kg) / biosolids concentration (mg/kg) < 0.1 and Mammalian NOAEL (mg/kg BW/day) / biosolids concentration (mg/kg) < 1.

Table 24 : Identification of substances of interest

Category	Substance	Biosolids concentration (mg/kg)		Soil PNEC (mg/kg)	Mammalian NOAEL (mg/kg BW/day)	Soil half life (days)	Soil PNEC (mg/kg) / Biosolids concentration (mg/kg)		Mammalian NOAEL (mg/kg BW/day) / Biosolids concentration (mg/kg)	
		Mean	Max				Mean	Max	Mean	Max
Bisphenol	Bisphenol A	0.921	4.850	3.7	5	30	4.02	0.76	5.43	1.03
Chlorobenzene's	1,2,4-Trichlorobenzene	0.053	0.070	0.3	10	180	5.66	4.29	188.68	142.86
	1,2,3-trichlorobenzene	0.064	0.368	0.001	10	180	0.0156	0.0027	156.25	27.17
	Pentachlorobenzene	0.090	0.100	0.012	8	250	0.1333	0.1200	88.89	80.00
	Hexachlorobenzene	0.046	0.050	0.0003	0.05	2000	0.0065	0.0060	1.09	1.00
Chlorophenol	2,3,4,5- tetrachlorophenol	0.100	0.115	0.0048	3	180	0.0480	0.0417	30.00	26.09
Nonylphenol	Nonylphenol	18.300	238.500	0.3	12	60	0.0164	0.0013	0.6557	0.0503
Organotins	Monooctylin	0.158	0.201	0.5	7	45	3.16	2.49	44.30	34.83
	Dioctylin	0.123	0.808	0.006	0.3	200	0.0488	0.0074	2.44	0.3713
	Trioctylin	0.011	0.033	1	0.025	500	92.5926	30.3030	2.31	0.7576
PBDE's	Deca BDE	0.300	2.570	98	1120	3500 - 7000	326.67	38.13	3733.33	435.80
	Octa BDE 203	0.002	0.007	12	2	2900	6000	1818	1000	303
	BDE 47	0.042	0.010	0.1	0.1	1800	2.38	10.00	2.38	10.00
	BDE 99	0.051	0.012	0.1	0.01	1800	1.9608	8.3333	0.1961	0.8333
PCB	Sum PCB	0.026	0.337	0.02	0.00001	15000	0.7752	0.0593	0.00039	0.00003
PFAS	FTCA 5:3	0.014	0.080	0.05	5	300	3.52	0.63	352.11	62.50
	PFOS	0.010	0.062	0.01	0.00063	Indefinite	1.00	0.16	0.0630	0.0102
Phthalates	DEHP	11.980	41.350	13	4.8	120	1.09	0.31	0.4007	0.1161
Siloxanes	Octamethylcyclotetra siloxane D4	5.390	29.300	0.13	5	3	0.02	0.004	0.9276	0.1706
VOCs	Toluene	6.000	120.800	2.89	312	7	0.48	0.02	52.00	2.58
	Dichloromethane	0.220	0.314	0.58	6	1	2.64	1.85	27.27	19.11
	Xylene M+P	0.310	3.580	2.31	250	7	7.45	0.65	806.45	69.83

It is important to emphasize here that, due to the preparation, extraction, and analysis techniques used by laboratories, analyzing a specific contaminant is not more expensive than analyzing all the contaminants in the substance family to which it belongs. Based on the information in Table 24, the following substance families are considered of high interest:

- Chlorobenzene's
- Chlorophenol’s
- Organotins
- PBDE's
- PCB’s
- PFAS
- Phthalates

Due to their short soil half life (< 90 days) the following substances families are considered of significant interest:

- Bisphenol’s
- Nonylphenol’s
- Siloxanes
- VOC's

6.3 Need for standardisation methods

The standardisation of biosolids analysis should be a mandatory step because biosolids consist of a mixture of organic matter and of substances of extremely different nature in quantities that vary greatly and for some of which in infinitesimal quantities. Without standardisation, the data used to decide if this material can be safely spread on land would be unreliable. If the methods for sample preparation, contaminant extraction and purification, as well as their measurement, are not clearly defined, the results obtained are not replicable or comparable. Furthermore, setting regulatory limit values cannot be considered without a standardised analytical protocol. The various stakeholders involved in and impacted by the spreading of biosolids must have complete confidence in the results presented to them. Finally, this allows for the implementation of accreditation procedures and the selection of competent laboratories.

6.4 Recommended approach to improve biosolids quality

6.4.1 Tackling contaminants at source

Biosolids are often stigmatised as a significant source of soil contaminants. In principle, urban wastewater treatment plants are designed to receive urban wastewater,as defined in the recast UWWTD.
Different regulatory tools exist which, if properly implemented, should prevent biosolids from being contaminated by potentially hazardous substances. The main ones are the REACH Regulation and the IED, as well as the recast Urban Wastewater Treatment Directive. The main protective measures stipulated by these texts are included below.

6.4.1.1 REACH

REACH aims to eliminate hazards before they enter a supply chain and implements active risk management through:

> **Authorisation**: For SVHCs, it must be proven that risks are controlled or that no safer alternatives exist. This acts as a powerful lever for substitution, compelling the chemical industry to search for and adopt safer alternatives early in the design process.
>
> **Restriction**: By creating lists of banned or restricted substances, REACH forces supply chains to redesign products (e.g., removing lead from plastics or certain phthalates from consumer goods), thereby "designing out" the contaminant at the source.

It is nevertheless clear that the implementation of the REACH regulation is not without flaws. The environmental crisis linked to PFAS demonstrates that the fate of these contaminants throughout the life cycle of the products containing them has not been sufficiently taken into account. Therefore when placing a substance on the market, it is imperative to assess its risk to end up in the wastewater system and its potential impact on the safety of Biosolids land application.

6.4.1.2 The Industrial Emission Directive (EU) 2024/1785

Some of the contaminants discharged into wastewater systems may originate from industrial discharges. However, these are regulated by the requirements of the IED e (Article 15), which stipulates that the release of polluting substances must not impede the operation of the wastewater treatment plant or the capacity to recover resources from the waste water treatment stream.

If these provisions are correctly applied, industrial connections to the sewer network must not compromise the potential use of biosolids on soils.

6.4.1.3 The recast UWWTD

In accordance with the IED, Article 14 of the recast UWWTD specifies that the discharges of non-domestic wastewater into collecting systems and urban wastewater treatment plants are subject to prior regulations or specific authorisations. The latter shall ensure that the release of polluting substances contained in non-domestic wastewater shall not impede the operation of the urban wastewater treatment plant and do not limit any capacity to recover resources, including the reuse of treated water and the recovery of nutrients or other material from urban wastewater or sludge. Additionally Member States shall ensure that competent authorities take the appropriate measures, including a review of and, where necessary, the revocation of prior regulations and specific authorisations, to identify, prevent and reduce as far as possible the sources of pollution in non-domestic wastewater where sludge arising from urban wastewater treatment is to be used in accordance with Council Directive 86/278/EEC (sludge directive).

As with the IED , these provisions relating to industrial connections to the sewer network should allow for the management of the risks associated with the use of biosolids on soils, if properly implemented.

6.4.2 Analysis frequency

Regular analysis of biosolids is a key parameter to guarantee their quality for end users. It also helps to reassure stakeholders such as agricultural product collectors, residents living near spreading areas, and environmental protection associations.

In most cases, regulations in force in EU Member States specify, in addition to the list of contaminants to be analyzed, the frequencies at which potential contaminants in biosolids should be tested. These frequencies are most often determined based on the treatment capacities of wastewater treatment plants per population equivalent.

Another approach is proposed here, based on contaminant concentrations measured during the first and previous year. If the analysis results are above or below the 50th percentile, the number of samples to be collected and analyzed is adjusted according to the recommendations in Table 25

Table 25 : Recommended analysis frequencies

Year type	Substance type	Substance concentration year n-1	Analysis frequency
First year	Substances with half life < 90 days	-	Twice a year
	Others	-	Quarterly
Following years	Substances with half life < 90 days	Lower than percentile 50	-
		Higher than percentile 50	Twice a year
	Others	Lower than percentile 50	Twice a year
		Higher than percentile 50	Quarterly

6.4.2 Action plan

The results of the analyses can also be used to compare biosolids from a specific source with commonly used standards. Based on this comparison, it is recommended to implement an action plan aiming at continuous improvement, with the following content and targets:

- **Lower than 25th percentile - Background level**: No specific action is recommended, apart from an in depth assessment of any new industrial discharge to the sewer.

- **25th to 50th percentile - Lower than average level**: Inventorying industrial discharges into the wastewater network to identify potential contaminant sources is recommended. Implementing a reduction plan and increasing the analysis frequency of the involved contaminants would be beneficial.

- **50th to 75th percentile - Higher than average level**: It is recommended to take action to detect and identify industrial discharges negatively impacting biosolids quality and assess if pre-treatment options can be considered at source. Regular monitoring of these parameters to track progress is advised.

- **Over 75th percentile - Upper level**: A swift action plan is required to limit contaminant concentrations in biosolids. Detection and identification of industrial discharges which negatively impact biosolids quality and require pre-treatment is urged. Following appropriate action, a new analysis campaign on the relevant parameters should be undertaken.

7 CONCLUSION

The implementation of this analysis campaign within the framework of the Biosolids Observatory provides in-depth knowledge of the composition of these materials resulting from the treatment of urban wastewater. By analyzing 30 representative samples across eight Member States—Italy, Spain, Sweden, Germany, Slovenia, Austria, Greece, and France this approach provides a reliable database for future risk assessments.

The data collected confirm the agronomic value of biosolids and their capacity to supply nutrients such as nitrogen and phosphorus to the plants avoiding the use of mineral fertiliser and the associated CO2 emissions. The presence of secondary nutrients (calcium, magnesium, sulfur) and micronutrients (zinc, copper) further enhances the fertiliser value of biosolids.The crucial role of organic matter, which makes up 50% of the dry matter of biosolids, has also to be taken into account. Regular biosolids land application improves soil structure, water retention, and microbial biodiversity, while also sequestering significant amounts of carbon.

