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Occurrence and levels of selected compounds in European compost and digestate samples

Results of a Pan European Screening exercise FATE-COMES

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Abstract

This report describes work conducted by the European Commission's Joint Research Centre (JRC) in the context of an Administrative Arrangement between DG Environment and the JRC.

This work aimed at the generation, within a limited timeframe, of a large amount of analytical data, with high scientific and statistical value, for a number of compost and digestate types (afterwards referred to as COMDIG samples), to help provide a general overview and estimation of that possible variability within and between different COMDIG materials.

The report includes the results of a targeted and independent screening of typical European situations of COMDIG materials with regard to the occurrence and levels of compounds of concern, many of which have never been assessed at a pan-European level.

In total, 139 samples, mostly taken as grab samples and originating from 15 countries, were assessed for 22 minor and trace elements and 92 organic compounds including ingredients of personal care products and pharmaceuticals.

The underlying analytical methods are carefully documented with regard to their performance characteristics. Where available, the so-called "horizontal" standards were followed.

The results obtained are assessed statistically.

Although the analysed single samples are insufficient to make any statement on the performance of the treatment processes leading to COMDIG samples, this collective of data provide a glimpse of the pan-European situation as regards the studied compounds.

Participating laboratories and roles

The findings presented in this report are the result of a large collaborative effort. For confidentiality reasons, the identities of the participating composting and digestion plants cannot be revealed in this public report, but are known to the European Commission. This anonymity shall not relieve our acknowledgment of the considerable support and in-kind contribution behind the exercise. As regards the practical execution of the project organization and the work in the laboratories the following persons contributed actively.

European Commission

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- Bernd Manfred Gawlik (Project Leader)
- Giovanni Locoro (Coordination of sampling operations and Logistics, CV-AAS, independent sampling)
- Michela Ghiani (Technical correspondence and liaison with sites)
- Sara Comero (ICP-AES, CV-AAS, Statistical evaluation)
- Robert Loos (Perfluoroalkyl substances; PFAS)
- Simona Tavazzi, Bruno Paracchini (Assistant Project Leader, LC-MS/MS)
- Giulio Mariani (Dioxins, PCBs, PBDEs analysis),
- Giulio Mariani, Gert Suurkuusk, (GC-MS)
- Gunther Umlauf (long-term study on POPs)
- Carmen Cristache, Isabelle Fissiaux, Agustin Alonso-Ruiz (Analytical support)

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VITO NV

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- Stefan Voorspoels (Phenols analysis)

List of Abbreviations and Symbols

Throughout this report the following abbreviations and symbols are used:

2,4-D	2,4-Dichlorophenoxyacetic acid
2,4,5-T	2,4,5-Trichlorophenoxyacetic acid
AAS	atomic absorption spectrometry
AES	atomic emission spectrometry
AMA	advanced mercury analyzer
BCR	Bureau Communautaire de Reference
BDL	below detection limit
COMDIG	compost and digestate sample
CRM	certified reference material
CV	cold-vapour
CV%	coefficient of variation
D4	Octamethylcyclotetrasiloxane
D5	Decamethylcyclopentasiloxane
D6	Dodecamethylcyclohexasiloxane
DEA	diethyl-ammonium
DDC	diethyl-dithiocarbamate
DG	Directorate-General
DL	dioxin-like
EC	European Commission
EU	European Union
F	factor (from PMF)
EVF	explained variation of factor
GC	gas chromatography
ICP	inductively coupled plasma
IES	Institute for Environment and Sustainability
IUPAC	International Union for Pure and Applied Chemistry
IPTS	Institute for Prospective and Technological Studies
JRC	Joint Research Centre
K	coverage factor
KOW	octanol/water partition coefficient
LC	liquid chromatography
LoD	limit of detection
LoQ	limit of quantification
MBT	Mechanical Biological Treatment
MCPA	2-methyl-4-chlorophenoxyacetic acid
MDL	method detection limit
MDM	Octamethyltrisiloxane
MD2M	Decamethyltetrasiloxane
MD3M	Dodecamethylpentasiloxane
MRM	multiple reaction monitoring
MS	mass spectrometry
PAH	polyaromatic hydrocarbon
PCA	principal component analysis
PCB	polychlorinated biphenyls
PCM	polycyclic musk
PFASs	perfluoroalkyl substances
PCDD/F	polychlorinated dibenzo-dioxin/furane
PFNA	perfluorononanoic acid
PFOA	perfluorooctanoic acid
PFOS	perfluorooctane sulfonate
PMF	positive matrix factorization
POP	persistent organic pollutant
R	repeatability
SOP	standard operation procedure
SRM	standard reference material
SSL	sewage sludge
STD	standard deviation
STP	sludge treatment plant
$u_{combined}$	combined uncertainty
U	expanded uncertainty
u(r)	combined uncertainty for repeatability
u(IP)	combined uncertainty for repeatability
u(t)	combined uncertainty for repeatability
UHPLC	Ultra High Pressure Liquid Chromatography
U.o.M.	unit of measurand
US	United States of America
WGS84	World Geodetic System 1984
WWTP	waste water treatment plant

Chemical elements are identified by the respective symbol according to IUPAC

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1 Introduction

The Waste Framework Directive [1], in the following referred to as 'the Directive' or WFD, among other amendments introduces a new procedure for defining end-of-waste (EoW) criteria, which are criteria that a given waste stream has to fulfill in order to cease to be waste. In this context, a methodology guideline to develop end-of-waste criteria has been elaborated by the Joint Research Centre's Institute for Prospective Technological Studies (JRC-IPTS) as part of the so-called 'End-of-Waste Criteria report' [2]. The European Commission is now working on preparing proposals for end-of-waste criteria for specific waste streams according to the legal conditions and following the JRC methodology guidelines.

In this context, scientific background data on the levels of organic and inorganic pollutants in different types of compost and digestate were requested from JRC IES to support the decision-making process for end-of-waste criteria. Especially the issue of allowing COMDIG from mechanical biological treatment is intensively debated, thus indicating the need for independent statistical data. Furthermore, the availability of inorganic and organic pollutant data turned out to be less ubiquitous for digestate than for some compost types. Following further reflections and internal discussions, it was decided to generate these necessary scientific data through a pan-European collaborative screening exercise.

The campaign consisted of measuring a large series of biodegradable waste samples in the best possible standardized way and aimed at the following two objectives:

1. Generate, within a limited timeframe, a large amount of analytical data, with high scientific and statistical value, for a number of compost and digestate types, to allow a general overview and estimation of possible variability within and between different compost/digestate materials.
2. Guarantee maximal objectivity, minimal variation and the smallest possible bias upon sampling by independent, unannounced control sampling performed by a single team composed of EC JRC staff only, at selected plants participating in the collaborative screening exercise.

This exercise experienced some difficulties in the beginning. However, through the Members of the Technical Working Group for End-of-Waste (EoW) for Biodegradable Waste, it was possible to obtain access to a significant number of relevant sites, most of which were members of the European Compost Network (ECN).

Thus, under the project-name FATE-COMES, 139 geo-referenced samples distributed over the following bio-waste categories were analysed by the JRC and its collaborating laboratories:

1. **BW Co**: Compost produced from separately collected organic waste from households and similar commercial institutions, including garden and park waste
2. **GW Co**:Compost produced from garden and park waste only (green compost)
3. **SS Co**:Sewage sludge compost produced from good quality sewage sludge and other separately collected organic waste (e.g. garden and park waste, straw, etc.)
4. **MBT Co**: Municipal Solid Waste compost generated by Mechanical Biological Treatment aimed at producing compost (derived from non-hazardous household waste and similar commercial waste where no separate collection of household waste is in place)
5. **BW Di**: Digestates from source separated biowastes from households and similar commercial institutions (liquid and solid fraction)
6. **Man BW Di**: Digestates from manure and source separated biowastes from households and similar commercial institutions (liquid and solid fraction)
7. **Man Ecr Di**: Digestates from manure and energy crops (liquid and solid fraction)
8. **MBT Di**: Digestate derived from Mechanical Biological Treatment of Municipal Solid Waste, aimed at producing digestate for use in agriculture (derived from non-hazardous household waste and similar commercial waste)
9. **Other** minor categories: These include bark compost or municipal solid waste compost like output generated by Mechanical Biological Treatment aimed at stabilizing a rest fraction sent to landfill.

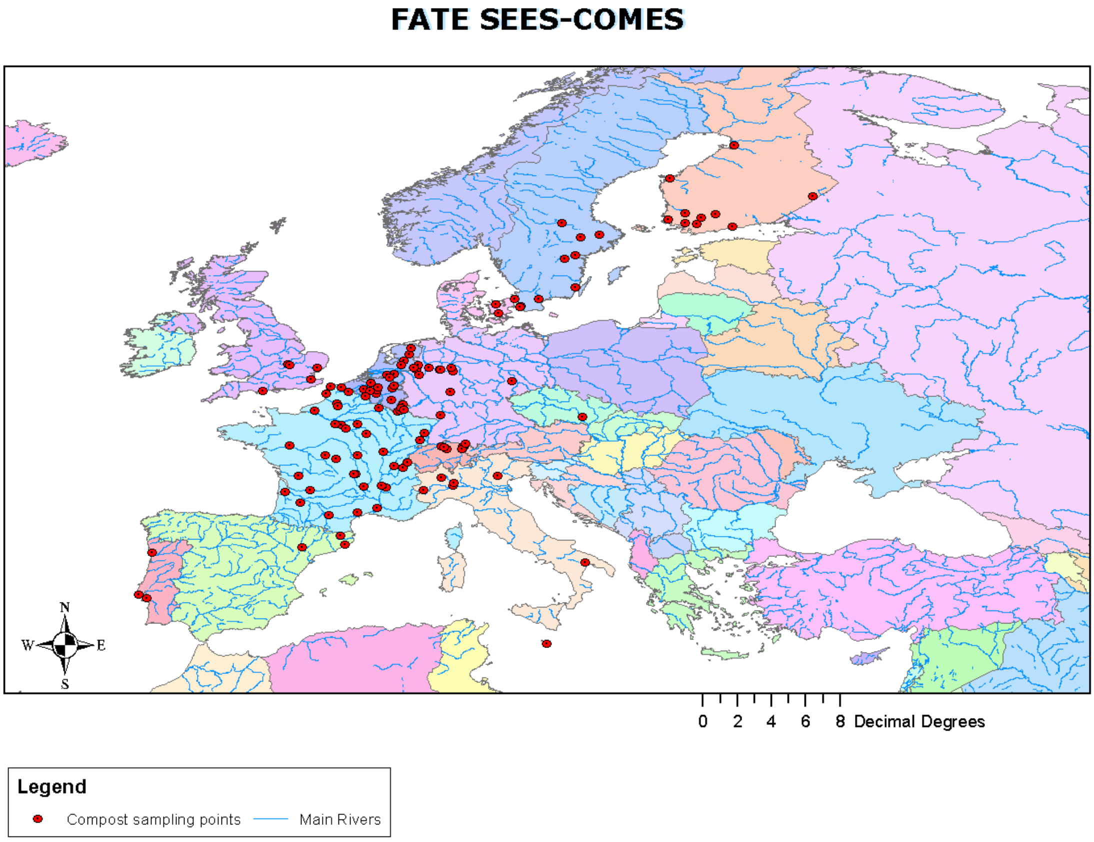


Figure 1: Maps of sampling sites accessed during FATE-COMES (proximate locations)

For the first objective, allowing a broad screening of different materials and technologies, samples were taken by the COMDIG producers, following a standardized sampling protocol and the sampling method EN 12579 for soil improvers and special methods for digestates, in sample containers provided by the JRC-IES, and shipped back to JRC-IES for analysis.