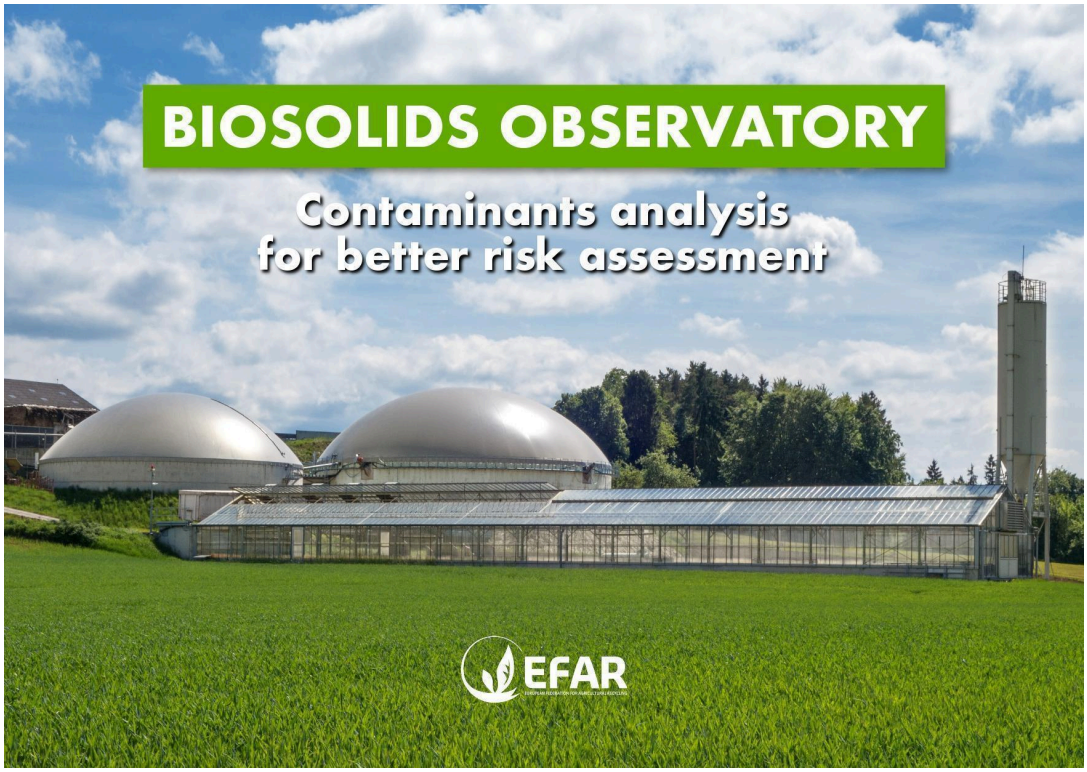
On the other hand the presence of contaminants in biosolids can constitute a significant limitation to their use on soils. The various EU regulations play a crucial role in preventing the dispersion of these substances in the environment and in ensuring that the quality of biosolids is compatible with their use on soils. Concentrations of inorganic contaminants (heavy metals) in samples provided by participants in the biosolids observatory are well below the limit values set by the Sludge Directive. Copper and zinc flows associated with the spreading of biosolids are significantly lower than those associated with certain animal wastes, such as pig slurry. Meanwhile, average cadmium flows do not even represent a quarter of the maximum set by the EU fertiliser regulation.

For organic compounds alkanes and polychlorinated naphthalenes concentrations are below the quantification limits.Compounds like dioxins and furanes that are indirectly released in the sewer network are systematically detected at concentrations above the quantification limits but below the strictest limits set by EU member states. Other compounds that can be directly discharged by households and industrial connections to sewers and for which threshold values have been set by certain EU member states (chlorobenzenes, chlorophenols, nonylphenols, PCBs, phthalates, PFAS) have concentrations that can vary considerably, but which are almost all below the most restrictive limit values set by EU members states.The exceedances recorded concern nonylphenol (3 samples) and toluene (1 sample). Finally for compounds whose concentrations in biosolids are not regulated in any EU country (bisphenols, organotins, PBDEs, siloxanes and pharmaceuticals) the biosolids observatory allows for the determination of reference values (min, max, average, percentiles). The results obtained also demonstrate the effectiveness of regulatory measures aimed at reducing the release of contaminants into the environment.

The analysis of over 7,500 data produced by the Biosolids Observatory also reveals that minimum and average contaminant concentrations are not directly related to the size of wastewater treatment plants. Only very large wastewater treatment plants (> 250,000 population equivalent) have significantly higher contaminant concentrations in their biosolids than small wastewater treatment plants (< 50 000 population equivalent). Furthermore, based on health and environmental criteria, the priority families of substances to be analyzed in biosolids are chlorobenzenes, chlorophenols, organotins, PBDEs, PCBs, PFAS and phthalates. The initial analyses of biosolids for these substances can be used to determine the analysis frequency for subsequent years .Based on the analysis results, it is recommended to implement action plans aimed at gradually reducing contaminant concentrations in biosolids.

ANNEXES

ANNEX A : Biosolids Observatory presentation brochure



What is the purpose of the Biosolids Observatory?

Biosolids land application is currently, and from far, the major route in the EU for sludge management. Indeed, this solution has many advantages such as nutrient recovery, carbon sequestration and the improvement of soil production capacity. Nevertheless, biosolids may contain contaminants from domestic, commercial and industrial discharges into the sewer system. Due to legitimate concerns regarding the protection of human health and the environment it is essential to have a recent overview of the biosolids quality at a European level. The purpose of the Biosolids Observatory is to collect, package, transport and analyse representative samples from a significant number of sewage treatment plants located in different EU countries. The resulting data can be used to feed risk assessment studies but also to better understand the potential impacts of the upcoming revision of the regulatory framework, notably the 86/278/EEC Directive.



EFAR

Who are the participants in the Biosolids Observatory?

Participants in the Biosolids Observatory can be local authorities responsible of wastewater treatment, operators of wastewater treatment plants or companies specialised in the treatment and land application of biosolids.



Is it possible to analyse organic materials other than biosolids?

It is possible to analyse organic materials used as soil amendment or to fertilise crops other than biosolids. This will provide a better picture of the origin and relative share of some of the contaminant flows spread on land.

Why should you join the Biosolids Observatory process?



Public and private entities involved in biosolids land application are committed to supply to the farmers a product that can be safely used as a fertiliser or soil improver. This liability requires a detailed knowledge of the biosolids composition. Testing your biosolids for a large scale of compounds will allow you to identify if their concentrations are in the standard range or at a high level. It will therefore provide important information on the need for further source control measures to reduce potentially harmful or unwanted contaminants in your biosolids.

How can you join the Biosolids Observatory?



You can easily join the Biosolids Observatory by sending an email to:
biosolidsobsevatory@gmail.com

EFAR

What is the role of the Biosolids Observatory Steering Committee ?

The Steering Committee is including participants of professional associations (at national or EU level) that have decided to promote the Biosolids Observatory project to its members. The Steering Committee will also welcome experts in the field of biosolids land application and associated risk assessment.

The Steering Committee is in charge of the validation of:

- The frequency of the testing campaigns.
- The scope of the analyses to be carried out.
- The approval of the information brochure which will explain the purpose and operating conditions of the Observatory.
- The choice of a partnering laboratory.
- The sampling procedure.
- The results presentation report and the conditions for its publication.



EFAR

What was the selection criteria of the labs in charge of biosolids analysis?

The choice of partnering laboratories is of the utmost importance for the credibility of the results obtained. AUREA and EUROLAB have been selected due to the extent of the guarantees they have provided in terms of quality assurance, accreditation, experience and reputation in terms of biosolids testing. AUREA and EUROLAB are committed to supply the following information to the Steering Committee:



- Scope of accreditation
- Methodology applied for extraction and measurement of each compound
- Detection and quantification limits
- Sample custody and traceability
- Supply of containers and logistics means of collection
- Procedures for exchanging information with the biosolids producer

EFAR

How should you sample your biosolids?

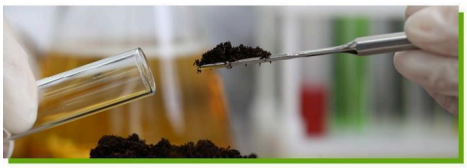
Sampling conditions are key to ensure that the biosolids samples are representative and to make sure that the samples are not tampered or contaminated during the sampling process or during transport to the laboratory.
Detailed sampling procedure will be supplied to the participants in order to provide all the necessary information regarding:

- Sampling point
- Sampling equipment
- Sample constitution
- Sample identification
- Sample storage and shipment



How many times would you have to sample your biosolids?

The first sampling campaign will occur during the first half of 2025. At that time you will have the possibility to send several samples if you wish to test the variability of your biosolids composition. Subsequently, the Steering Committee will ask you to participate in the following testing campaigns at a frequency that will be considered as most appropriate. You will be free to participate or not.



At what stage of the biosolids production process would you have to take to sample?

The biosolids samples will have to be taken at the final stage of their production process which means at the point where they would be ready for delivery to the final users.

EFAR

How should you send your biosolids to the lab?

The means necessary for identification, packaging and delivery of samples will be provided directly by the laboratory.

What parameters will be tested?

Different options are proposed in order to determine the quantities of beneficial elements and contaminants potentially present in the biosolids. The list of contaminants has been validated by the Steering Committee on the basis of the risks they may represent for health and/or the environment.



Should you provide specific information on the wastewater treatment plant where the biosolids are produced?



In order to interpret the results obtained you will have to fill in a dedicated questionnaire detailing among others the following information:

- Wastewater treatment plant location and size in population equivalent
- Identified industries connected to the sewer
- Wastewater treatment process
- Biosolids production process
- Tonnage of biosolids produced per year

EFAR

What analysis methods will be applied?

Standardised analysis methods will be used where they exist. If this is not the case, the partner laboratory will have to justify the performance of the method that will be used.



What will be the costs of biosolids testing and who will support them?

The costs of analysis will be charged directly to the entity who has sent the biosolids sample to the lab. Following the review of scientific publications on the impact of biosolids land application three analysis packages have been established. The basic package is corresponding to the contaminants that are the most relevant and which must be systematically analysed. Additional packages 1 and 2 cover contaminants of interest without their analysis being considered essential. Depending on the package that will be chosen, the costs will range from 1385€ to2215€ per sample.

Can your participation in the Biosolids Observatory remain anonymous?

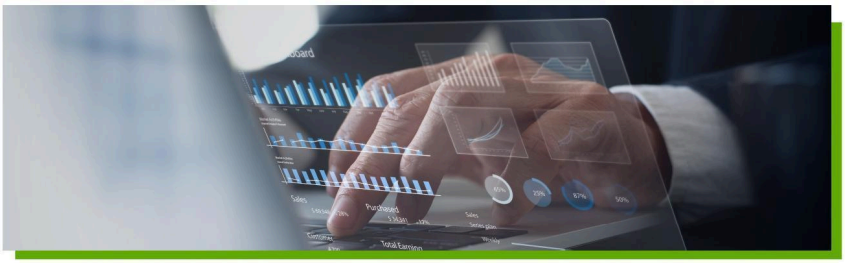
If requested, your participation in the Biosolids Observatory will remain anonymous for the following information: location of the wastewater treatment plant (except for the country), exact size of the wastewater treatment plant (results will be presented per range of size, e.g. from 50 000 to 100 000 p.e.).

EFAR

How will the results of the biosolids analysis be used?

The biosolids analysis results will be included in a report which will detail:

- The purpose of the Biosolids Observatory
- The members of the Steering Committee
- Justification of the selection of parameters to be tested
- Analysis methods
- Results obtained
- Comparison of the results with other testing campaigns
- Relative contribution of Biosolids in soil contamination



What is the timeline for the implementation of the Biosolids Observatory?

Timeline for the implementation of the Biosolids Observatory as follows:



EFAR

BIOSOLIDS OBSERVATORY

Improving knowledge for safe recovery

Contact : biosolidsobsevatory@gmail.com

EFAR

ANNEX B : Biosolids production site questionnaire

BIOSOLIDS OBSERVATORY PROJECT
WWTP QUESTIONNAIRE

1 PARTICIPANT INFORMATION

- Legal name:
- Please specify if public or private entity:
- Registration number or EBR extract:
- VAT number:
- Legal address:

2 CONTACT OF THE STAFF IN CHARGE OF THE PROJECT FOLLOW UP

- First name:
- Surname:
- Phone number:
- Email:

3 WWTP INFORMATION

(If you operate a Biosolids treatment site which is not located on the WWTP go to section 8; if you want to send an organic material which is not a Biosolids go to section 9).

- Operator (if different from the participant):
- Site address:

- On site contact for shipment kit sending and sample collection:
    - First name
    - Surname:
    - Phone number:
    - Email:

4 WWTP CHARACTERISTICS

- Population served in pe (population equivalent):
- Primary treatment (Yes or No if YES please specify the type of treatment):
- Secondary treatment (Yes or No if YES please specify the type of treatment):
- Tertiary treatment (Yes or No if YES please specify the type of treatment):

- Quaternary treatment (Yes or No if YES please specify the type of treatment):
- Polymer or chemical reagents addition (Yes or No please specify type and addition ratio):

5 CONNECTION TO THE SEWER

- Percentage of the inlet wastewater flow in BOD or COD coming from industries: …%
- Are you aware of any specific industrial discharge to the sewer (Yes or No if YES please specify the type of activity using the SIC CODE and the part of the inlet wastewater flow in BOD or COD if known.

6 SLUDGE PRODUCTION

- Anaerobic digestion (Yes or No) :
- Sludge yearly production wet weight: …… tonnes
- Average Dry matter in %: ….
- Final destination (land application, land reclamation, landfilling, incineration, ……): ……..

7 SLUDGE POST TREATMENT

- None
- Liming (Yes or No if Yes please specify liming rate on dry matter):
- Composting (Yes or No if Yes please specify the bulking agent used and its incorporation ratio in wet weight):
- Thermal drying:
- Solar drying:
- Other: please specify

8 TREATMENT SITE INFORMATION (if you have filled sections 3 to 7 go directly to section 10).

- Operator (if different from the participant):
- Site address:

- On site contact for shipment kit sending and sample collection:
    - First name
    - Surname:
    - Phone number:
    - Email:

- Description of the treatment process (please specify if any additional material(s) are added to the Biosolids and the mixing ratio on dry weight and if you are mixing Biosolids from different WWTP)
  ……
  ……
  ……
  ……
  ……
  ……
  ……

9 SENDING OF A ORGANIC MATERIAL WHICH IS NOT A BIOSOLIDS

- Description of the origin and of the production process (if any) of the organic material sent for analysis.
  ……
  ……
  ……
  ……
  ……
  ……

10 DESCRIPTION OF THE SAMPLING POINT

Please provide hereafter a short description of the place where the sample will be taken:

……
……
……
……

11 REQUEST FOR ANONYMITY

I want my participation to remain anonymous regarding the name of my legal entity and the location of the wastewater treatment plant except for the country of establishment (Yes or No):

I agree to share my biosolids analysis results with the Biosolids Observatory Steering Committee.

Done in …… by the ../../2025

Signature of the authorised person and legal entity stamp.

ANNEX C : Sampling procedure

BIOSOLIDS OBSERVATORY PROJECT SAMPLING PROCEDURE

Thank you for joining the Biosolids Observatory Project; you will find hereafter the information you need to order your analysis, sample and ship your biosolids to the lab and provide the required information for the treatment of your data. In case of doubt don't hesitate to drop an email to biosolidsobservatory@gmail.com we will be back to you rapidly.
Please keep in mind that proper sampling is crucial regarding the quality of the results. Indeed some contaminant concentrations and the associated quantification limits are of some µg per kg which represents a tenth of a pinch of salt in the 28 tonnes load of an articulated lorry.

Ordering

You have to choose your analysis package amongst the following options:

basic package for a cost of 1250 € VAT excluded
additional package 1 for a cost of 185 € VAT excluded
additional package 2 for a cost of 645 € VAT excluded

Extra fee of 135 € VAT excluded will be charged for the shipment costs.

Scope of analysis per package is detailed here.

You have to fill in your order on your usual order form and send it by email to biosolids.campaign@aurea.eu. Please mention as supplier AUREA: 1, rue Samuel Champlain, 17074 La Rochelle FRANCE. In the email you have to specify your contact details (email, address, phone number), your entity registration number, and VAT number. You will also have to indicate the address where the biosolids shipment kit has to be sent. Please notice that if you wish to send several samples you will need to complete an order form for each one of them.

AUREA will then create your customer account on its CRM and will provide you with the necessary information to login on AUREA online (attention : the interface is in French only). AUREA will integrate your order into AUREA online.

Within 10 working days, the AUREA logistic department will automatically send the shipment kit to the contact address : a cooler containing six glass jars of one litre capacity and ice packs. This procedure will be included in the cooler, along with a transparent pouch for the shipping voucher (see 4.3).

The WWTP questionnaire that you have received at the same time as this sampling procedure has to be filled in and sent back to biosolidsobservatory@gmail.com.
Sampling planning

Sampling has to be planned after reception of the sampling equipment as described in §3 and of the shipment kit that will be sent by AUREA. You will have to send an email to biosolids.campaign@aurea.eu to confirm your order, recalling its initial reference and indicating the sample reference, and the planned sampling date. Then you will receive from AUREA :
2.a : an email containing a PDF pre-filled analysis request form, that you shall print on a blank sheet. This printed sheet has to be placed in the cooler when the samples will be sent to the lab.
2.b : a logistics memo explaining how to load and pack the cooler for shipment to the laboratory.

You will define the sampling point taking into account that it must allow the easy sampling of the biosolids or of the biosolids derived product (e.g. compost) under the form that will be delivered to the final user. Two days before sampling it is necessary to make sure that the wastewater treatment plant or the biosolids treatment plant is operating under normal conditions. When planning the sampling campaign keep in mind that it is recommended to send the biosolids sample to the lab by the beginning of the week to avoid the possible slowdown in delivery times during the weekend.

Sampling equipment

The sampling equipment encompasses a trowel or transplanter and a minimum 10 litres capacity bucket with lid both made of stainless steel.
The sampling equipment must be cleaned and rinsed before the sampling session and must be free from rinsing water. The equipment must not have any influence on the sample (neither risk of contamination nor dilution).
The staff in charge of the biosolids sampling must wear nitrile gloves and a mask.

Sampling methodology

The sample taken and analysed must be representative of the biosolids production and should be not less than 15% dry matter to ensure that laboratories have enough material to carry out all analyses. Keep in mind that you have to send a volume of 6 litres of biosolids to the lab; therefore for the minimum of 25 elementary samples (see § 4.1 and 4.2) the volume of each one of them has to be of 25 cl.

Sampling can be carried out:
either over during the biosolids production (continuous sampling),
or on a batch already produced (e.g. compost stockpile).
Batch sampling will be preferred whenever possible.

4.1 Continuous sampling

To obtain a representative sample of the biosolids production a minimum of 25 elementary samples must be taken evenly distributed during a predetermined period of not less than 5 days.
The size of each elementary sample must be identical. These elementary samples are placed and mixed in the 10 litre stainless steel bucket; the latter has to be kept near the sampling point with the lid on for the duration of the sampling campaign. A warning sign has to be placed on the lid to specify that the bucket is strictly and solely used for biosolids sampling and that the lid must be kept on.

4.2 Batch sampling

To obtain a representative sample of the batch to be analysed a minimum of 25 elementary samples must be taken at different points and different depths of the biosolids stockpile.
The collection of elementary samples must be evenly distributed throughout the batch at a minimum depth of 20 cm of the surface and in areas where water accumulation has not occurred.
The size of each elementary sample must be identical. These elementary samples are placed and progressively thoroughly mixed in the 10 litre stainless steel bucket.

4.3 Lab sample preparation and packaging

By the middle of the sampling campaign you will have to send an email to biosolids.campaign@aurea.eu to request a pick-up, specifying the desired pick-up date. AUREA will return an email to the participant with a shipping voucher that you shall print before the pick-up.

The elementary samples shall be mixed thoroughly with the stainless steel transplanter or trowel in the 10 litres bucket before placing a quantity of 6 litres, collected at different depths of the bucket, in the wide-mouth glass jars provided by AUREA.

Sample collection and shipment

You will have to fill the glass jars the day before the scheduled collection date to limit the alteration and modification of the sample composition and to reduce the risks of gas accumulation in the glass jars. According to the logistics memo (2.b) the latter will be placed in the cooler accompanied with a previously frozen ice pack, with a copy of the purchase order and with the AUREA pre-filled analysis request form (see 2.a) . You will then have to close the cooler and seal it with strong adhesive tape, and finally stick the transparent pouch (see 1) containing the shipping voucher (4.3) on its top.

The shipping company will collect the cooler on site and will transport it to AUREA’s laboratory located in La Rochelle (France). After reception and registration of your sample you will receive an acknowledgment of receipt by email.

If the parcel cannot be ready on the date initially communicated, you must inform AUREA (biosolids.campaign@aurea.eu) as soon as possible, indicating the new desired shipping date.

Results supply and invoicing

After completion of the analyses AUREA will send you analysis reports under pdf and Excel format. If you wish you will have the possibility to connect to AUREA online to get partial analysis results.
Analysis reports will be followed by the invoice that has to be paid forthwith and no later than 30 days after the receipt of the invoice, by transfer on the following bank account : FR76 3000 3033 9200 0203 5067 851 (BIC : SOGEFRPP).

ANNEX D : Analysis methods

Performing laboratory	Subsection and chemical determination	Measurement method & reference [1]	Analytical method description	Conditions for the validation of the results
AUREA Agrosciences – La Rochelle -France	Nutrients : **Dry matter***	(a) Internal method from NF EN ISO 12880	The dry matter is determined with about 50 g of raw sample. The sample is dried at 105 °C ± 5 °C during at least 20 h.	The temperature of the oven is controlled over the entire drying time. AUREA takes part in several annual inter-laboratory tests (BIPEA and AGLAE) to evaluate and verify the method performances continuously.
AUREA Agrosciences – La Rochelle -France	Nutrients : **Nitrogen NTK***	(b) NF EN 13342	The method consists in a Kjeldahl digestion with a strong acid in the presence of a catalyst (selenium) and at a temperature up to 390 °C. 0,25 g to 4 g of homogenized wet sample is mineralized according to the dry matter. A distillation is performed in an alkaline medium and the released ammonia form is stabilized with an excess of boric acid solution. The ammonia form is quantified by titration with chlorohydric acid.	Initial validation of the method was carried out on the basis of the NF T90-210 standard, using representative matrices spiked at different levels of the application range. Each analysis batch includes quality controls : procedural blank, external reference materials and synthetic materials. AUREA takes part in several annual inter-laboratory tests (BIPEA and AGLAE) to evaluate and verify the method performances continuously.
AUREA Agrosciences – La Rochelle -France	Nutrients : **Hydrogen potential (pH)***	(c) NF EN 15933 (Potentiometry)	Hydrogen potential (pH) is measured in an aqueous suspension of a test sample. 5 volumes of water and one volume of homogenized raw sample are agitated during 1	Initial validation was carried out using inter-laboratory test samples. Each analysis batch includes quality controls (reference standards).