For the second objective, the JRC selected some COMDIG producing plants from the list of participating producers and performed an unannounced sampling according to the same sampling protocol and method EN 12579 (other methods for digestates) as for the received samples.

Both exercises were ensured by the JRC, including shipment of empty and filled sampling containers to and from the stations by a contracted carrier, selected sample taking by the JRC team, chemical analyses, data treatment and publication of results. Participants provided as in-kind contribution personnel resources for organizing sampling and shipment preparation (i.e. preparing the packages).

The analysed compounds included the most frequently occurring PCBs, PAHs, PCDD/F, PBDEs, phenols, siloxanes, impurities, heavy metals and pesticides as well as some less investigated and emerging compounds such as perfluorinated surfactants, sweeteners, pharmaceuticals and polycyclic musks.

The results of FATE-COMES, which are presented in this report, should feed the discussions regarding end-of-waste criteria such as e.g. product quality, input materials or quality assurance. The campaign was done in conjunction with two parallel exercises on sewage sludge and effluents of wastewater treatment plants. The design of the experiment follows previous successful pan-European measurement campaigns such as FATE-EUMORE (surface water) and FATE-GROWS (groundwater) and has been nowadays also considered as a support tool for the difficult prioritisation processes under the European Water Framework Directive 2000/60/EC.

Table 1: Chemicals analyzed in FATE COMES survey

Metals	Hg	**Phenols**	Nitrophenol
	Ag		Phenol
	As		o-Cresol
	Ba		m-Cresol
	Co		p-Cresol
	Cr		2-Chlorophenol
	Cu		2,6-Dimethylphenol
	Mn		o-Ethylphenol
	Mo		3-Chlorophenol
	Ni		2,5-Dimethylphenol
	Pb		4-Chlorophenol
	Se		2,4-Dimethylphenol
	Ti		m-Ethylphenol
	V		(p-Et+3,5-DiMe)phenol
	Zn		2,3-Dimethylphenol
	Al		3,4-Dimethylphenol
	Fe		2,6-Dichlorophenol
	Mg		4-Chloro-3-methylphenol
	Cd		2,5-Dichlorophenol
	Sb		2,4-Dichlorophenol
	K		2,3,5-trimethylphenol
	P		3,5-Dichlorophenol
PCM	Cashmeran		2,3-Dichlorophenol
	Celestolid		3,4-Dichlorophenol
	Phantolid		4-chloor-3,5-dimethylphenol (=dettol)
	Traesolid		2,4,6-Trichlorophenol
	Galaxolid		2,3,6-Trichlorophenol
	Tonalid		2,3,5-Trichlorophenol
PAH	Phenantrene		2,4,5-Trichlorophenol
	Antracene		2,3,4-Trichlorophenol
	Fluoranthene		3,4,5-Trichlorophenol
	Pyrene		2,3,5,6-Tetrachlorophenol
	Benzo(a)antracene		2,3,4,6-Tetrachlorophenol
	Chrysene		Octylphenol
	Benzo(b)fluoranthene		2,3,4,5-Tetrachlorophenol
	Benzo(k)fluoranthene		Nonylphenol
	Benzo(e)pyrene		Pentachlorophenol
	Benzo(a)pyrene		Bisphenol A
	Perylene		2,4-Dinitrophenol

	Indeno(1,2,3-cd)pyrene
	Dibenz(a,h)antracene
	Benzo(g,h,i)perylene
	Dibenzo(a,l)pyrene
	Dibenzo(a,h)pyrene
	Dibenzo(a,i)pyrene
	Dibenzo(a,e)pyrene
	Coronene
PFASs	PFOA
	PFNA
	PFOS
Pesticides	2,4-D
	Dichlorprop
	Mecoprop
	MCPA
	2,4,5-T
	Bentazone
	Imidacloprid
PBDE	BDE-17
	BDE-28
	BDE-47
	BDE-49
	BDE-66
	BDE-71
	BDE-85
	BDE-99
	BDE-100
	BDE-119
	BDE-138
	BDE-153
	BDE-154
	BDE-183
	BDE-196
	BDE-197
	BDE-203
	BDE-206
	BDE-207
	BDE-208
	BDE-209
Siloxanes	Octamethyltrisiloxan (MDM)
	Octamethylcyclotetrasiloxan (D4)
	Decamethyltetrasiloxan (MD2M)
	Decamethylcyclopentasiloxan (D5)

Sweeteners	Acesulfame K
	Sucralose
	Saccharin
Pharmaceuticals	Diclofenac
	Ibuprofen
	Ketoprofen
	Acetylsalicylic acid
	Naproxen
	Bezafibrate
	Gemfibrozil
	Chloramphenicol
	Clofibric acid
Dioxins and furans	2378-TCDD
	12378-PeCDD
	123478-HxCDD
	123678-HxCDD
	123789-HxCDD
	1234678-HpCDD
	OCDD
	2378-TCDF
	12378-PeCDF
	23478-PeCDF
	123478-HxCDF
	123678-HxCDF
	234678-HxCDF
	123789-HxCDF
	1234678-HpCDF
	1234789-HpCDF
	OCDF
PCBs	PCB-81
	PCB-77
	PCB-126
	PCB-169
	PeCB-105
	PeCB-114
	PeCB-118
	PeCB-123
	HxCB-156
	HxCB-157
	HxCB-167
	HpCB-189
	TriCB-28
	TeCB-52

	Dodecamethylpentasiloxan (MD3M) Dodecamethylcyclohexasiloxan (D6)		PeCB-101 HxCB-138
Impurities	>20 mm plastic light > 5 mm stones > 2 mm plastic rigid > 2 mm plastic light > 2 mm stones > 2 mm glass > 2 mm metals		HxCB-153 HpCB-180

2. EXPERIMENTAL DESIGN AND PROCEDURES OF THE STUDY

2.1. Description of the campaign and selection of sampling sites

In order to reduce the organizational and financial efforts for participating plants, there was no obligation to perform independent sampling by external accredited sample takers and plants were allowed to perform the sampling themselves. Where possible, JRC recommended using EN 12579 for solid samples and EN ISO 5667-13- 1997 "Water quality -Sampling - Part 13: Guidance on sampling of sludges from sewage and water-treatment works" for liquid samples. Alternatively, plants could use their usual sampling method.

The European Compost Network prepared a sampling protocol, which was a modified version of the Sampling Record described in their Quality Assurance Scheme and which was distributed by the JRC to the participating plants.

An Administrative Arrangement was established between DG ENV and the JRC with the purpose to provide support to DG ENV for the revision of the Sewage Sludge Directive.

In order to facilitate the collaboration with the COMDIG plants, a clear mandate e.g. from the responsible Commission service to the JRC was needed.

This mandate clearly guaranteed that the obtained results would have not used to "judge" the performance of a given COMDIG plant, but aimed at the compilation of knowledge on emerging organic contaminants that may pose a problem.

The contribution of each country to the campaign is summarised in the Table 2.

Table 2 Contribution of EU countries

Country	**No. of sample**
Austria:	8
Belgium	13
Czech Republic	1
Denmark	2
Finland	13
France	39
Germany	17
Italy	7
Luxembourg	4
Malta	1
Portugal:	3
Spain:	4
Switzerland :	5
Sweden:	10
The Netherlands:	7
United Kingdom	5

Each participant was asked to compile the accompanying documentation (i.e.: sampling bill) with the following relevant information:

- Country Address
- Geographic coordinates (WGS84)
- Sample typology
- Attachments (possible photos, SOPs, or further information deemed useful)

A COMDIG sample inventory was build up at JRC for sample distribution, analytical processing and data coordination.

Data were registered in the IES Environmental Laboratory Data Information Management System, which allowed also retrieving the data on a geo-referenced basis.

Upon completion the samples were stored in the IES Compost Sample Archive in case that a need for further characterisation arose. Since this was an action limited in time, the size of the archive was manageable and cheap.

Exact location and origin of the compost samples is confidential and will not be disseminated.

2.2. Experimental methods

2.2.1. Heavy metals

The methods for the determination of heavy metals and mercury content by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) and Could Vapour-Atomic Adsorption Spectrometry (CV-AAS) techniques, respectively, according to the ISO 17025 requirement and the prEn16170, prEN16174 and prEN16175-1, were fully validated and implemented in the analysis of sludge samples.

The two methods were validated using Certified Reference Materials (CRMs) such as: BCR 141R 'Calcareous Loam Soil', BCR 142 'Light Sandy Soil', "San Joaquin Soil" SRM 2789 and LCG 6181 'sewage sludge'.

The calibration curves, detection and quantification limits, trueness as well as repeatability were determined. The budget uncertainty was also estimated (including a full uncertainty budget and Ishikawa-diagram) according to the guide EURACHEM/CITAC Guide CG 4.

2.2.1.1. Sample preparation

Sludge samples were freeze-dried using GAMMA 1-16 LSC (Christ) instrument in order to reduce water content. After that samples were homogenized and ground in an agate ball mixer mill to reduce particle size to a maximum of 630µm.

A Multiwave 3000 microwave (Anton Paar) device was employed for samples digestion.

*A*bout 0.1 g of each sample (soil, sludge, compost and CRMs) was weighted and introduced into a high-pressure, closed, Teflon decomposition vessel. The mixture of 1.5 millilitres of HNO_3 and 4.5 millilitres of HCl (*i.e.* a defined mixture known as *'aqua regia'*) were carefully added to each sample and the vessels were gently shaken, sealed and digested in microwave oven under previously optimized operating conditions. Blank solutions were prepared by applying the same procedure and reagent solutions without sample.

The microwave autoclave can simultaneously digest up to 48 samples in the reaction chamber under identical experimental conditions. The maximum pressure of the reaction chamber with sample vessels inside was set to 1225 bar. Then the vessels were heated in the microwave autoclave for 35 min reaching a temperature of maximum 140 °C and a pressure of approximately 20bar. The pressure and temperature were monitored during all the analysis by the use of a T/P (Temperature/Pressure) sensor. Before opening the reaction chamber, the digests were allowed to cool for about 180 min to well below the boiling point of the acid mixture at atmospheric pressure.

Each extract was filtered in a 50 ml glass flask using a clean glass funnel and a Minisart RC 25 filter. The vessel and the vessel cup were subsequently rinsed three times with Milli-Q water and the rinse water was filtered in the same flask. At the end, the flask was completed to volume.

The resulting samples were stored at 4 ºC until analyses.

2.2.2. ICP-OES analysis

For ICP analysis an aliquot of the digested samples was transferred to the ICP sample holder vials. The following elements were determined: Ag, Al, As, Ba, Cd, Co, Cr, Cu, Fe, Mg, K, Mn, Mo, Ni, P, Pb, Sb, Se, Ti, V and Zn. The low calibration range was from 0.02 to 0.5 mg/l. The high calibration range was from 0.5 to 5 mg/l.

Table 3: LoD and LoQ for the selected metals in COMDIG samples

Elements	***LOD***	***LOQ***	**Elements**	***LOD***	***LOQ***
Ag	0.07	0.14	*Mo*	0.28	0.56
Al	1.53	3.06	*Ni*	0.27	0.53
As	2.84	5.67	*Pb*	1.16	2.33
Ba	0.02	0.04	*Sb*	0.81	1.61
Cd	0.07	0.15	*Se*	1.78	3.56
Co	0.15	0.30	*Ti*	0.03	0.05
Cr	0.32	0.64	*V*	0.66	1.33
Cu	0.26	0.52	*Zn*	2.12	4.23
Fe	6.66	13.32	*P*	3.03	6.06
Mg	3.58	7.15	*K*	4.83	9.66
Mn	0.02	0.03			

For the elements Ba, Mn, Se and Ti, a blank was used for the computation.

Table 4: Average recoveries for the selected metals obtained in COMDIG samples

Elements	***LOW***	***HIGH***
Ag	84%	82%
Al	-	59%
As	83%	94%
Ba	88%	45%
Cd	85%	90%
Co	96%	94%
Cr	66%	98%
Cu	89%	99%
Fe	-	85%
Mg	-	88%
Mn	87%	94%
Mo	91%	86%
Ni	81%	98%
Pb	66%	95%
Sb	83%	91%

Se	83%	92%
Ti	90%	92%
V	93%	97%
Zn	-	95%
P	-	116%
K	-	27%

Table 5: Expanded uncertainty of ICP-OES determination

Analyte	***LOW***	***HIGH***
Ag	4.0%	5.9%
Al	-	6.1%
As	7.6%	3.1%
Ba	5.3%	7.1%
Cd	4.5%	6.4%
Co	7.4%	3.9%
Cr	5.8%	1.1%
Cu	3.5%	5.1%
Fe		5.4%
Mg		6.5%
Mn	4.1%	6.8%
Mo	2.5%	3.5%
Ni	5.4%	1.9%
Pb	7.0%	2.4%
Sb	6.8%	10.1%
Se	3.1%	9.3%
Ti	8.3%	10.5%
V	4.3%	3.1%
Zn	-	5.9%
P	-	14.2%
K	-	20.0%

Could Vapour-Atomic Adsorption Spectrometry (CV-AAS) analysis

The determination of Hg was carried out by Cold Vapour-Atomic Absorption Spectrometry (CV-AAS) technique using an Advanced Mercury Analyser instrument (AMA 254, Altec).

Samples were measured after Lyophilisation (freeze-drying) process.

Table 6: CV-AAS operational conditions

Parameter	
Drying time	60s
Decomposition time	200s
Cuvette clear time	45s
Delay	0s
Cell to use for analysis	Low / High cell
Metric to use for calculation	Peak area

The low calibration range was from 0.05 to 0.5 mg/l. The high calibration range was from 0.5 to 5 mg/l.

In order to estimate LoD and LoQ, due to the non-availability of a soil, sludge and compost sample containing Hg at very low concentration, a blank was analysed. Ten replicates were made in order to compute the standard deviation.

For 10 measurements and at a 95% confidence level ($\alpha = 0.05$) the $\Phi_{n\ \alpha}$ factor is equal to 1.9.

LOQ is computed using a *k* factor of 2, which give a 50% of accuracy.

We get for LOD and LOQ the following values:

LOD = 4 µg/L
LOQ= 8 µg/L

Low recoveries were computed using the following certified reference materials (CRMs): BCR 141R *calcareous loam soil* (0.25mg/kg Hg) and BCR 142R *Light sandy soil (*0.067mg/kg Hg).

For method validation, CRMs were analysed in triplicate for five different days. Results are presented in theTable 7.

Table 7: Results of replicate analysis of CRM BCR 141R and 142R

	Day 1	***Day 2***	***Day 3***	***Day 4***	***Day 5***	***Average***
BCR 141R	113%	103%	103%	104%	108%	106%
BCR 142R	107%	96%	95%	99%	106%	101%

For the *high recovery* the CRMs: SRM 2789 *San Joaquin Soil* (4.9 mg/kg Hg) and LCG 6181 (1.4 mg/kg Hg) were used. The results are presented in the following Table 8.

Table 8: Results of replicate analysis of CRM LCG 618 and SRM 2789

	Day1	***Day 2***	***Day 3***	***Day 4***	***Day 5***	***Average***
LCG 6181	110%	122%	117%	120%	115%	117%
SRM 2789	118%	120%	106%	109%	111%	113%

In order to take into account a confidence level, the combined uncertainty is to be multiplied by a coverage factor, *k*, to produce the expanded uncertainty. The choice of this factor was done taking into account a 95% confidence level, which give a coverage factor of 2.

The expanded uncertainty is given by:

$$u_{expanded} = k \cdot u_{combined}$$

To compute the expanded uncertainty we chose the higher combined uncertainty in both low and high calibration.

In percentage terms, an expanded uncertainty of 7% in low calibration and 8% in high calibration was obtained.

2.2.3. Polycyclic musk compounds

A gas chromatography coupled to mass spectrometric detection (GC-MS) method for the determination of polycylic musk compounds in compost samples was developed.

The method was developed for the analysis of the following compounds: cashmerane, celestolide, phantolide, traesolide galaxolide and tonalide.

After addition of an internal standard (deuterated tonalide and hexachlorbenzene-c13) the samples (1 g) were extracted with 20 ml ethanol/sodium acetate puffer. Additionally 400 µl DEA-DCC (diethylammoniumdiethyldithiocarbamate) were added as a complexing agent. The samples were shaken overhead for about 2.5 hours. After addition of 20 ml n-hexane the samples were shaken for another 60 minutes. The extracts were centrifuged for a better phase separation (3000 U/min, 5min) and the hexane phase was separated. After another extraction with 5 ml of n-hexane, the organic phase was evaporated to approximately 5 ml and a clean-up step was performed with aluminium oxide (2 g deactivated by baking at 400 °C for 4 hours and activated with 10 % water). The analytes were eluted by a mixture of n-hexane/ethylacetate (90:10, v:v). The extracts were evaporated to less than 900 µl with a gentle stream of nitrogen. After addition of an injection standard the extracts were filled up to a final volume of 1 ml and an aliquot (1 µl) is injected into a GC-MS system. The substances were detected using the EI-GC-MS in the SIM mode.

2.2.3.1. GC-MS analysis

The operating conditions for GC-MS analysis are reported below:

Table 9: Operating condition for GC-MS PMCs analysis

Column:	
J&W DB5-MS	
Nominal length 60m	
Nominal Diameter 0.25 mm	
Nominal film thickness 0.25 µm	
Mode constant flow	
Initial flow 1.5 ml / min Helium	
Oven:	
Initial Temperature 40°C	
Initial Time 1’	
Ramps: 6°c/min up to 120°C 10°C/min up to 330°C hold for 3 min.	
Run Time 38 min	
Front Inlet:	
Mode splitless	Initial Temperature 260 °C
Initial Temperature 260 °C	Equilibration Time 1 ‘

Pressure --	Initial Time --
Purge Flow --	Rate --
Purge Time --	Final Temp --
Total Flow 1.5 ml/min	Hold Time --
Gas saver --	
Gas Type Helium	
MS Quad (℃) not heated	
MS Source (℃) 255 °C	

Table 10: LoD and LoQ of PMC determination by GC-MS

	Cashmerane	**Celestolide**	**Phantolide**	**Traesolide**	**Galaxolide**	**Tonalide**
	µg/kg d.m.					
LOD	5	7.5	5	5	10	5
LOQ	10	15	10	10	20	10
Recovery	84%	91%	85%	87%	81%	80%
Est. Uncertainty	26%	29%	18%	24%	22%	17%

Table 11: Recovery of PMCs

	Cashmerane	**Celestolide**	**Phantolide**	**Traesolide**	**Galaxolide**	**Tonalide**
Recovery	84%	91%	85%	87%	81%	80%
Est. Uncertainty	26%	29%	18%	24%	22%	17%

Table 12: Estimated uncertainty of PCMs determination

	Cashmerane	**Celestolide**	**Phantolide**	**Traesolide**	**Galaxolide**	**Tonalide**
Est. Uncertainty	26%	29%	18%	24%	22%	17%

2.2.4. Siloxanes

A gas chromatography coupled to mass spectrometric detection (GC-MS) method for the determination of siloxanes in compost was developed and characterized.

The method was developed for the analysis of the following compounds: octamethyltrisiloxan (MDM), octamethylcyclotetrasiloxan (D4), decamethyltetrasiloxan (MD2M), decamethylcyclopentasiloxan (D5), dodecamethylpentasiloxan (MD3M), and dodecamethylcyclohexasiloxan (D6).

After addition of an internal standard (tetrachlorbenzene $^{13}C_6$) the samples (1 g) were extracted with 20 ml ethanol/sodium acetate puffer. Additionally 400 µl DEA-DCC (diethylammoniumdiethyldithiocarbamate) was added as a complexing agent. The samples were shaken overhead for about 2.5 hours. After addition of 20 ml n-hexane the samples were shaken for another 60 minutes. The extracts were centrifuged for a better phase separation (3000 U/min, 5min) and the hexane phase was separated. After another extraction with 5 ml of n-hexane, the organic phase was evaporated to approx.. 5 ml and a clean-up step was performed with aluminium oxide (2 g deactivated by baking at 400 °C for 4 hours and activated with 10 % water). The analytes were eluted by a mixture of n-hexane/ethylacetate (90:10, v:v). The extracts were evaporated to less than 900 µl with a gentle stream of nitrogen. After addition of an injection standard, the extracts were filled up to a final volume of 1 ml and an aliquot (1 µl) was injected into a GC-MS system. The substances were detected using the EI-GC-MS in the SIM mode.

2.2.4.1. GC-MS analysis

The operating conditions for GC-MS analysis are reported below:

Table 13: Operating conditions for GC-MS siloxanes analysis

Column:	
J&W DB5-MS	
Nominal length 60m	
Nominal Diameter 0.25 mm	
Nominal film thickness 0.25 µm	
Mode constant flow	
Initial flow 1.5 ml / min Helium	
Oven:	
Initial Temperature 40°C	
Initial Time 1’	
Ramps: 6°c/min up to 120°C 10°C/min up to 330°C hold for 3 min.	
Run Time 38 min	
Front Inlet:	
Mode splitless	Initial Temperature 260 °C
Initial Temperature 260 °C	Equilibration Time 1 ‘
Pressure --	Initial Time --
Purge Flow --	Rate --
Purge Time --	Final Temp --
Total Flow 1.5 ml/min	Hold Time --
Gas saver --	
Gas Type Helium	
MS Quad (℃) not heated	
MS Source (℃) 255 °C	

Table 14: LoD and LoQ of siloxanes determination by GC-MS

	MDM	D4	MD2M	D5	MD3M	D6
			µg/kg d.m.			
LOD	5	30	5	30	5	60
LOQ	10	60	10	60	10	120

Table 15: Recovery of siloxanes

	MDM	D4	MD2M	D5	MD3M	D6
Recovery	71%	77%	86%	91%	85%	90%

Table 16: Estimated uncertainty of siloxanes determination

	MDM	D4	MD2M	D5	MD3M	D6
Est. Uncertainty	25%	37%	25%	28%	29%	11%

2.2.5. PAHs

The methods for the determination of polycyclic aromatic hydrocarbons content by gas chromatography coupled to mass spectrometric detection (GC-MS) was fully validated and implemented for the determination of PAHs content in COMDIG samples.

The method was characterized using Reference Materials such as contaminated soil samples from Intercalibration trials (*i.e.*: contaminated soils S13 and SU6, UNICHIM Interlaboratory Trials "Policyclic Aromatic Hydrocarbon in environmental matrices", 2007 and 2010, respectively). The selectivity, linearity, detection and quantification limits, trueness, repeatability, recovery and stability of the extracts were determined. The uncertainty estimation was based on method performance. This approach is based on the fact that the combined influence of many effects is quantified simultaneously by estimating repeatability, intermediate precision and trueness.