[1] The reference is a letter from (a) to (p) that is used in the analytical performance table (DL, QL).

			hour at 20 °C. The pH measurement is performed with an automatic pH meter.	AUREA takes part in several annual inter-laboratory tests (BIPEA and AGLAE) to evaluate and verify the method performances continuously.
AUREA Agrosciences – La Rochelle -France	Nutrients : **Organic matter***	(d) Internal method from NF EN 15935 (Calcination)	The content of organic matter is determined on the dried and ground sample by calcination at 550 ± 25 °C for 2 hours.	Initial validation was carried out using inter-laboratory test samples. Each analysis batch includes a quality control (external reference material). AUREA takes part in several annual inter-laboratory tests (BIPEA and AGLAE) to evaluate and verify the method performances continuously.
AUREA Agrosciences – La Rochelle -France	Nutrients : **P2O5*; K2O*; CaO*; MgO*, SO3*** Inorganic contaminants : **Ag, As*, Cd*, Cu*, Ni*, Pb*, Zn***	(e) NF EN ISO 11885 & NF EN 13346 (e) NF EN ISO 11885 & NF EN 13346	A representative quantity of the dried and ground sample is digested with aqua regia (hydrochloric acid/nitric acid proportion = 3/1 v/v). The diluted digestate is analyzed by inductively coupled plasma optical emission spectroscopy. The amount of each element is quantified using a calibration of increasing concentration standards.	Initial validation of the method was carried out according to NF T90-210 standard, using representative matrices spiked at different levels of the application range. Each analysis batch includes quality controls (procedural blank, external reference materials and reference standards). Internal standard dilution is used to assess matrix effects. AUREA takes part in several annual inter-laboratory tests (BIPEA and AGLAE) to evaluate and verify the method performances continuously.

AUREA Agrosciences – La Rochelle -France	Inorganic contaminants : **Hg***	(f) NF EN 13346 & NF ISO 16772	A representative quantity of the dried and ground sample is digested with aqua regia (hydrochloric acid/nitric acid proportion = 3/1 v/v). The diluted digestate is analyzed by atomic fluorescence spectroscopy. The amount of mercury is quantified using a calibration.	Initial validation of the method was carried out according to NF T90-210 standard, using representative matrices spiked at different levels of the application range. Each analysis batch includes quality controls (procedural blank, external reference materials and reference standards). AUREA takes part in several annual inter-laboratory tests (BIPEA and AGLAE) to evaluate and verify the method performances continuously.
CARSO-LSEHL – Vénissieux – France as a subcontractor of AUREA Agrosciences	PFAS (perfluoro sulfonic acids) : 40 compounds **PFBA - CAS NUM : 375-22-4, PFPA - CAS NUM : 2706-90-3, PFHXA - CAS NUM : 307-24-4, PFHpA - CAS NUM : 375-85-9, PFOA - CAS NUM : 335-67-1, PFNA - CAS NUM : 375-95-1, PFDA - CAS NUM : 335-76-2, PFUnA - CAS NUM : 2058-94-8,**	(g) Internal method (M_ST286)	A representative quantity of lyophilized and ground sample is extracted with acidified water and then acetonitrile according to the QuEChERS method. After purification and addition of a solvent (methanol), the extract is analyzed by HPLC/MS/MS.	Initial validation of the method was carried out using representative matrices spiked at two levels of the application range and by monitoring of control charts. Each analysis batch includes quality controls (procedural blank and biosolid spiked with a mixture of PFAS). CARSO-LSEHL is registered to take part in the BIPEA interlaboratory tests in 2026 on sediments, using the same method.

	PFDODA - CAS NUM : 307-55-1, **PFTRDA - CAS NUM : 72629-94-8,** **PFBS - CAS NUM : 375-73-5,** **PFPS - CAS NUM : 2706-91-4,** **PFHXS - CAS NUM : 355-46-4,** **PFHpS - CAS NUM : 375-92-8,** **PFOS - CAS NUM : 1763-23-1,** **PFNS - CAS NUM : 68259-12-1,** **PFDS - CAS NUM : 335-77-3,** **PFUNDS - CAS NUM : 749786-16-1,** **PFDODS - CAS NUM : 79780-39-5,** **PFTRDS - CAS NUM : 791563-89-8,** **PFTEDA - CAS NUM : 376-06-7,** **PFOSA - CAS NUM : 754-91-6,** **FTSA6:2 - CAS NUM : 27619-97-2,** **FTS4:2 - CAS NUM : 757124-72-4,** **FTSA8:2 - CAS NUM : 39108-34-4,**			

	FTCA5:3 - CAS NUM : 914637-49-3, FTCA6:2 - CAS NUM : 53826-12-3, HFPO-DA - CAS NUM : 13252-13-6, ADONA - CAS NUM : 919005-14-4, 11CLPF3OUDS - CAS NUM : 763051-92-9, 5H-PFPA - CAS NUM : 376-72-7, 9CLPF3ONS - CAS NUM : 73606-19-6, FTCA3:3 - CAS NUM : 356-02-5, FTCA7:3 - CAS NUM : 812-70-4, FTSA10:2 - CAS NUM : 108026-35-3, MEFOSA - CAS NUM : 31506-32-8, NFDHA - CAS NUM : 151772-58-6, PFEESA - CAS NUM : 113507-82-7, PFMBA - CAS NUM : 863090-89-5, PFMPA - CAS NUM : 377-73-1			
CARSO-LSEHL – Vénissieux – France as a subcontractor of AUREA Agrosciences	PBDE (polybrominated diphenyl ethers) : 18 PBDE **BDE 17*, 28*, 47*, 66*, 71*, 77*, 85*, 99*, 100*, 138*, 153*, 154*, 181*, 183*, 190*, 203*, 205*, 209***	(h) Internal method (MET-081), based on the standard EPA 1614	A liquid/solid extraction by toluene is carried out on a sample previously lyophilized and spiked with a solution of 9 ^{13}C-labelled PBDEs standards (isotopic dilution). After purification on multi-layer columns and addition of the	Initial validation of the method was carried out by using representative matrices spiked at different levels of the application range. Each analysis batch includes quality controls (procedural blank, procedural control

			internal recovery standards, PBDEs analysis is performed by HRGC/HRMS with a resolution at 7000.	sample and internal recovery standards yields). CARSO-LSEHL takes part in several annual inter-laboratory tests (including InterCIND) to evaluate and verify the method performances continuously.
CARSO-LSEHL – Vénissieux – France as a subcontractor of AUREA Agrosciences	NP+NPE (nonylphenols and octylphenols + ethoxylates) : **nonylphenol,** **NP1OE,** **NP2OE,** **octylphenol,** **OP1OE** **OP2OE**	(i) Internal method (M_ST281)	A representative quantity of lyophilized and ground sample is extracted with acidified water and then acetonitrile according to the QuEChERS method. After purification and addition of a solvent (methanol diluted), the extract is analyzed by HPLC/FLD.	Initial validation of the method was carried out using representative matrices spiked at two levels of the application range and by monitoring of control charts. Each analysis batch includes quality controls (procedural blank, material blank and internal recovery standards yields).
CARSO-LSEHL – Vénissieux – France as a subcontractor of AUREA Agrosciences	Organotins : **monobutyltin,** **dibutyltin,** **monophenyltin,** **diphenyltin,** **triphenyltin,** **tetraphenyltin,** **monooctyltin,** **dioctyltin,** **trioctyltin,** **tricyclohexyltin,** **tributyltin,** **tetrabutyltin**	(j) Internal method (M-ST057)	A representative quantity of lyophilized and ground sample is extracted by acetic acid digestion, which is followed by liquid/liquid hexane extraction of the OSNs in solution in the acid with derivatisation (alkylation). The derived molecules are analyzed by GC/MS/MS.	Initial validation of the method was carried out using representative matrices. Each analysis batch includes quality controls (procedural blank, procedural control sample and internal recovery standards yields). CARSO-LSEHL takes part in annual inter-laboratory tests (BIPEA 38b) to evaluate and verify the method performances continuously.
CARSO-LSEHL – Vénissieux – France	Miscellaneous: chlorobenzens and chlorophenols			

as a subcontractor of AUREA Agrosciences	Chlorobenzens : **Monochlorobenzene, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 1,2,3-trichlorobenzene, 1,2,4-trichlorobenzene, 1,3,5-trichlorobenzene,**	(k) NF ISO 22155	Test sample is taken from a lyophilized representative sample and extracted using methanol. After stirring, an aliquot of the methanolic extract is added to a headspace flask with a defined amount of water and then hermetically sealed. The vial is thermostated at 80°C to achieve a stable distribution between the gas and liquid phases. A few microlitres of the gas phase are automatically injected by an injection valve. The gas extract is analyzed by headspace GC/MS chromatography.	Initial validation of the method was carried out using representative matrices. Injection and extraction yields are verified by adding different internal standards. At each batch of preparation, a methanol control prepared in the same way as the samples but without solid sample is carried out. The performance of the analyzer is checked daily by injecting several quality controls at different concentrations. CARSO-LSEHL takes part in several inter-laboratory tests (LGC and AGLAE) on the accredited matrices sediment and soil.
	Chlorobenzens : **hexachlorobenzene HCB, pentachlorobenzene,** Chlorophenols (20 compounds) : **2,3,4,5-tetrachlorophenol, 2,3,4,6-tetrachlorophenol, 2,3,4-trichlorophenol,**	(l) Internal method	A representative quantity of lyophilized and ground sample is extracted with water and acetonitrile according to the QuEChERS method. After concentration and extraction with CH_2Cl_2, the extract is analyzed by GC/MS/MS.	Initial validation of the method was carried out using representative matrices. Each analysis batch includes quality controls (procedural blank, procedural control sample and internal recovery standards yields). CARSO-LSEHL takes part in annual inter-laboratory tests (BIPEA 38b) for hexachlorobenzene HCB and pentachlorobenzene to evaluate

	2,3,5-trichlorophenol, 2,3,6-trichlorophenol, 2,3-dichlorophenol, 2,4,5-trichlorophenol, 2,4-dichlorophenol, 2,5-dichlorophenol, 2,6-dichlorophenol, 2-chloro, 5-methylphenol, 2-chloro, 6-methylphenol, 2-chlorophenol, 3,4-dichlorophenol, 3,5-dichlorophenol, 3-chlorophenol, 4-chloro, 2-methyl, phenol, 4-chloro, 3-methyl, phenol, 4-chlorophenol, pentachlorophenol			and verify the method performances continuously.
AUREA Agrosciences – La Rochelle -France	Physical contaminants : **Plastics, glass, metals and stones from 2 to 5 mm and > 5 mm**	(m) Internal method from NF U44-164	Bleach destroys non-synthetic organic matter, with the exception of woody materials which are not to be considered as inerts. The remaining inerts are sorted by densimetry using water then a saturated calcium chloride solution with a density of 1,35. Destruction on non-synthetic organic matter with bleach facilitates manual sorting of the inerts.	Initial validation of the method was carried out by comparing sample results with those obtained by an accredited laboratory. AUREA takes part in several annual inter-laboratory tests (BIPEA and AGLAE) to evaluate and verify the method performances continuously.

CARSO-LSEHL – Vénissieux – France as a subcontractor of AUREA Agrosciences	Miscellaneous: BTEX + VOC (excluding chlorobenzenes) BTEX : **benzene,** **ethylbenzene,** **toluene,** **xylene o,** **xylene m + p** + 40 VOC solvents : **1,2,3-trimethylbenzene,** **1,2,4-trimethylbenzene,** **1,3,5-trimethylbenzene,** **4-isopropyl toluene,** **isopropylbenzene,** **MTBE (methyl tertiobutyl ether),** **n-butylbenzene,** **styrene,** **tert butylbenzene,** **1,1,1,2-tetrachloroethane,** **1,1,1-trichloroethane,** **1,1,2-trichloroethane,** **1,1,2-trichlorotrifluoroethane,** **1,1-dichloroethane,** **1,1-dichloroethylene,** **1,2-dibromoethane,** **1,2-dichloroethane,** **1,2-dichloropropane,** **2,3-dichloropropylene,** **3-chloropropene,**	(n) Internal method (M_ST095), based on the standard NF EN ISO 22155	Samples are taken from an untreated raw sample with the least possible disturbance to the sample and extracted using methanol. After stirring, an aliquot of the methanolic extract is added to a headspace flask with a defined amount of water and then hermetically sealed. The vial is thermostated at 80°C to achieve a stable distribution between the gas and liquid phases. A few microlitres of the gas phase are automatically injected by an injection valve. The gas extract is analyzed by headspace GC/MS chromatography.	Initial validation of the method was carried out using representative matrices. Injection and extraction yields are verified by adding different internal standards. At each batch of preparation, a methanol control prepared in the same way as the samples but without solid sample is carried out. The performance of the analyzer is checked daily by injecting several quality controls at different concentrations. CARSO-LSEHL takes part in several inter-laboratory tests (LGC and AGLAE) on the accredited matrices sediment and soil.

	bromochloromethane, bromoform, Cis 1,2-dichloroethylene, Cis 1,3-dichloropropylene, carbon tetrachloride, chloroform, dibromochloromethane, dichlorobromomethane, dichloromethane, hexachloroethane, Trans 1,2-dichloroethylene, Trans 1,3-dichloropropylene, trichlorofluoromethane, tetrachlorethylene, trichlorethylene, hexachlorobutadiene, vinyl chloride, 2-chlorotoluene, 3-chlorotoluene, 4-chlorotoluene			
CARSO-LSEHL – Vénissieux – France as a subcontractor of AUREA Agrosciences	Phtalates: **butyl benzyl phtalate, di n-butyl phtalate, di n-octyl phthalate, DEHP, diethyl phtalate, di isobutyl phtalate, dimethyl phtalate**	(o) Internal method	A representative quantity of lyophilized and ground sample is extracted by solid-liquid accelerated solvent extraction (ASE) with a mixture of acetone and hexane (20/80). After concentration and extraction with CH2Cl2, the extract is analyzed by GC/MS/MS.	Initial validation of the method was carried out using representative matrices. Each analysis batch includes quality controls (procedural blank, procedural control sample and internal recovery standards yields).

				There are no inter-laboratory tests for phthalates in the biosolid matrix.
CARSO-LSEHL – Vénissieux – France as a subcontractor of AUREA Agrosciences	Dioxins + furans: **PCDD* and PCDF*, TEQ (OMS 2005)**	(p) Internal method (MET-003), based on the standards EPA 1613 and NF EN 16190	A liquid/solid extraction by toluene is carried out on a sample previously lyophilized and spiked using a solution of 16 ^{13}C-labelled PCDD/F standards (isotopic dilution). After purification on multi-layer columns and addition of the internal recovery standards, PCDD/F analysis is performed by HRGC/HRMS at 10000 resolution.	Initial validation of the method was carried out by using representative matrices spiked at different levels of the application range. Each analysis batch includes quality controls (procedural blank, procedural control sample and internal recovery standards yields). CARSO-LSEHL takes part in several annual inter-laboratory tests (including InterCIND) to evaluate and verify the method performances continuously.

*COFRAC-Accredited analysis

2,2',3,5'-tetrachlorobiphenyl	PCB 44	41464-39-5	C12H6Cl4	5 µg/kg
2,2',4,4',5,5'-hexachlorobiphenyl	PCB 153	35065-27-1	C12H4Cl	5 µg/kg
2,2',4,4',5-pentachlorobiphenyl	PCB 99	38380-01-7	C12H5Cl5	5 µg/kg
2,2',4,5,5'-pentachlorobiphenyl	PCB 101	37680-73-2	C12H5Cl5	5 µg/kg
2,2',5,5'-tetrachlorobiphenyl	PCB 52	35693-99-3	C12H6Cl4	5 µg/kg
2,2',5-trichlorobiphenyl	PCB 18	37680-65-2	C12H7Cl3	5 µg/kg
2,3,3',4,4',5,5'-heptachlorobiphenyl	PCB 189	39635-31-9	C12H3Cl7	5 µg/kg
2,3,3',4,4',5-hexachlorobiphenyl	PCB 156	38380-04-4	C12H4Cl6	5 µg/kg
2,3,3',4,4',5'-hexachlorobiphenyl	PCB 157	69782-90-7	C12H4Cl6	5 µg/kg
2,3,3',4,4'-pentachlorobiphenyl	PCB 105	32598-14-4	C12H5Cl5	5 µg/kg
2,3,3',4',6-pentachlorobiphenyl	PCB 110	38380-03-9	C12H5Cl5	5 µg/kg
2,3',4,4',5,5'-hexachlorobiphenyl	PCB 167	52663-72-6	C12H4Cl6	5 µg/kg
2,3,4,4',5-pentachlorobiphenyl	PCB 114	74472-37-0	C12H5Cl5	5 µg/kg
2,3',4,4',5-pentachlorobiphenyl	PCB 118	31508-00-6	C12H5Cl5	5 µg/kg
2,3',4,4',5'-pentachlorobiphenyl	PCB 123	65510-44-3	C12H5Cl5	5 µg/kg
2,4,4'-trichlorobiphenyl	PCB 28	7012-37-5	C12H7Cl3	5 µg/kg
2,4',5-trichlorobiphenyl	PCB 31	16606-02-3	C12H7Cl3	5 µg/kg
3,3',4,4',5,5'-hexachlorobiphenyl	PCB 169	32774-16-6	C12H4Cl6	5 µg/kg
3,3',4,4',5-pentachlorobiphenyl	PCB 126	57465-28-8	C12H5Cl5	5 µg/kg
3,3',4,4'-tetrachlorobiphenyl	PCB 77	32598-13-3	C12H6Cl4	5 µg/kg
3,4,4',5-tetrachlorobiphenyl	PCB 81	70362-50-4	C12H6Cl4	5 µg/kg

Table 2.

Chloroparaffins			
Name	**CAS**	**Formula**	**LOQ**
SCCP C10-C13	98-95-3	C10–C13H(Cl)n	500 µg/kg
LCCP C14-C30	121-73-3	C14–C30H(Cl)n	500 µg/kg

Table 3.

3. Equipment

3.1 Acetone
3.2 n-Hexane
3.3 C18 dSPE
3.4 15 ml calibrated glass test tubes
3.5 Soxhlet extractor
3.6 Cellulose thimbles for extraction
3.7 Sodium sulfate
3.8 General laboratory equipment
3.9 TRACE 1300 GC coupled with TSQ Evo 8000 mass spectrometer

4. Extraction Solution

4.1 Hexane-Acetone Solution 50:50 (v/v).

In a 2L volumetric flask, add 500 ml of acetone using a graduated cylinder. Bring to volume with n-hexane. Add 100 µl of the solution from section 6.1.9. Shake vigorously and transfer to a brown glass bottle.

5. Sample Preparation

5.1 Blank Preparation

Along with the samples being analyzed, perform a process blank. This is crucial, as some analytes may be contaminated in the blanks. Use a Soxhlet position for the Process Blank (without the thimble, but with only the extraction solvent, following the full extraction procedure).

5.2 Extraction for Sample Preparation

Weigh 10g ± 0.01 of the dried sample into a cellulose thimble and place it in the Soxhlet extractor. If the sample is wet, add a spatula of sodium sulfate. Record the exact weight (10g), the final volume (5mL), and the dilution factor (TQ) on the work sheet. Add 100 mL of the 50:50 hexane-acetone solution (approximately up to the VELP label level) containing the surrogates (5.1) into the Soxhlet beaker.
Extract the sample using these parameters: Injection 140°C - 60min / Washing 60min / total duration approx. 2 hours. Concentrate the sample using Soxhlet with these parameters: Recover 110°C - 10min; until about 10mL is achieved, keeping the thimble raised and the red valve closed.
Transfer the extract into a graduated glass test tube and concentrate to a final volume of 5 mL using a nitrogen flow. Add 200 mg of C18 dSPE (approximately 5 spatulas), shake vigorously, and centrifuge the samples for 5 minutes at 2000 rpm. Transfer 1 mL of the extract into 2 mL glass vials and seal immediately with GC-compatible caps.

5.2.1 Spiked Sample and LCS (50 µg/L in vial)

Weigh 10g ± 0.01 of the dried sample into a cellulose thimble. Using a manual pipette, add 25 µL of the 5 mg/L hexane solution (6.1.1) and 10 µL of the 1 mg/L surrogate solution. Proceed as described above. Repeat the procedure using a contaminant-free sand matrix, referred to as LCS.