The method was developed for the analysis of the following compounds: Phenantrene, Antracene, Fluoranthene, Pyrene, Benzo(a)antracene, Chrysene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(e)pyrene, Benzo(a)pyrene, Perylene, Indeno(1,2,3-cd)pyrene, Dibenz(a,h)antracene, Benzo(g,h,i)perylene, Dibenzo(a,l)pyrene, Dibenzo(a,h)pyrene, Dibenzo(a,i)pyrene, Dibenzo(a,e)pyrene, Coronene.

Naphthalene, Acenaphthylene, Acenaphtene and Fluorene were not determined because of their high volatility and their unlikely presence in COMDIG lyophilised samples.

About 0.1 g of lyophilized COMDIG sample were weighted in a 10 mL glass centrifuge tube; 50 µL of Custom PAH Surrogate Standard Mixture (0.5 ng/µL) were added together with 0.5 mL of extraction solvent (Hexane: Acetone, 80:20, %v/v). The samples were mixed by vortex for 10 seconds and ultra-sonicated for 10 minutes at 1000 rpm. The supernatant was then collected into a clean 10 mL glass centrifuge tube. A second extraction was performed on the original sample, adding a second aliquot of 0.5 mL of extraction solvent (Hexane: Acetone, 80:20, %v/v). The supernatant from the second extraction was decanted into the same 10 mL glass centrifuge vial where the first was collected. The resulting sample was then mixed by vortex for 10 seconds, centrifuged at 1000 rpm for 10 minutes and added with 50 µL of Custom PAH Syringe Standard Mixture (0.5 ng/µL) before being transferred in amber glass vial for analysis.

GC-MS analysis

The operating conditions for GC-MS analysis are reported below:

Table 17: Operating conditions for GC-MS PAHs analysis

Column:			
SGE ID-BPX-50			
Nominal length	60 m		
Nominal Diameter	250 µm		
Nominal film thickness	0.25 µm		
Mode	constant flow		
Initial flow	1 mL/min		
Oven:			
Initial Temperature	100°C		
Initial Time	3 min		
Ramps:	#		
15°C/min up to 220°C			
2°C/min up to 300°C and held for 20 min			
3°C/min up to 340°C and held for 30 min			
Run Time 114.33 min			
Front Inlet (CIS4):		**Gerstel CIS 4**	
Mode	Splitless	Initial Temperature	100 °C
Initial Temperature	0°C	Equilibration Time	0.05 min
Pressure	144.5 kPa	Initial Time	0.05 min
Purge Flow	50 mL/min	Rate	12°C /sec
Purge Time	1 min	Final Temp	300°C
Total Flow	53.7 mL/min	Hold Time	3 min
Gas saver	off		
Gas Type	Helium		
MS Quad	150°C		
MS Source	230°C		

The analytes were identified using their retention times and selected ion masses. The quantification was made using the response factors between analytes and their isotopically labelled internal surrogate standards. The retention times were detected by analysing periodically the standard solution containing all the compounds and isotopically labelled surrogates and syringe standards.

Linearity of developed procedure in sludge samples was studied for the low concentration range (30 to 500 ng/g) and high concentration range (0 to 9610 ng/g), by analysing 4 calibration solutions for each range.

For all compounds at both concentration levels the R^2 values were >0.99. It can be stated, that the analytical method is linear in this range.

The LoD and LoQ were estimated analysing blank samples containing analytes at very low level with signal to noise ratio (RMS S/N) from 8 to 35. The following formulas (recommended by EURACHEM [3]) were used to calculate the LOD and LOQ values:

$$\text{LOD} = \text{blank} + 3s_L$$

$$\text{LOQ} = \text{blank} + 10s_L$$

where the blank is mean value of ten analyses of blank samples and s_L is the standard deviation of these ten replicates.

The LOD and LOQ for the analytes in soil and COMDIG samples are shown in the following Table 18.

Table 18: LoD and LoQ of PAHs determination by GC-MS

Compound	**LOD ng/g**	**LOQ ng/g**
Phenanthrene	7.2	10.7
Anthracene	4.6	7.8
Fluoranthene	4.3	5.3
Pyrene	4.8	6.0
Benzo(a)anthracene	4.0	5.6
Chrysene	4.7	6.8
Benzo(b)fluoranthene	7.6	10.7
Benzo(k)fluoranthene	6.5	11.6
Benzo(e)pyrene	7.4	11.5
Benzo(a)pyrene	4.3	6.4
Perilene	4.8	7.4
Indeno(1,2,3-cd)pyrene	7.9	13.6
Dibenzo(a,h)anthracene	4.6	7.9
Benzo(g,h,i)perilene	6.6	11.6
Dibenzo(a,l)pyrene	58.7	92.9
Dibenzo(a,h)pyrene	56.4	97.0
Dibenzo(a,i)pyrene	585.9	848.0
Dibenzo(a,e)pyrene	664.1	961.9
Coronene	53.6	88.1

Recovery values were evaluated by the ratio between each surrogate compound and the opportune labelled compound added to sample extracts as syringe standard. Recovery was calculated in two different concentration levels using the data received on the repeatability and intermediate precision study. The average recovery results are shown in the following Table 19.

Table 19: Recovery of PAHs

Compound	**S13, high C**	**SU6, low C**
	Recovery	
Phenantrene	62%	76%
Antracene	64%	78%
Fluoranthene	67%	89%
Pyrene	68%	83%
Benzo(a)antracene	74%	80%
Chrysene	74%	80%
Benzo(b)fluoranthene	75%	72%
Benzo(k)fluoranthene	75%	72%
Benzo(e)pyrene	70%	70%
Benzo(a)pyrene	76%	70%
Perylene	69%	71%

Indeno(1,2,3-cd)pyrene	67%	59%
Dibenz(a,h)antracene	74%	69%
Benzo(g,h,i)perylene	56%	51%
Dibenzo(a,l)pyrene	63%	33%
Dibenzo(a,h)pyrene	63%	33%
Dibenzo(a,i)pyrene	63%	33%
Dibenzo(a,e)pyrene	63%	33%
Coronene	31%	30%

Expanded uncertainty (U) was estimated using the approach, where the repeatability, intermediate precision and trueness estimation results were combined, using the following formula:

$$U = k\sqrt{u_r{}^2 + u_{ip}{}^2 + u_t^2};$$

where,

$$u_r = \frac{s_r}{\sqrt{n}} ,$$

where **s$_r$** is the relative repeatability standard deviation from the validation study and **n** is the number of replicates performed;

$$u_{ip} = \frac{s_d}{\sqrt{d}},$$

where **s$_d$** is the relative day-to-day variation from the validation study and **d** is the number of days over which the measurements were spread;

$$u_t = \sqrt{\frac{s_t^2}{n_t} + \frac{\sum u_{mat}^2}{n_{mat}^2}},$$

where **s$_t$** and **n$_t$** are accordingly the relative standard deviation and the number of replicates of the trueness experiment of the validation study and **u$_{mat}$** and **n$_{mat}$** are accordingly the relative uncertainty and the number of materials used for trueness estimation. As the certified soil samples from Intercalibration trials were used as CRM, the u_{mat} was calculated as follows:

$$u_{mat} = \frac{s_i}{\sqrt{n_i}},$$

where **s$_i$** is the standard deviation of the results in intercalibration trials and **n$_i$** is the number of laboratories participated in this trial;

k is the coverage factor, a coverage factor of 2 is chosen to give about 95% probability.

The relative influences of repeatability, intermediate precision and trueness (bias) are shown in the Table 20. Because of the lack of the CRMs, it was not possible to estimate the uncertainty for each compound.

Table 20: Repeatability, intermediate precision and trueness of PAHs determination

Compound	**HIGH conc.**			**LOW conc.**		
	u(r)	**u(ip)**	**u(t)**	**u(r)**	**u(ip)**	**u(t)**
Phenanthrene	0.3%	4.3%	10%	0.2%	4.8%	
Anthracene	0.9%	3.0%	10%	4.2%	5.5%	12%
Fluoranthene	0.7%	4.1%	10%	0.7%	4.1%	11%
Pyrene	0.5%	3.2%	10%	0.5%	4.3%	10%
Benzo(a)anthracene	0.2%	2.8%	10%	0.5%	5.8%	10%
Chrysene	0.3%	2.2%	10%	0.3%	5.6%	10%
Benzo(b)fluoranthene	0.3%	2.0%	10%	0.4%	4.7%	10%
Benzo(k)fluoranthene	0.3%	1.7%	10%	1.4%	5.3%	10%
Benzo(e)pyrene	0.3%	1.6%		0.2%	4.4%	10%
Benzo(a)pyrene	0.5%	1.3%	10%	0.6%	6.4%	11%
Perilene	1.1%	2.0%		1.2%	5.7%	

Indeno(1,2,3-cd)pyrene	0.9%	2.9%		1.4%	6.3%	12%
Dibenzo(a,h)anthracene	0.9%	2.2%	11%	2.3%	4.5%	
Benzo(g,h,i)perilene	0.7%	2.3%	11%	0.5%	5.4%	10%
Dibenzo(a,l)pyrene						
Dibenzo(a,h)pyrene				1.3%	8.3%	
Dibenzo(a,i)pyrene						
Dibenzo(a,e)pyrene						
Coronene	2.1%	5.8%		0.6%	8.1%	

Taking into account that the estimated combined uncertainties for analytes did not vary a lot (relative standard deviation is less than 10%) and there were no available data that could be used for uncertainty evaluation for each analyte the mean combined uncertainty must be applied for each compound. The mean uncertainty was calculated from expanded uncertainties for low concentration level as they are bigger than the same figures calculated for high concentration level. The expanded relative uncertainty that applies for all analytes was calculated to be 24%. The estimated combined uncertainties together with expanded uncertainties are shown in the following Table 21.

Table 21: Combined uncertainties and expanded uncertainties for PAHs

Compound	**High conc.**		**Low conc.**	
	u	**U**	**u**	**U**
Phenanthrene	11%	**22%**		
Anthracene	11%	**21%**	14%	**28%**
Fluoranthene	11%	**22%**	12%	**23%**
Pyrene	10%	**21%**	11%	**22%**
Benzo(a)anthracene	11%	**21%**	12%	**24%**
Chrysene	11%	**21%**	12%	**24%**
Benzo(b)fluoranthene	10%	**20%**	11%	**22%**
Benzo(k)fluoranthene	10%	**20%**	12%	**24%**
Benzo(e)pyrene			11%	**22%**
Benzo(a)pyrene	10%	**20%**	12%	**25%**
Indeno(1,2,3-cd)pyrene			13%	**27%**
Dibenz(a,h)anthracene	12%	**23%**		
Benzo(g,h,i)perilene	11%	**22%**	11%	**23%**
AVERAGE		**21%**		**24%**
Rel. St. Deviation		**5%**		**8%**

2.2.6. AhR-active compounds

For the determination of AhR-active compounds samples were extracted by dichloromethane (2g of dry matter with 150 ml of dichloromethane using automatic extractor Büchi System B-811, 1 hour extraction). Extracts were concentrated (automatic extractor) to approximate volume 5 ml and transferred to vials and then further concentrated by nitrogen stream to the last drop and then re-dissolved in methanol (0,5ml) and stored frozen until analyses.

The H4IIE-luc, rat hepato-carcinoma cells stably transfected with the luciferase gene under control of the arylhydrocarbon receptor (AhR) was used (Giesy et al. 2002). H4IIE-luc cells were cultured in Dulbecco's modified Eagle medium (DMEM)(PAA, Austria) with 10% fetal calf serum in incubator with 5% CO2 at 37°C. For testing, H4IIE-luc cells were seeded into 96-well plates (15000 cells per well). After 24 hours, dilution series of tested samples, calibration (0.4-500pM TCDD - 2,3,7,8-tetrachloro-dibenzo-p-dioxin) and solvent control were added (final concentration of the solvent was 0.5% v/v). Exposures were conducted in three replicates for 24 hours. After the exposure, luminescence intensity was measured using Promega Steady Glo kit (Promega, Mannheim, Germany). Dioxin-like potencies were determined using the equi-effective approach and the results were expressed as dioxin-like equivalents (TEQbio) with respect to standard 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD).

For a subset of 20 compost and digestate samples a portion of the sample (200µL) was further treated to remove less-persistent pollutants (like PAHs) using sulfuric acid silica gel column. Persistent organic compounds were then eluted from the column by a mixture of dichloromethane/hexane (40ml), concentrated by nitrogen stream to the last drop and dissolved in 200µL of methanol again. This procedure removed non-persistent compound, and the final sample contained only POPs such as PCDD/Fs, PCBs and OCPs. Comparison of dioxin-like effects between the crude sample (containing both PAHs and chlorinated POPs) with the H2SO4-treated sample (only POPs) provided more detailed insight to the actual chemicals responsible for biological effects observed.

2.2.7. PCDD/Fs and PCB chemical analysis of selected samples based on CALUX Bioassay results

All standards (calibration sets, natives and mass labelled) were purchased from Wellington Laboratories (Canada). The extracts prepared in dichloromethane were spiked with ^{13}C PCDDs/Fs (according to EN-1948) and ^{13}C dl-PCBs (77, 81, 126, 169, 105, 114, 118, 123, 156, 157, 167 and 189). The concentrated extracts were cleaned-up on a sulfuric acid-modified (44% w/w) silica column, eluted with 40 mL DCM/n-hexane mixture (1:1). Fractionation was achieved in a micro column (6 mm i.d) containing from the bottom to top: 50 mg silica, 70 mg charcoal (Darco G60, Sigma-Aldrich)/silica (1:40) and 50 mg of silica. The column was pre-washed with 5 mL of toluene, followed by 5 mL of DCM/cyclohexane mixture (30%), then the sample was applied and eluted with 9 mL DCM/cyclohexane mixture (30%) in fraction 1 (mono-ortho dl-PCBs) and 40 mL of toluene in fraction 2 (PCDDs/Fs, non-ortho dl-PCBs). Each fraction was concentrated using the stream of nitrogen in a TurboVap II concentrator unit (Caliper LifeSciences, USA) and transferred into an insert in a vial. The syringe standards (^{13}C 1,2,3,4-TCDD and 1,2,3,7,8,9-HxCDD, ^{13}C PCBs 70, 111, 138 and 170) were added to all samples. The final volume prepared for analyses was 50 microliters. HRGC/HRMS instrumental analysis of PCDDs/Fs and dl-PCBs was performed on an 7890A GC (Agilent, USA) equipped with a 60m x 0.25mm x 0.25um DB5-MS column (Agilent J&W, USA) coupled to an AutoSpec Premier MS (Waters, Micromass, UK). The MS was operated in EI+ mode at the resolution of >10 000.

Analysis of indicator PCBs was performed by GC-MS/MS using 6890N GC (Agilent, USA) equipped with a 60m x 0.25mm x 0.25um DB5-MS column (Agilent J&W, USA) coupled to Quattro MicroGC MS (Waters, Micromass, UK) operated in EI+. Injection was splitless 1 µL at 280°C, He as carrier gas at 1.5 mL min-1. The GC temperature programme was 80°C (1 min hold), then 15°C min-1 to 180°C, and finally
5°C min-1 to 300°C (5 min hold).

2.2.8. PCDD/Fs, EC-6 PCBs, DL-PCBs and PBDEs chemical analysis in the framework of comparative sampling

The analysis of all compounds was done using isotope dilution and HRGC/HRMS techniques.

68-CVS and 68-LCS were native and ^{13}C-labelled internal standards for 12 congeners DL-PCBs (Wellington Laboratories Guelph, Ontario, Canada). EC-4058 was native for indicator-PCBs (CIL, Andover, Massachusetts, USA). ^{13}C-labelled PCB-31, PCB-111 and PCB-170 were used as recovery standards (Wellington Laboratories Guelph, Ontario, Canada).

EPA-1613CVS, EPA1613LCS and EPA-1613ISS were native, ^{13}C-labelled internal and recovery standards respectively for 17 PCDDs/Fs. The standards were obtained from Wellington Laboratories (Guelph, Ontario, Canada).

Ten 13C-labelled PBDE congeners were used as internal standards, (in accordance with IUPAC nomenclature: BDE-28, BDE-47, BDE-99, BDE-100, BDE-153, BDE-154, BDE-183; BDE-197, BDE-207 and BDE-209), Nine present in MBDE-MXE-STK solution (in accordance with IUPAC nomenclature: BDE-28, BDE-47, BDE-99, BDE-153, BDE-154, BDE-183; BDE-197, BDE-207 and BDE-209) and one BDE-100 was added from the solution MBDE-100. ^{13}C-labelled BDE-126 and BDE-206 were used as recovery standards. BDE-MXE was native solution. All PBDE standards were obtained from Wellington Laboratories (Guelph, Ontario, Canada).

All organic solvents used were Dioxin analysis grade (Sigma-Aldrich, Buchs SG, Switzerland). Sulphuric acid was 98% extra pure (VWR International s.r.l., Milan, Italy). Multi-residual clean-up of PCDD/F, PCBs and PBDEs was conducted on ready to use acidic silica/silica, basic alumina and carbon columns (Fluid Management Systems (FMS) Inc., Watertown, MA, USA).

2.2.8.1. Extraction and Clean-up for PCDD/Fs, PCBs and PBDEs:

The samples were lyophilized, disaggregated and homogenised in a mortar, and finally sieved < 2 mm. 5g of dry sample was extracted with a mixture of n-hexane/acetone (220/30) by Soxhlet for 24 h after spiking with isotope-labelled surrogate standards. Copper powder was added to the solvent during the extraction to remove Sulphur.

The extract was subjected to an automated clean-up for the purification and separation of the fractions containing PCDD/F, PCBs and PBDEs.

After treatment of the raw extract with conc. H_2SO_4 extract purification was executed with an automated clean-up system (Power-Prep P6, Fluid Management Systems (FMS) Inc., Watertown, MA, USA). This system was previously described [4] [5] [6] [7] uses a multi-layer silica column (acid/neutral), basic alumina and carbon column combination. Two fractions were collected, one containing Mono-ortho PCBs, Indicator-PCBs and PBDEs and one for Non-ortho PCBs and PCDD/Fs. After evaporation of the solvents to near dryness, the syringe standards were added and a final volume of 30-100 µl was adjusted.

2.2.8.2. Pooled samples extraction and Clean-up for PBDEs

The samples were lyophilized, disaggregated and homogenised in a mortar, and finally sieved < 2 mm. Pools were made on the basis of different characteristics of the treatment plants. The purpose was to characterize the emissions of PBDEs in these systems.

Pooled compost GW Co, SS Co and MBT Co were analysed in duplicate in order to evaluate the pool homogeneity.

From 0.4 to 1g of dry pooled sample was extracted three time with a mixture of n-hexane/acetone (220/30) by ultrasonic for 20 min. after spiking with isotope-labelled surrogate standards. Copper powder was added to the solvent during the extraction to remove Sulphur.

The extract was centrifuged at 1500 RPM for 10 min., separated from the solid fraction, concentrated and submitted to purification.

The raw extract purification was executed with an automated clean-up system SPE module (J2 Scientific, Missouri, USA).

This system, previously described [8] [9] used a multi-layer silica column (acid/neutral) (Supelco, Bellefonte, PA, USA).

After evaporation of the solvents to near dryness, the syringe standards were added and a final volume of 30-100 µl was adjusted.

All instrumental analysis of PCDD/Fs, PCBs and PBDEs were based on isotope dilution using HRGC-HRMS (high resolution gas chromatography – high resolution mass spectrometry) for quantification on the basis of [10], [11], [12].

2.2.8.3. Instrumental analysis in the framework of comparative sampling

Non-ortho PCBs, PCDD/Fs and PBDEs were analyzed on double HRGC (Thermo Trace GC Ultra, Thermo Electron, Bremen, Germany), coupled with a DFS high resolution mass spectrometer HRMS (Thermo Electron, Bremen, Germany) operating in the EI-mode at 45 eV with a resolution of >10000. For Non-ortho PCBs, PCDD/Fs the two most abundant ions of the isotopic molecular cluster were recorded for both native and labelled congeners.

For tri- to octa-brominated congeners two ions of the isotopic molecular cluster were recorded, for nona- and deca-brominated congeners two isotopic ions of the cluster M^+-2Br were recorded for both native and labelled congeners.

The Non-ortho PCBs and PCDD/Fs were separated on a BP-DXN 60 m long with 0.25 mm i.d. (inner diameter) and 0.25 µm film (SGE, Victoria, Australia). The following gas-chromatographic conditions were applied for non-ortho PCBs, PCDD/Fs: split/splitless injector at 280 °C, constant flow at 1.0 ml min^{-1} of He, GC-MS interface at 300 °C and a GC program rate: 160 °C with a 1 min. hold, then 2.5 °C min^{-1} to 300 °C and a final hold at 300 °C for 8 min.

PBDEs were analyzed on a Sol-Gel-1ms, 15 m with 0.25 mm i.d. and 0.1 µm film GC column (SGE, Victoria, Australia). The following gas-chromatographic conditions were applied: PTV injector with temperature program from 110 to 300 °C at 14.5 °C sec^{-1}, constant flow at 1.0 ml min^{-1} of He, GC-MS interface at 300 °C and a GC program rate: 110 °C with a 1 min. hold, then 20 °C min^{-1} to 300 °C and a final hold at 300 °C for 6 min. The selection of the chromatographic conditions was optimized following the literature indications [13], [5], [14], [15].

Mono-ortho PCBs and Indicator-PCBs were analysed on a GC (HP-6890, Hewlett Packard, Waldbronn, Germany) coupled with a VG Autospec Ultima high resolution mass spectrometer (Micromass, Manchester, UK) operating in EI-mode at 34 eV with a resolution of >10000.

Indicator-PCBs and Mono-ortho PCBs were separated on HT-8 capillary columns, 60 m long with 0.25 mm i.d. (inner diameter) and 0.25 µm film (SGE, Victoria, Australia).

Gas chromatographic conditions for Mono-ortho PCBs were: Split/splitless injector at 280 °C, constant flow at 1.5 ml min^{-1} of He, GC-MS interface at 280 °C and a GC program rate: Starting from 120 °C with 20 °C min^{-1} to 180 °C, 2 °C min^{-1} to 260 °C, and 5 °C min^{-1} to 300 °C isotherm for 4 min.

The quantified isomers were identified through retention time comparison of the corresponding standard and the isotopic ratios between two ions was recorded for all halogenated compounds analysed

Analytical blanks were performed and analysed during the samples analysis.

The averages of the internal standard recoveries were 50%, 66% and 65% respectively for PCDD/Fs, PCBs and PBDEs.

2.2.9. Perfluoroalkyl substances

Two perfluoroalkyl carboxylates (PFOA (C_8) and PFNA (C_9)) and perfluorooctane sulfonate (PFOS) were analysed by ultra-high pressure liquid chromatography coupled to tandem mass spectrometric detection (UHPLC-MS-MS). Internal quantification was applied for PFAS determination by the use of labeled surrogate analogues (PFOA $^{13}C_4$, PFNA $^{13}C_5$, and PFOS $^{13}C_4$).