6. GC-MS/MS Analysis

Instrument: GC TRACE 1300 coupuled with TSQ Evo 8000 Mass Spectrometer.
Column: XLB 30m x 0.25µm x 0.25 µm
Processing Method Name: Semivolatiles IPA-PCB soils
Instrument method Name: Semivolatiles IPA-PCB soils
Detector: Mass Spectrometer with ionization a 70eV and acquisition in SRM

6.1 Autosampler Parameters

General	
Injector type	Injector A (PTV)
Injector type	Single
Injection Mode	Basic

Rapid Mode	Disable
Syringe Volume (µL)	10
Needle Length (mm)	57
Sample injection volume (µL)	5
Plunger Strokes	3
Air Volume (µL)	0.5
Filling Volume (µL)	2
Sample vial depth (mm)	30

6.2 Injector Parameters

Front Inlet Temperature (°C)	80				
		Rate (mL/min)	**Temp** (mL/min)	**Time** (mL/min)	**Flow** (mL/min)
	Injection			0.10	
	Transfer	2.5	330	1	
	Cleaning	14.5	350	20	50
Split flow control	50 mL/min				
Purge Flow Control	5mL/min				
Gas Sever Flow	20mL/min				
Costant Flow	1.2mL/min				

6.3 GC Oven Ramp Parameters

Prep Run Timeout	10 min		
Oven Equilibration Time	0.10 min		
Ramped Temperature	**Rate** (°C/min)	**Target value** (°C)	**Hold Time** (min)
	-	70	1
	30	270	0
	5	340	4
Split flow control	50 mL/min		
Purge Flow Control	5mL/min		
Gas Sever Flow	20mL/min		

6.4 Mass Spectrometer Parameters

Name	RT	Window	Mass	Product Mass	Collision Energy
Nitrobenzene CAS 98-95-3	4,144	5	51	50,1	10
Nitrobenzene CAS 98-95-3	4,144	5	77,1	51,1	10
Nitrobenzene CAS 98-95-3	4,144	5	123,1	77,1	10
Naftalene-D8	4,62	5	136,1	84,1	20
Naftalene-D8	4,62	5	136,1	108,1	15
Naftalene	4,64	5	128,1	78,1	20
Naftalene	4,64	5	128,1	102	15
1-Cloro-3-nitrobenzene CAS 121-73-3	5,013	5	75,1	73,1	30
1-Cloro-3-nitrobenzene CAS 121-73-3	5,013	5	75,1	74,1	10
1-Cloro-3-nitrobenzene CAS 121-73-3	5,013	5	111	75,1	10
1-Cloro-4-nitrobenzene CAS 100-00-5	5,05	5	75,1	74	15
1-Cloro-4-nitrobenzene CAS 100-00-5	5,05	5	99	73	10
1-Cloro-4-nitrobenzene CAS 100-00-5	5,05	5	111	75,1	10
1-Cloro-2-nitrobenzene CAS 88-73-3	5,11	5	75,1	74,1	10
1-Cloro-2-nitrobenzene CAS 88-73-3	5,11	5	111	75,1	10
1-Cloro-2-nitrobenzene CAS 88-73-3	5,11	5	111	75,1	15
2.5-Dicloronitrobenzene CAS 89-61-2	5,714	5	109	74	10
2.5-Dicloronitrobenzene CAS 89-61-2	5,714	5	133	73	20
2.5-Dicloronitrobenzene CAS 89-61-2	5,714	5	145	109	10
3.4-Dicloronitrobenzene CAS 99-54-7	5,902	5	74,1	73,1	15
3.4-Dicloronitrobenzene CAS 99-54-7	5,902	5	109	74,1	10
3.4-Dicloronitrobenzene CAS 99-54-7	5,902	5	145	109	10
Acenapthylene	6,05	5	152,1	102	25
Acenapthylene	6,05	5	152,1	126,1	25
Acenapthylene-D10	6,17	5	162,2	158,1	20
Acenapthylene-D10	6,17	5	162,2	160,1	20
Acenapthene	6,2	5	153,1	151,1	30
Acenapthene	6,2	5	153,1	152,1	15
1.3-Dinitrobenzene CAS 99-65-0	6,213	5	63,1	62,1	15
1.3-Dinitrobenzene CAS 99-65-0	6,213	5	64,1	63,1	5
1.3-Dinitrobenzene CAS 99-65-0	6,213	5	168	75,1	20
1.2-Dinitrobenzene CAS 528-29-0	6,25	5	75,1	74,1	15
1.2-Dinitrobenzene CAS 528-29-0	6,25	5	76,1	50	10
1.2-Dinitrobenzene CAS 528-29-0	6,25	5	168,1	75,1	20
Fluorene	6,66	5	165,1	115	25
Fluorene	6,66	5	165,1	139	25
PCB-18	7,35	5	186	150	30
PCB-18	7,35	5	186	151,1	20
Fenantrene-D10	7,5	5	188,2	158,1	35
Fenantrene-D10	7,5	5	188,2	160,1	20

Fenantrene	7,52	5	178,1	152,1	20
Fenantrene	7,52	5	178,1	176	25
Antracene	7,58	5	178,1	152,1	20
Antracene	7,58	5	178,1	176	25
PCB-31	7,75	5	255,9	151,1	45
PCB-31	7,75	5	255,9	186	25
PCB-28	7,78	5	255,9	151,1	45
PCB-28	7,78	5	255,9	186	20
PCB-52	7,97	5	291,8	222	25
PCB-52	7,97	5	291,8	257	10
PCB-44	8,13	5	291,9	220	25
PCB-44	8,13	5	291,9	257	10
PCB-95	8,48	5	325,8	255,9	25
PCB-95	8,48	5	325,8	290,9	10
PCB-101	8,68	5	325,8	255,9	25
PCB-101	8,68	5	325,8	290,9	10
Fluorantene	8,71	5	202,1	200	30
Fluorantene	8,71	5	202,1	201,1	20
PCB-99	8,73	5	325,8	255,9	25
PCB-99	8,73	5	325,8	290,9	10
Pyrene	8,99	5	202,1	200	35
Pyrene	8,99	5	202,1	201,1	20
PCB-110	9,05	5	325,8	253,9	25
PCB-110	9,05	5	325,8	255,9	25
PCB-81	9,07	5	291,9	220	25
PCB-81	9,07	5	291,9	222	25
PCB-151	9,11	5	359,8	289,9	25
PCB-151	9,11	5	359,8	324,9	10
PCB-77	9,18	5	291,9	220	25
PCB-77	9,18	5	291,9	222	25
PCB-149	9,22	5	359,8	289,9	25
PCB-149	9,22	5	359,8	324,9	10
PCB-123	9,31	5	325,8	253,9	25
PCB-123	9,31	5	325,8	255,9	25
PCB-118	9,36	5	325,9	253,9	25
PCB-118	9,36	5	325,9	255,9	25
PCB-146	9,43	5	359,8	289,9	25
PCB-146	9,43	5	359,8	324,9	10
PCB-114	9,49	5	325,8	253,9	25
PCB-114	9,49	5	325,8	255,9	25
PCB-153	9,51	5	359,8	289,9	25
PCB-153	9,51	5	359,8	324,9	10

PCB-105	9,67	5	325,8	253,9	25
PCB-105	9,67	5	325,8	255,9	25
PCB-138	9,85	5	359,8	289,9	25
PCB-138	9,85	5	359,8	324,9	10
PCB-187	9,97	5	393,8	323,8	25
PCB-187	9,97	5	393,8	358,8	10
PCB-183	10,04	5	393,7	323,8	25
PCB-183	10,04	5	393,7	358,8	10
PCB-126	10,13	5	325,8	253,9	25
PCB-126	10,13	5	325,8	255,9	25
PCB-167	10,22	5	359,8	289,9	25
PCB-167	10,22	5	359,8	324,9	10
PCB-128	10,27	5	359,8	287,9	25
PCB-128	10,27	5	359,8	289,9	25
PCB-177	10,38	5	393,8	323,8	25
PCB-177	10,38	5	393,8	358,8	10
PCB-156	10,6	5	359,8	287,9	25
PCB-156	10,6	5	359,8	289,9	25
PCB-157	10,66	5	359,8	287,9	25
PCB-157	10,66	5	359,8	289,9	25
PCB-180	10,7	5	393,7	323,8	25
PCB-180	10,7	5	393,7	358,8	10
Benzo(a)antracene	10,85	5	228,1	226	30
Benzo(a)antracene	10,85	5	228,1	227,1	15
Crisene-D12	10,87	5	240,2	236,1	30
Crisene-D12	10,87	5	240,2	238,2	15
Crisene	10,93	5	228,1	226	30
Crisene	10,93	5	228,1	227,2	15
PCB-170	11,16	5	393,7	323,8	30
PCB-170	11,16	5	393,7	358,8	10
PCB-169	11,27	5	359,8	287,9	25
PCB-169	11,27	5	359,8	289,9	25
PCB-189	11,74	5	393,8	321,8	25
PCB-189	11,74	5	393,8	323,8	25
Benzo(b)fluorantene	13,32	5	252	226,1	25
Benzo(b)fluorantene	13,32	5	252	250	30
Benzo(k)fluorantene	13,37	5	252	226,1	25
Benzo(k)fluorantene	13,37	5	252	250	30
Benzo(e)pyrene	14,04	5	252,1	250	35
Benzo(e)pyrene	14,04	5	252,1	251,1	15
Benzo(a)pyrene	14,2	5	252,1	250	35
Benzo(a)pyrene	14,2	5	252,1	251,1	20

Perylene-D12	14,34	5	264,2	236,1	30
Perylene-D12	14,34	5	264,2	260,1	35
Perylene	14,42	5	252,1	250	35
Perylene	14,42	5	252,1	251,1	15
Dibenzo(a,h)antracene	17,25	5	278,1	276	30
Dibenzo(a,h)antracene	17,25	5	278,1	277,1	20
Indeno(1,2,3, cd)Pyrene	17,28	5	276,1	274	40
Indeno(1,2,3, cd)Pyrene	17,28	5	276,1	275,1	25
Benzo(g,h,i)perylene	18,03	5	276,1	274	40
Benzo(g,h,i)perylene	18,03	5	276,1	275,1	25
Dibenzo(a,e)pirene	21,07	5	302,1	300,1	35
Dibenzo(a,e)pirene	21,07	5	302,1	301,1	15
Dibenzo(a,i)pyrene	22,14	5	302,1	300,1	35
Dibenzo(a,h)pyrene	22,6	5	302,1	276,1	30
Dibenzo(a,h)pyrene	22,6	5	302,1	300,1	35
Dibenzo(a,l)pyrene	22,81	5	302,1	276,1	30
Dibenzo(a,l)pyrene	22,81	5	302,1	300	35
Benzylbenzoate	4,7	2	105	51	30
Benzylbenzoate	4,7	2	105	77	15
CP C10-C30 89ms	6	6	89	53	10
CP C10-C30 102ms	6	6	102	67	10
CP C10-C30 115ms	6	6	115	79	5
CP C10-C30 129-77ms	6	6	129	77	15
CP C10-C30 129-93ms	6	6	129	93	5

MS Transfer line Temp (°C)	290
Ion Source Temp (°C)	320
Ionization Mode	EI
Acquisition Mode	SRM

Determination of active pharmaceutical ingredients in soils and sludges

1. Purpose

This procedure aims to describe the operational methods and define specific details of the testing method for the determination of pharmaceutical active compounds in soils and sludges using liquid chromatography coupled with high-resolution mass spectrometry (HPLC-MS/MS).
The Table below shows the compounds under analysis.