The PFASs were extracted from the COMDIG samples by solid-liquid extraction (SLE) with methanol in an ultrasonic bath followed by Envi-Carb graphitized carbon clean-up. This “matrix effect-free” extraction method for the determination of various PFASs in soil, sediment and sludge with LOQs in the ng/g range was described by Powley et al. [16]. The analytical protocol is straightforward and robust.

The extraction efficiency, detection and quantification limits were determined.

2.2.9.1. Sample preparation

About 1 gr. of lyophilized COMDIG were weighted in a 50 mL Sarstedt PP conical centrifuge tube, added with 100 µL of internal standard solution (PFOA $^{13}C_4$, PFOS $^{13}C_4$ and PFNA $^{13}C_5$, 1 mg/L in methanol) and 10 mL of pure methanol. The sample was mixed by vortex for 30 seconds and ultra-sonicated for 18 minutes. The supernatant was decanted into a second, clean 50 mL Sarstedt PP conical centrifuge tube. The original sample was extracted twice again and the supernatants added to the first extraction one. The 30 mL combined extract were evaporated to 10 mL volume using gentle stream of nitrogen at 35°C. 1 mL of the evaporated combined extract was transferred into 1.5 mL disposable polypropylene microcentrifuge tubes containing 25 mg of ENVI-Carb sorbent previously acidified with 50 µl of glacial acetic acid. The sample was mixed by vortex for 30 second and centrifuged at 6720 rcf for 30 minutes. 0.8 mL were then evaporated to 0.2 mL volume under gentle stream of nitrogen added of 0.2 mL of water and analysed by LC-MS/MS.

2.2.9.2. Extraction efficiency

Extraction efficiency was evaluated by subsequent extraction of a selected compost or digestate sample, according to the procedure reported above.

The results are summarised in the following table:

Table 22: Extraction efficiency for PFASs

	Mean Extraction efficiency (n=3, three sequential extractions)	St. Dev	CV%
PFNA	85.5	2.93	3.4
PFOA	88.7	2.53	2.9
PFOS	93.0	0.09	0.1

The compound-dependent method detection limits (MDLs or LODs) for the procedure were calculated from the mean concentrations of procedural blanks plus 3 times the standard deviation.

The LOQ for the analytes in samples are shown in the following Table:

Table 23: LoQ of PFASs determination by UHPLC-MS/MS

Conc (ng/g)		
PFOA	**PFNA**	**PFOS**
0.6	0.1	0.07

2.2.10. Non-target screening

Further to the analysis of the agreed chemicals, the multi-residue analytical method, based on ultra-high performance liquid chromatography tandem mass spectrometry (UHPLC-MS/MS), allowed the monitoring the semi-quantitative determination of several chemicals not initially included in the list of chemicals to be analysed in COMDIG materials.

For this simple reason this activity is reported as "Non-target screening" and regarded more than 60 multiple-class compounds, including pesticides, phenols, sweeteners, pharmaceuticals, benzotriazoles and personal care products.

Semi-quantitative determination was performed using external standard quantification method comparing the area counts of the compound's MRM transitions in the sample and the corresponding MRM transition in the analytical standard.

The studied compounds and their respective MRM transitions are listed in the table below.

Table 24: Selected organic contaminants and relative MRM transitions

Compound	MRM transitions	Compound	MRM transitions
Atrazine	216 > 174; 104	Mecoprop	213 > 141; 105
Atrazine-desethyl	188 > 146; 104	Bentazone	239 > 132; 197
Atrazine-desisopropyl	174 > 104; 79	MCPA	199 > 141; 105
Terbutylazine	230 > 174; 132	Dichlorprop	233 > 161; 125
Terbutylazine-desethyl	203 > 78	Nitrophenol	138 > 108; 92
Terbutryn	242 > 186; 91	2,4-Dinitrophenol	183 > 109; 123
Simazine	202 > 104; 132	Acesulfame K	162 > 78; 82
Propazine	230 > 146; 188	Sucralose	395 > 359
Diuron	233 > 72; 133	Saccharin	182 > 42; 106
Isoproturon	207 > 72; 165	Acetylsalicylic acid	137 > 93
Chlortoluron	336 > 235; 219	Carbamazepine	237 > 194; 165
Linuron	249 > 160; 133	Ibuprofen	205 > 161; 159

Alachlor	270 > 238; 162	Diclofenac	294 > 250; 214
Metolachlor	284 > 252; 176	Ketoprofen	253 > 209; 197
Diazinon	305 > 169; 97	Naproxen	229 > 169; 185
Molinate	188 > 126; 98	Gemfibrozil	249 > 121; 106
Metoxuron	229 > 72; 156	Clofibric acid	213 > 127; 85
Hexazinone	253 > 171; 85	Bezafibrate	360 > 274; 154
Carbaryl	202 > 145; 127	Atenolol	267 > 145; 190
Carbendazim	192 > 160; 105	Metopropol	268 > 116; 103
Carbofuran	222 > 123; 165	Propanolol	260 > 255; 237
Chloridazon	222 > 77; 65	Sotalol	273 > 133; 255
Chloridazon-desphenyl	213 > 72; 140	Tamoxifen	372 > 72; 129
Chloridazon-methyl-desphenyl	60 > 88; 101	Triclosan	287 > 35
Fenitrothion	278 > 109; 79	Caffeine	195 > 138; 110
Flusilazole	316 > 165; 247	DEET	192 > 91; 119
Iprodion	331 > 246	1H-Benzotriazole	120 > 65; 92
Ioxynil	370 > 127; 215	1-Methyl-benzotriazole	134 > 77; 106
Imidacloprid	254 > 153; 86	Benzothiazole	136 > 109; 65
Methabenzthiazuron	222 > 165; 150	2,4,5-T	255 > 197; 161
Tolylfluanid	347 > 137; 238		
Vinclozolin	316 > 284; 75		
2,4-D	219 > 161; 125		

2.2.10.1. Sample preparation

About 1 gram of lyophilized COMDIG was weighted in a 50 mL Sarstedt PP conical centrifuge tube, added with 100 µL of internal standard solution (PFOA $^{13}C_4$, PFOS $^{13}C_4$ and PFNA $^{13}C_5$, 1 mg/L in methanol) and 10 mL of pure methanol. The sample was mixed by vortex for 30 seconds and ultra-sonicated for 18 minutes. The supernatant was decanted into a second, clean 50 mL Sarstedt PP conical centrifuge tube. The original sample was extracted twice again and the supernatants added to the first extraction one. The 30 mL combined extract were evaporated to 10 mL volume using gentle stream of nitrogen at 35°C. 1 mL of the evaporated combined extract was transferred into 1.5 mL disposable polypropylene microcentrifuge tubes containing 25 mg of ENVI-Carb sorbent previously acidified with 50 µl of glacial acetic acid. The sample was mixed by vortex for 30 second and centrifuged at 6720 rcf for 30 minutes. 0.8 mL were then evaporated to 0.2 mL volume under gentle stream of nitrogen added of 0.2 mL of water and analysed by LC-MS/MS.

2.2.10.2. Criteria followed for quantification

The rationale behind the semi-quantitative determination of polar compounds in the "Non-target Screening" is based on the capability of Envicarb to adsorb compounds via dispersive interaction with π electrons. In case of chemicals containing no π electrons, there is no possibility for specific π – π interactions between the sorbent and analytes of interest. The purification of COMDIG material is due to the association of organic compounds showing any degree of aromaticity (π electrons). More aromatic compounds exhibit, obviously, a stronger association to Envi-carb, resulting in a loss of concentration in methanolic solution treated with the sorbent.

It has been demonstrated that the response of analytes in methanolic solution put in contact with acidified ENVI-Carb, in most cases, did not varied in considerable extent to affect the concentration calculation (data not shown).

For most of the compounds, the reported concentration was underestimated at maximum 2-5 times (so in the same order of magnitude). This error could be considered acceptable for a semi-quantitative screening method.

The criteria followed for analytes semi-quantitative determination are the following: two MRM transitions between the precursor ions and two most abundant fragment ions were monitored for almost every compound. The first one was used for quantification purposes, whereas the second one was to confirm the presence of the target compounds in the sample. In this way, the number of identification points (IPs) needed to confirm the detection of target analytes, according to the EU Regulations (4 IP, 1 for precursor ion and 1.5 for each transition product) was reached [17]. Besides the monitoring of MRM transitions, other identification criteria were used for quantification:

- LC retention time of the compound in the standard compared to those obtained in the samples. Retention time in the sample must be within ± 2% the retention time of the analyte in the analytical standard.
- The relative abundance of the two selected MRM transitions in the sample must be within ± 20% of the ratio obtained in the analytical standard.

2.2.11. Physical Impurities

A bleach washing method was applied for impurities determination in COMDIG samples.

After drying the COMDIG material was bleach washed on a 2 mm sieve. The fraction >2 mm was dried and the fractions of coarse stones (>5 mm) and plastics (>20 mm) and differentiated impurities (>2 mm) were determined.

3. Results and Discussion of FATE COMES

EU legislation with specific organic pollutant limit values for COMDIG materials currently does not exist.

At Member State level, substantial national and regional legislation can be found that is directly or indirectly destined at regulating organic pollutant limits in compost and digestate.

Table 25 gives an overview of legally binding limits and guide values for organic pollutants in COMDIG and similar materials in different European countries.

Table 25: Limits and guide values for organic pollutants in compost and digestate materials in different EU countries

	AT **(a)**	**BE (Fl)** **(b)**	**BE** **(Wal; digestate)** **(c)**	**DE** **(d)**	**DK** **(e)**	**FR** **(compost)** **(f)**	**LU** **(g)**	**SI** **(h)**	**CH** **(i)**
PAH (mg/kg dm)	6 (sum for 6 congeners**)	Individual limits for 10 congeners	5 (PAH_{16})		3 (sum for 11 congeners***)	Individual limits for 3 congeners	10* (PAH_{16})	3	4* (PAH_{16})
PCB (mg/kg dm)	0.2 (PCB_6)	0.8 (PCB_7)	0.15 (PCB_7)		0.08* (PCB_7)	0.8 (PCB_7; only for sewage sludge compost)	0.1* (PCB_6)	0.4 (1st class) 1 (2nd class) (PCB_6)	
PCDD/F (ng I-TEQ /kg dm)	20		100				20*		20*
PFC (mg/kg dm)	0.1			0.1					
AOX (mg/kg dm)	500		250						
LAS (mg/kg dm)			1500*		1300				
NPE (mg/kg dm)			25*		10				
DEHP (mg/kg dm)			50*		50				

a) Düngemittelverordnung; b) VLAREA Regulation c) AGW du 14/06/2001 favorisant la valorisation de certains déchets d) Düngemittelverordnung e) Slambekendtgørelsen f) NFU 44-051 and NFU 44-095 g) Guidance value h) Official Gazette of the Republic of Slovenia, no. 62/08 i) Guidance value from ChemRRV 814.81.

In the present chapter, the graphical representation of analytical results is reported.

The results are displayed as cumulative graphs scaled from 0 to 100% of the total sample population for a material type, with every concentration data point representing an actual sample measurement. This representation helps visualizing the spread on the data and allows checking how many samples of a COMDIG type surpass a certain threshold concentration.

Data relative to categories BW Co, GW Co, SS Co, MBT Co, BW Di, Man BW Di, Man Ecr Di and MBT Di are represented in the graphs, while category "Other" is not represented. The reason for this exclusion is that it does not really belong to the category of compost and digestate from biowaste.