Analite	CAS	Formula	Method	LOQ
Gabapentin-Lactam	64744-50-9	C9H15NO	A	1 µg/kg
Sulfamethoxazole	723-46-6	C10H11N3O3S	A	1 µg/kg
Metoprolol	51384-51-1	C15H25NO3	A	1 µg/kg
Azithromycin	83905-01-5	C38H72N2O12	A	1 µg/kg
Propyphenazone	479-92-5	C14H18N2O	A	1 µg/kg
Carbamazepine	298-46-4	C15H12N2O	A	1 µg/kg
Ketoprofen	22071-15-4	C16H14O3	A	1 µg/kg
Clarithromycin	81103-11-9	C38H69NO13	A	1 µg/kg
Diclofenac	15307-86-5	C14H11Cl2NO2	A	1 µg/kg
Irbesartan	138402-11-6	C25H28N6O	A	1 µg/kg
Lamotrigine	84057-84-1	C9H7Cl2N5	A	1 µg/kg
5-Methyl-Benzotriazole	136-85-6	C7H7N3	A	1 µg/kg
Amisulpride	71675-85-9	C17H27N3O4S	A	1 µg/kg
Ciprofloxacin Hydrochloride	86393-32-0	C17H21ClFN3O4	A	50 µg/kg
Ofloxacin/Levofloxacin	82419-36-1	C18H20FN3O4	A	50 µg/kg
Erythromycin	114-07-8	C37H67NO13	A	50 µg/kg
Citalopram	59729-32-7	C20H21FN2O	B	50 µg/kg
Clotrimazole	23593-75-1	C17H18FN3O3	B	50 µg/kg
Triclosan	3380-34-5	C12H7Cl3O2	B	50 µg/kg
17-Beta-estradiol	*50-28-2*	C18H24O2	B	50 µg/kg
17a-Ethinylestradiol	*57-63-6*	C20H24O2	B	50 µg/kg
Estrone	*53-16-7*	C18H22O2	B	50 µg/kg
Ibuprofen	15687-27-1	C13H18O2	B	50 µg/kg

These compounds are analysed using two different extraction procedures, referred to as "Method A" and "Method B".

2. Field of application

This testing method is applicable to all compounds listed in Table below, within the concentration range of 1 to 20 µg/kg for Method A, and from 50 to 1000 µg/kg for Method B.

3. Materials and Equipments

3.1 Ultrapure water
3.2 Methanol;
3.3 LC-MS grade Formic Acid 99%;
3.4 Acetonitrile;
3.5 PTFE syringe filters;
3.6 Quechers salts (MgSO4 and PSA);
3.7 Certified reference materials, either as individual compound salts or as analyte mixtures;
3.8 General laboratory glassware and equipment.
3.9 HPLC-HRMS Orbitrap®;

4. Sample preparation

4.1 Method A

4.1.1 Blank Preparation

Prepare the process blank as follows: in a 50 mL Falcon tube, add 25 mL of extraction solution. Proceed with the sample as described below.

4.1.2 Extraction for Sample Preparation

Weigh 10 ±0.1 g of sample into a 50 mL polypropylene Falcon tube. Pre-heat the ultrasonic bath to 80°C. Add 20 mL of extraction solution and shake manually to ensure proper mixing. Place the tubes in the ultrasonic bath and sonicate for 20 minutes. After extraction, allow samples to cool, then vortex for 5 minutes. Centrifuge at 7000 rpm for 5 minutes. In a 2 mL vial, mix 10 µL of internal standard solution at 100 µg/L, 900 µL of diluent solution, 100 µL of the centrifuged sample.

4.1.3 Recovery test – 500 ng/l in vial.

Prepare the recovery test in duplicate. Weigh 10 ±0.1 g of sample into a 50 mL Falcon tube, add 100 µL of native standard solution at 1 mg/L and process using extraction method B.

4.2 Method B

4.2.1 Blank Preparation

Prepare the process blank as follows: add 20 mL of hexane extraction solution to a 40 mL glass vial.

4.2.2 Extraction for Sample Preparation

Weigh 1.0 ± 0.5 g of sample into a 20 mL hexane-rinsed glass vials and add 10 µL of fortified standard solution at 10 mg/L. Shake manually, vortex for 20 minutes, and centrifuge at 3000 rpm for 3 minutes. Transfer 10 mL of the supernatant into a 10 mL conical glass vial, and evaporate to approximately 1 mL. Centrifuge again at 3000 rpm for 3 minutes, and transfer the final extract into a 2 mL glass vial.

4.2.3 Recovery test – 200 µg/L in vial.

Prepare the recovery test in duplicate. Weigh 1.0 ± 0.5 g of sample into a 40 mL glass vial, add 10 µL of fortified standard solution at 10 mg/L, then proceed with the extraction as described above.

5. Analysis UPLC-HRMS

5.1 Instrumental Parameters Method A

Instrument: UPLC Ultimate 3000 Dionex copuled with a High-Resolution Mass Detector Orbitrap.
Column: Raptor C18 50mm x 2.1mm x 1.8µm
Source: HESI polarity switching
Detector: Orbitrap Mass Spectrometer

5.1.1 Chromatography parameters

Column Temperature	35°C ± 2°C		
Mobile Phases	A) Water milliQ con 0.1% Formic Acid		
	B) Acetonitrile with 0.1% Formic Acid		
Chromatography	**Time (min)**	**Mobile Phase A (%)**	**Mobile Phase B (%)**
	0	95	5
	1	95	5
	2	80	20
	5	50	50
	7	50	50
	9	5	95
	9.01	95	5
	10	95	5
Flux	0.3 mL/min		
Injection Volume	80 µL		
Run Time	11 minutes		
Diluent	Water milliQ – Methanol 80/20 (v/v)		

5.1.2 Mass Spectrometry Parameters

5.1.2.1 ESI-Source Parameters

Polarity	Switching
Sheat gas flow rate	45
Aux gas flow rate	10
Sweep gas flow rate	0

Spray voltage	2.8 kV
Capillary Temp	150°C
S-lens RF level	50
Aux gas heater Temp	350°C

5.1.2.2 Full-Scan acquisition Parameters

Detector	Orbitrap Mass Spectrometer
	Full SCAN, ESI
	Resolution 70000 FWHM

Standard	**CAS**	**m/z**	**Ionizzation**
Amisulpride	71675-85-9	370.1795	[M+H]
Lamotrigine	84057-84-1	256.01513	[M+H]
Metoprolol	51384-51-1	268.19072	[M+H]
Azithromycin	83905-01-5	749.5158	[M+H]
5-Methyl-Benzotriazole	136-85-6	134.07109	[M+H]
Sulfamethoxazole	723-46-6	254.0593	[M+H]
Gabapentin-Lactam	64744-50-9	154.12264	[M+H]
Clarithromycin	81103-11-9	748.48416	[M+H]
Carbamazepine	298-46-4	237.10223	[M+H]
Irbesartan	138402-11-6	429.23973	[M+H]
Propyphenazone	479-92-5	231.14918	[M+H]
Ketoprofen	22071-15-4	253.08701	[M-H]
Diclofenac	15307-86-5	294.0094	[M-H]
Ofloxacin/Levofloxacin	82419-36-1	362.15106	[M+H]
Ciprofloxacin Hydrochloride	86393-32-0	332.14021	[M+H]
Erythromicin	114-07-8	734.4693	[M+H]
Ketoprofen D3	159490-55-8	256.10584	[M-H]
Ofloxacin D3	1173147-91-5	365.16989	[M+H]
Carbamazepine D10	132183-78-9	247.16501	[M+H]

5.2 Instrumental Parameters Method B

Instrument: TRACE1300 coupled with triple quadrupoleTSQ9000.
Column: Restek Rxi-XLB 30m, 0.25 ID 0.25um
Instrument Method Name: Drugs in sludges
Processing Method Name: Drugs in sludge
Source: EI

5.2.1 Autosampler parameters

General	
Injector type	Injector A (PTV)
Injector type	Single
Injection Mode	Basic
Rapid Mode	Disable
Syringe Volume (µL)	10
Needle Length (mm)	57
Sample injection volume (µL)	1
Plunger Strokes	3
Air Volume (µL)	0.5
Filling Volume (µL)	2
Sample vial depth (mm)	30

5.2.2 Injector parameters

Front Inlet Temperature (°C)	80				
		Rate (mL/min)	**Temp** (mL/min)	**Time** (mL/min)	**Flow** (mL/min)
	Injection	-	-	0.10	-
	Transfer	8	290	1	-
	Cleaning	14.5	330	20	50
Split flow control	50 mL/min				
Purge Flow Control	5mL/min				
Gas Sever Flow	20mL/min				

5.2.3 GC Oven Ramp Parameters

Prep Run Timeout	10 min			
Oven Equilibration Time	0.10 min			
Ramped Temperature	**Retention Time** (min)	**Rate** (°C/min)	**Target value** (°C)	**Hold Time** (min)
	0	0	80	1
	1	30	320	2
	11	20	340	2

Split flow control	50 mL/min
Purge Flow Control	5mL/min
Gas Sever Flow	20mL/min
Costant Flow	1.2mL/min

5.2.4 Mass Spectrometry Parameters

Analite	Mass	Product Mass
Citalopram	324	58
Clotrimazole	344	277
Triclosan	289	218
17-Beta-estradiol	272	272
17a-Ethinylestradiol	296	213
Estrone	270	270
Ibuprofen	206	161
MS Transfer line Temp (°C)	290	
Ion Source Temp (°C)	320	
Ionization Mode	EI	

Determination of Bisphenol in Soils and Sludges

1. Purpose

This procedure describes the operational methods and defines specific aspects of the internal method for the determination of Bisphenol A, B, S, F, and AF in soils and sludges using liquid chromatography coupled with high-resolution mass spectrometry (HPLC-MS/MS).
The table below shows the compounds under analysis.

Analite	CAS	Formula	LOQ
Bisphenol F	620-92-8	C13H12O2	50 µg/kg
Bisphenol A	80-05-7	C15H16O2	50 µg/kg
Bisphenol B	77-40-7	C16H18O2	50 µg/kg
Bisphenol AF	1478-61-1	C15H16O2	50 µg/kg
Bisphenol S	80-09-1	C12H10O4S	50 µg/kg
Bisphenol D16	-	C15D1602	Internal standard

2. Area of application

This testing method is applicable to bisphenols in the concentration range of 50 to 1000 µg/kg.

3. Materials and Equipment

3.1 Ultrapure water
3.2 Methanol;
3.3 HPLC-grade acetonitrile
3.4 Acetonitrile;
3.5 2mL glass vials with screw caps (Restek);
3.6 Pre-slit screw caps for 2mL vials;
3.7 Pipettes P1000;
3.8 Pipettes P20;
3.9 HPLC-HRMS Orbitrap®;
3.10 Analytical balance (±0.001 g)
3.11 Certified reference materials (as salts of individual compounds or as analyte mixtures)
3.12 General laboratory glassware

4. Sample Preparation – Dried Sludges

4.1 Blank Preparation

Prepare the process blank as follows: in a 50 mL Falcon tube, add 20 mL of methanol. Process in the same way as other samples.

4.2 Extraction for Sample Preparation

Weigh 10 ±0.1 g of sample into a 50 mL PP Falcon tube. Preheat the ultrasonic bath to 80°C. Add 20 mL of methanol and mix thoroughly. Sonicate for 20 minutes. Allow to cool, then vortex for 5 minutes and centrifuge at 7000 rpm for 5 minutes. In a 2 mL vial, mix 10 µL of internal standard solution at 1 mg/L, 900 µL of diluent solution, 100 µL of centrifuged sample. Filter with 13 mm PTFE filters if necessary. Use pre-slit LC vial caps.

4.3 Recovery Test – 2 µg/L in vial

Prepare the recovery test in duplicate. Weigh 10 ±0.1 g of sample into a 50 mL Falcon tube. Add 40 µL of 10 mg/L spike solution and process in the same way as other samples.

Note: The extraction procedure is the same as for pharmaceuticals, but instrument methods and spike levels differ.