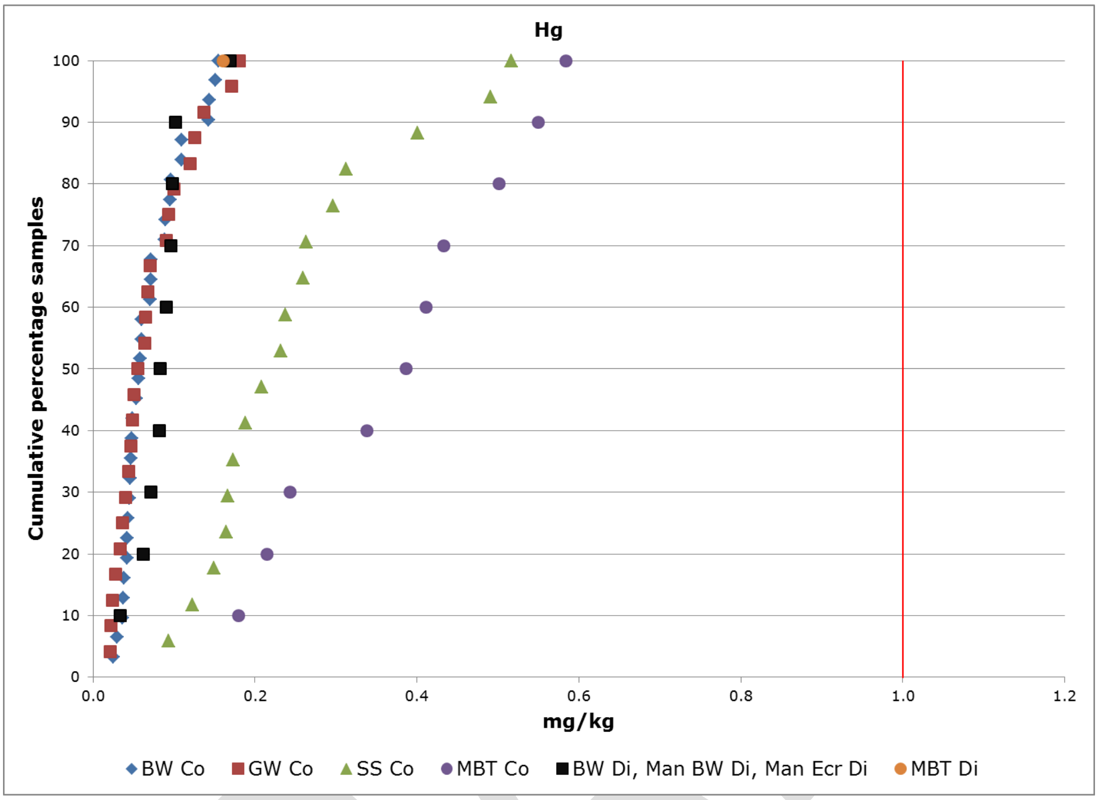
All data are expressed on dry matter (d.m.) basis unless indicated otherwise.

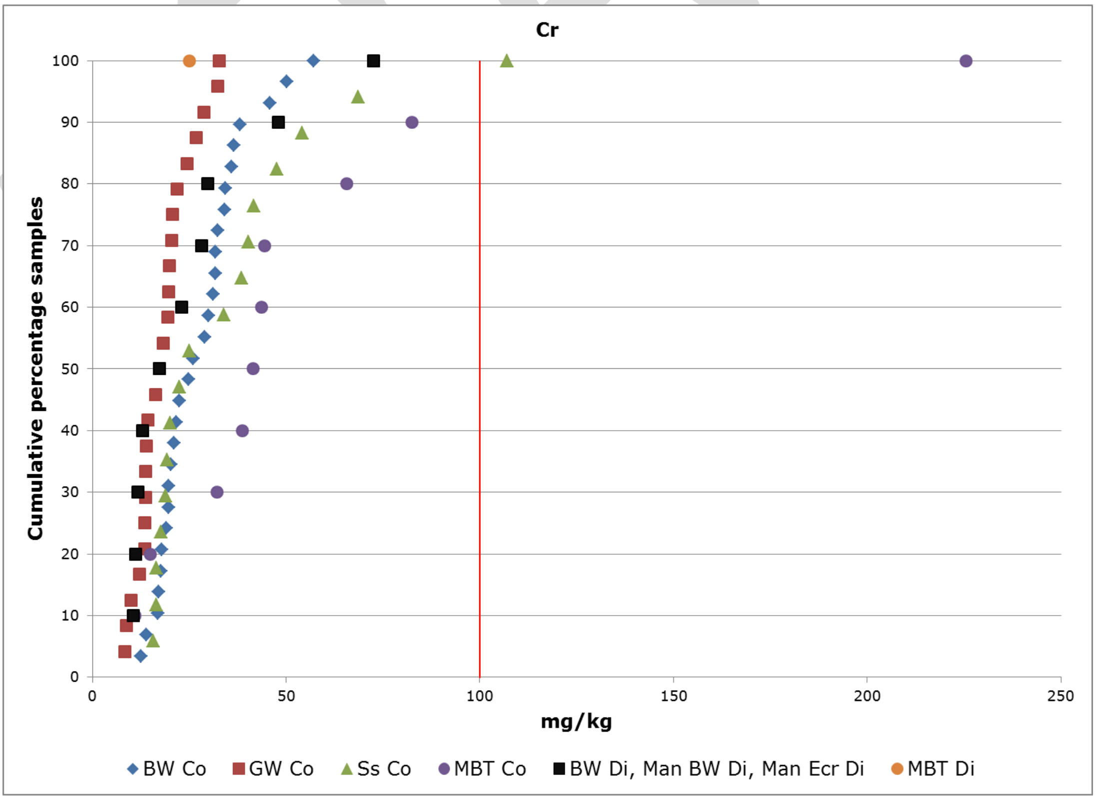
Supplementary information (individual analytical data and/or descriptive statistics) are included in Annex 1.

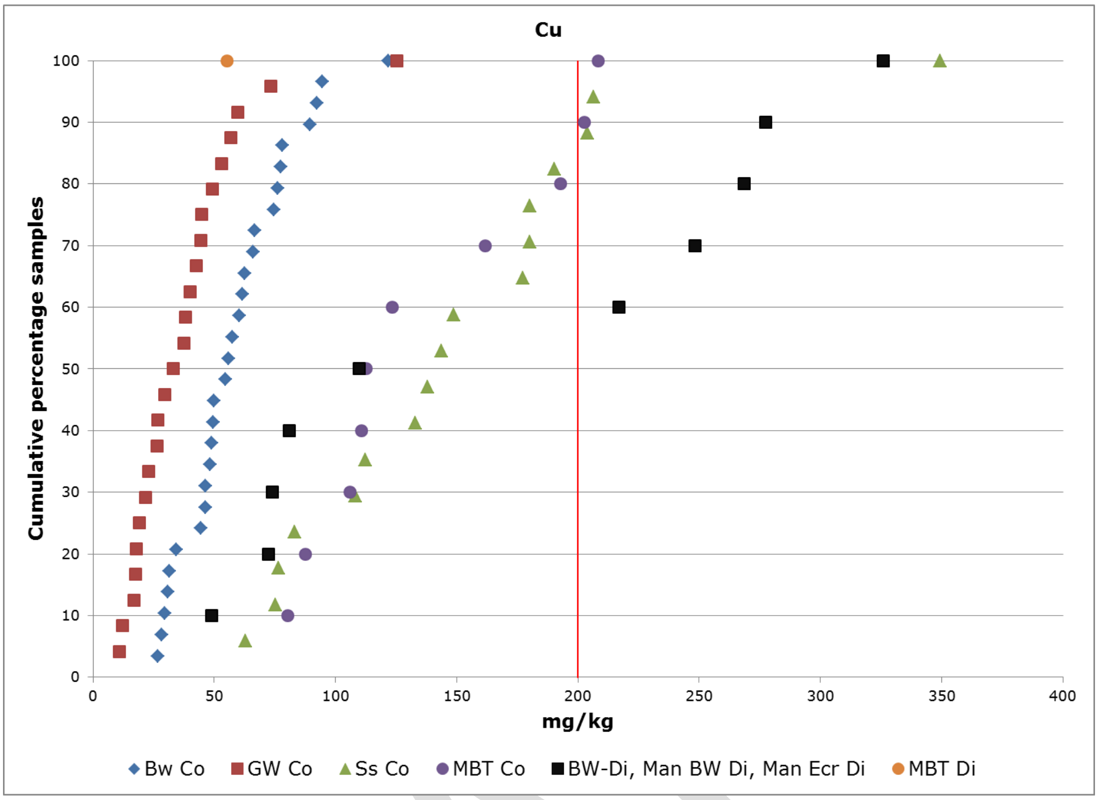
3.1. Heavy metals

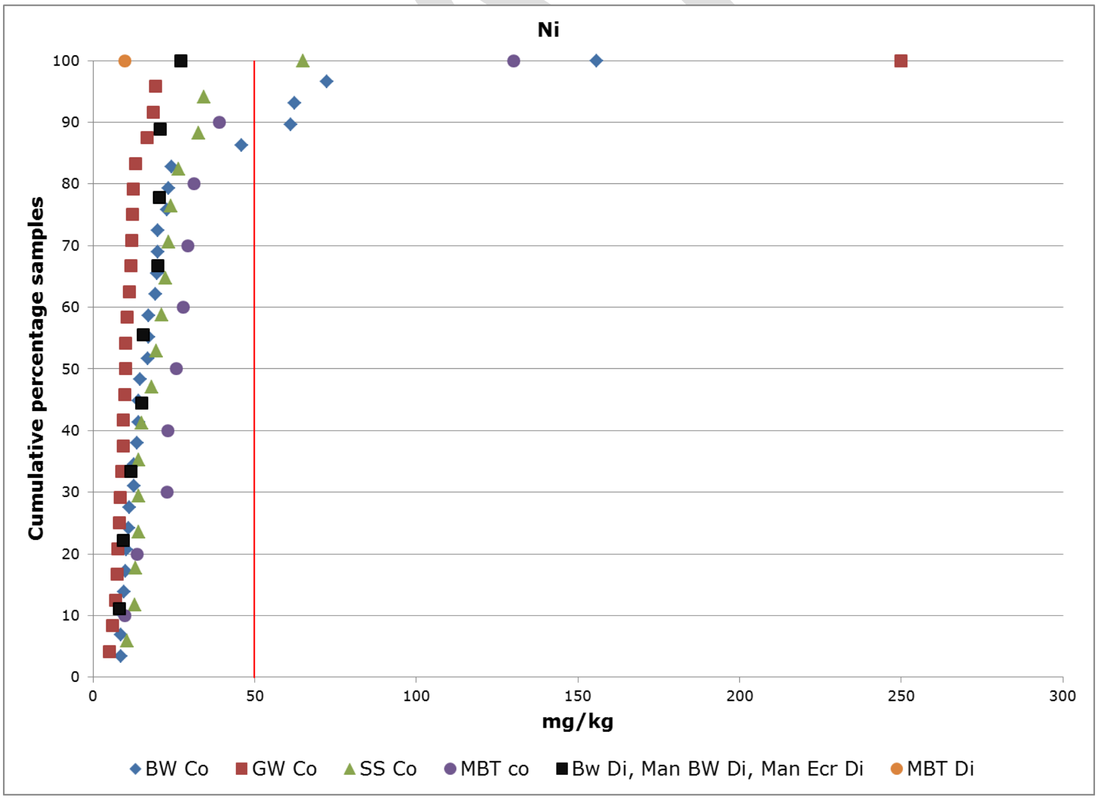
The results for heavy metals analysis are reported in Annex 1, and depicted in Figures 2.