5. Sample Preparation – Dried Sludges

5.1 Blank Preparation

Prepare the process blank as follows: in a 40 mL vial, add 10 mL of methanol. Proceed with the sample as described above.

5.2 Extraction for Sample Preparation

Weigh 1 ±0.1 g of sample into a 40 mL vial (adjust weight based on correction factor if dry weight result is required). Preheat ultrasonic bath to 80°C. Add 20 mL of methanol and mix. Sonicate for 20 minutes, allow to cool, vortex for 5 minutes, and centrifuge at 7000 rpm for 5 minutes. In a 2 mL vial, mix 10 µL of internal standard solution at 1 mg/L, 800 µL of diluent solution, 200 µL of centrifuged sample. Filter if needed. Use pre-slit LC vial caps.

5.3 Recovery Test – 2 µg/L in vial.

Prepare the recovery test in duplicate. Weigh 1 ±0.1 g of sample into a 40 mL vial, add 20 µL of 10 mg/L spike solution, and proceed as described above.

6. Analysis UPLC-HRMS

Instrument: UPLC Ultimate 3000 Dionex copuled with a High-Resolution Mass Detector Orbitrap.
Column: Raptor C18 100mm x 2.1mm x 1.8µm
Source: HESI negative polarity
Detector: Orbitrap Mass Spectrometer

6.1 Chromatography

Column Temperature	35°C ± 2°C
Mobile Phases	A) Ultra-pure water
	B) LC-MS grade Methanol

	Time (min)	Mobile Phase A (%)	Mobile Phase B (%)
Chromatography	0	80	20
	1	80	20
	3	50	50
	6	2	98
	8	2	98
	8.01	80	20
	10	80	20
Flux	0.35 mL/min		
Injection Volume	10 µL		
Run Time	10 minutes		
Diluent	Water milliQ – Methanol 80/20 (v/v)		

6.2 Mass Spectrometry Parameters

6.2.1 ESI-Source Parameters

Polarity	Negative
Sheat gas flow rate	48
Aux gas flow rate	11
Sweep gas flow rate	0
Spray voltage (kV)	3.7
Capillary Temp (°C)	300
S-lens RF level	52
Aux gas heater Temp (°C)	400

6.2.2 Mass Spectrometer Parameters

6.2.2.1 General Mass Spectrometer Parameters

General	
In Source CID	-
Targeted-MS²	
Resolution	70.000
Isolation Window	1.0 m/z
Default Charge	1
AGC Target	1e6
Maximum IT	auto

Microscans	1
Spectrum Data Acquisition	Profile

6.2.2.2 PRM Acquisition Parameters

Ione Parent → 227.1079; 241.1234; 241.19457; 249.0227; 199.07645; 335.05175 [M-H]

Frammenti → 228.10775; 212.0843; 250.0242; 200.07645; 336.05489

NCE → 30 e 50

Determination of Siloxanes and Chlorinated Naphthalenes in Sludges

1. Purpose

This procedure describes the operational methods and defines specific aspects of the internal method for the determination of siloxanes and chlorinated naphthalenes in sludges using gas chromatography and tandem mass spectrometry (GC-MS/MS).
The table below shows the compounds under analysis.

Analite	CAS	Formula	LOQ
Octamethylcyclotetrasiloxane	64744-50-9	C8H24O4Si4	0.2 mg/kg
Decamethylcyclopentasiloxane	723-46-6	C10H30O5Si5	0.2 mg/kg
Dodecamethylcyclohexasiloxane	51384-51-1	C12H36O6Si6	0.2 mg/kg
1-Chloronaphthalene	83905-01-5	C10H7Cl	0.2 mg/kg
2-Chloronaphthalene	479-92-5	C10H7Cl	0.2 mg/kg
1,2,3,4-Tetrachloronaphathalene	298-46-4	C10H4Cl4	0.2 mg/kg

2. Field of application

This method is applicable to the listed compounds in the concentration range of 200 to 2500 µg/kg.

3. Materials and Equipment

3.1 Hexane
3.2 40 mL screw-cap glass vials
3.3 2 mL glass vials (Restek)
3.4 Screw caps for 40 mL vials
3.5 Screw caps for 2 mL vials
3.6 Disposable pipette tips
3.7 Pipette P1000
3.8 Pipette P200
3.9 Pipette P20
3.10 GC system Thermo TRACE1300 + TSQ8000Evo triple quadrupole
3.11 Analytical balance (±0.001 g)
3.12 Certified reference materials (individual salts or analyte mixtures)
3.13 General laboratory glassware

4. Sample Preparation

4.1 Blank Preparation

In a 40 mL vial, add 10 mL of hexane. Proceed with the sample as for normal samples.

4.2 Extraction for Sample Preparation

Weigh 2 ±0.1 g of sample into a 40 mL glass vial (adjust for dry weight if necessary). Add 20 mL of hexane and vortex for 20 minutes. In a 2 mL vial, add 1 mL of extract, 10 µL of internal standard solution at 10 mg/L. Filter using 0.45 µm PTFE filters if necessary.

4.3 Recovery Test – 100 µg/L in vial

Prepare the recovery test in duplicate. Weigh 2 ±0.1 g of sample into a 40 mL glass vial. Add 10 µL of 100 mg/L spike solution and proceed as for normal samples.

5. GC-MS/MS Analysis

Instrument: TRACE1300 coupled with triple quadrupoleTSQ8000Evo.
Column: Restek Rxi-XLB 30m, 0.25 ID 0.25um
Instrument Method Name: Siloxanes + PCN sludge
Processing Method Name: Siloxanes + PCN sludge
Source: EI

5.1 Autosampler Parameters

General	
Injector type	Injector A (PTV)
Injector type	Single
Injection Mode	Basic
Rapid Mode	Disable
Syringe Volume (µL)	10
Needle Length (mm)	57
Sample injection volume (µL)	5
Plunger Strokes	3
Air Volume (µL)	0.5
Filling Volume (µL)	2
Sample vial depth (mm)	30

5.2 Injector Parameters

Front Inlet Temperature (°C)	80				
		Rate (mL/min)	**Temp** (mL/min)	**Time** (mL/min)	**Flow** (mL/min)
	Injection	-	-	0.10	-

	Transfer	8	290	1	-
	Cleaning	14.5	330	20	50
Split flow control	50 mL/min				
Purge Flow Control	5mL/min				
Gas Sever Flow	20mL/min				

Column helium flow: 1.2 ml/min

5.3 GC Oven Ramp Parameters

Prep Run Timeout	10 min			
Oven Equilibration Time	0.10 min			
Ramped Temperature	**Retention Time (min)**	**Rate (°C/min)**	**Target value (°C)**	**Hold Time (min)**
	0	-	-	-
	1	0	70	1
	12	30	340	2
Split flow control	50 mL/min			
Purge Flow Control	5mL/min			
Gas Sever Flow	20mL/min			
Costant Flow	1.2mL/min			

5.4 Mass Spectrometer Parameters

Analite	RT	Mass	Product Mass	Collision Energy
Octamethylcyclotetrasiloxane	3.01	267	178.9	30
Octamethylcyclotetrasiloxane	3.01	267	250.9	20
Decamethylcyclopentasiloxane	3.81	341	73	20
Decamethylcyclopentasiloxane	3.81	341	325.1	10
Dodecamethylcyclohexasiloxane	4.8	281	249	20
Dodecamethylcyclohexasiloxane	4.8	281	265	10
1-Chloronaphthalene	5.73	162	126.1	25
1-Chloronaphthalene	5.73	162	127.1	25
2-Chloronaphthalene	5.74	127.1	77.1	15
2-Chloronaphthalene	5.74	127.1	126	15
1,2,3,4-Tetrachloronaphathalene	8.21	194	159	30
1,2,3,4-Tetrachloronaphathalene	8.21	263.9	194	30
SV Internal standard mix 2mg/mL	-	-	-	-

MS Transfer line Temp (°C)	290
Ion Source Temp (°C)	320
Ionization Mode	EI

Determination of Semi-Volatile Organic Compounds in Soils Using Gas Chromatography-Mass Spectrometry (GC-MS)

1. Purpose

The purpose of this operational procedure is to outline the method for the determination of the semi-volatile organic compounds listed below, using GC-MS/MS in soils, in accordance with EPA methods 3541 (1994) and 8270D (2018).

2. Area of Application

This procedure is applicable to soils and land, and allows for the determination of the compounds listed in Tables 1, 2 at concentrations equal to or greater than 0.005 mg/kg. For the compounds in Table 3, the applicable concentration is equal to or greater than 0.500 mg/kg.

Polycyclic aromatic hydrocarbons (IPA)			
Name	**CAS**	**Formula**	**LOQ**
Benzo[a]pyrene	50-32-8	C20H12	5 µg/kg
Benzo[b]fluoranthene	205-99-2	C20H12	5 µg/kg
Benzo[g,h,i]perylene	191-24-2	C22H12	5 µg/kg
Benzo[k]fluoranthene	207-08-9	C20H12	5 µg/kg
Chrysene	218-01-9	C18H12	5 µg/kg
Dibenzo[a,e]pyrene	192-65-4	C24H14	5 µg/kg
Dibenzo[a,h]pyrene	189-64-0	C24H14	5 µg/kg
Dibenzo[a,i]pyrene	189-55-9	C24H14	5 µg/kg
Dibenzo[a,l]pyrene	191-30-0	C24H14	5 µg/kg
Indeno[1,2,3-cd] pyrene	193-39-5	C22H12	5 µg/kg
Dibenz[a,h]anthracene	53-70-3	C22H14	5 µg/kg

Table 1.

Polycloro Biphenyls (PCB)				
Name		**CAS**	**Formula**	**LOQ**
2,2',3,3',4,4',5-heptachlorobiphenyl	PCB 170	35065-30-6	C12H3Cl7	5 µg/kg
2,2',3,3',4,4'-hexachlorobiphenyl	PCB 128	38380-07-3	C12H4Cl6	5 µg/kg
2,2',3,3',4,5',6'-heptachlorobiphenyl	PCB 177	52663-70-4	C12H3Cl7	5 µg/kg
2,2',3,4,4',5,5'-heptachlorobiphenyl	PCB 180	35065-29-3	C12H3Cl7	5 µg/kg
2,2',3,4,4',5',6-heptachlorobiphenyl	PCB 183	52663-69-1	C12H3Cl7	5 µg/kg
2,2',3,4,4',5'-hexachlorobiphenyl	PCB 138	35065-28-2	C12H4Cl6	5 µg/kg
2,2',3,4',5,5',6-heptachlorobiphenyl	PCB 187	52663-68-0	C12H3Cl7	5 µg/kg
2,2',3,4',5,5'-hexachlorobiphenyl	PCB 146	51908-16-8	C12H4Cl6	5 µg/kg
2,2',3,4',5',6-hexachlorobiphenyl	PCB 149	38380-04-0	C12H4Cl6	5 µg/kg
2,2',3,5,5',6-hexachlorobiphenyl	PCB 151	52663-63-5	C12H4Cl6	5 µg/kg
2,2',3,5',6-pentachlorobiphenyl	PCB 95	38379-99-6	C12H5Cl5	5 µg/kg

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