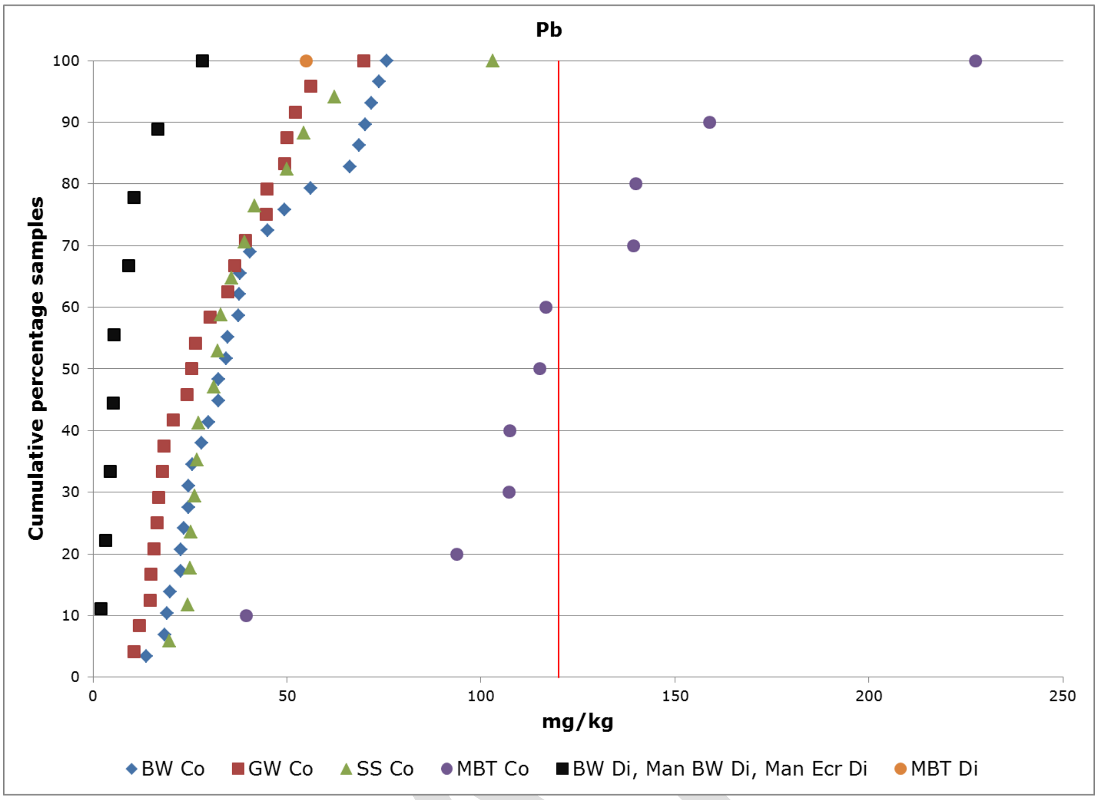
Concentration in COMDIG materials ranged from 0.01 to 1.28 mg/kg for Hg, from 3.01 to 225.5 mg/kg for Cr, from 0.13 to 487.74 mg/kg for Cu, from 3.04 to 244.99 mg/kg for Ni, from 1.07 to 270.10 mg/kg for Pb, from 1.06 to 1304.87 mg/kg for Zn and from 0.04 to 2.77 mg/kg for Cd.

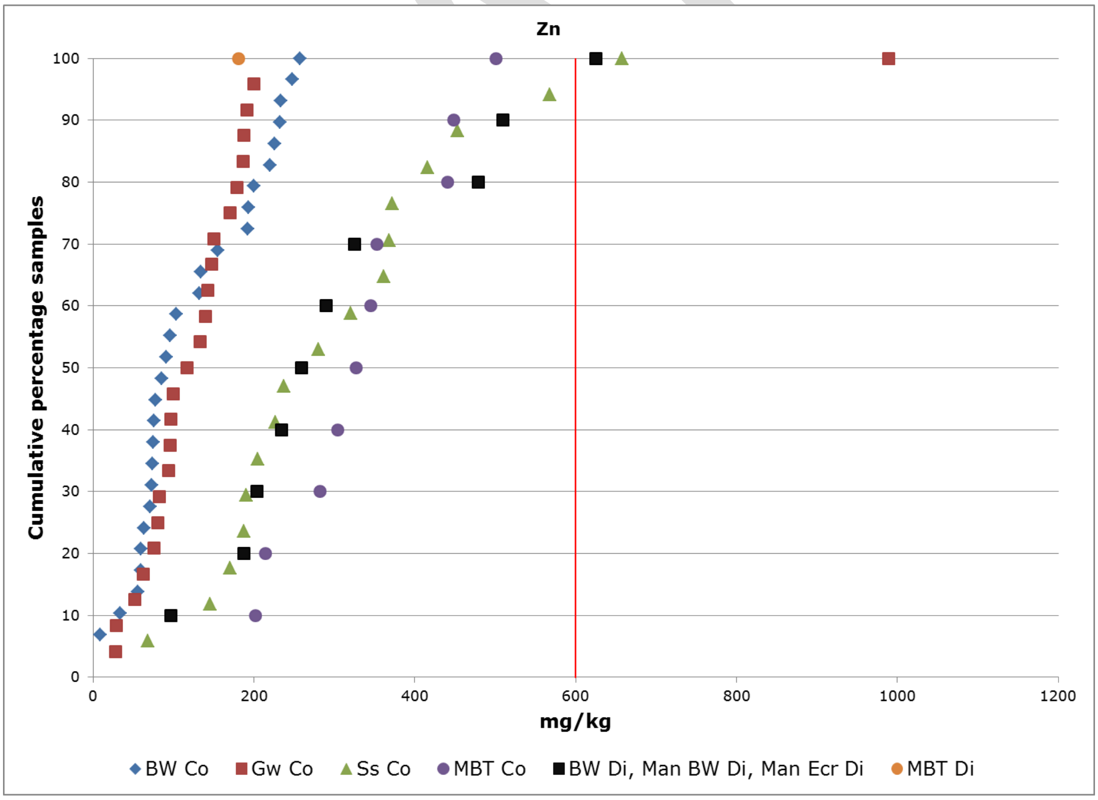





Cu
Cumulative percentage samples
0
10
20
30
40
50
60
70
80
90
100
0
50
100
150
200
250
300
350
400
mg/kg
Bw Co
GW Co
Ss Co
MBT Co
BW-Di, Man BW Di, Man Ecr Di
MBT Di


Ni
Cumulative percentage samples
0
10
20
30
40
50
60
70
80
90
100
0
50
100
150
200
250
300
mg/kg
BW Co
GW Co
SS Co
MBT co
Bw Di, Man BW Di, Man Ecr Di
MBT Di


Pb
Cumulative percentage samples
0
10
20
30
40
50
60
70
80
90
100
0
50
100
150
200
250
mg/kg
◆ BW Co
■ GW Co
▲ SS Co
● MBT Co
■ BW Di, Man BW Di, Man Ecr Di
● MBT Di


Zn
Cumulative percentage samples
0
10
20
30
40
50
60
70
80
90
100
0
200
400
600
800
1000
1200
mg/kg
◆ BW Co
■ Gw Co
▲ Ss Co
● MBT Co
■ BW Di, Man BW Di, Man Ecr Di
● MBT Di

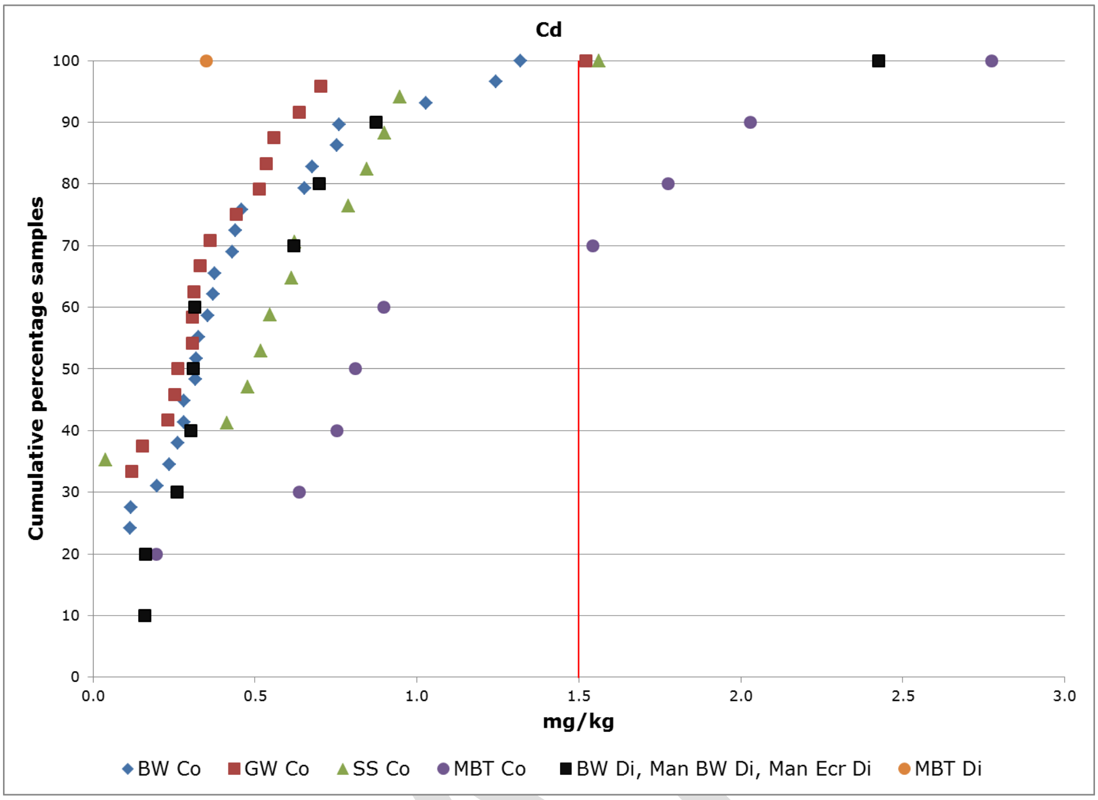


Figure 2: Cumulative percentage graph for some selected heavy metals in different kind of COMDIG samples The red bar represent the proposed Eu End-of-Waste limit value (Co=compost, Di=digestate, BW=source separated biowaste and green waste; GW=source separated green waste; SS=sewage sludge; MBT=mechanical biological treatment; Man=manure; Ecr=energy crops).

From the subgraphs, the following can be concluded:

- **Hg**: all samples, meet the proposed limit of 1 mg/kg dry matter. Sewage sludge compost and MBT compost clearly display generally higher Hg concentrations than COMDIG materials from source separation.
- **Cr**: nearly all samples meet the proposed limit of 100 mg/kg dry matter, except one sewage sludge compost sample and one MBT compost sample.
- **Cu**: compost from source separated biowaste or green waste generally meets the proposed limit value of 100 mg/kg dry matter, except for two samples (1 in each category). Sewage sludge compost, MBT compost and digestate hardly meet the proposed limit values.
- **Ni**: most samples meet the proposed 50 mg/kg dry matter limit value, except 4 separately collected biowaste compost samples, 1 green waste compost sample, 1 sewage sludge compost sample and 1 MBT compost sample.
- **Pb**: nearly all samples meet the proposed limit of 120 mg/kg dry matter, except 4 MBT compost samples.
- **Zn**: compost from source separated biowaste or green waste generally meets the proposed limit value of 600 mg/kg dry matter, except for one green waste compost sample, one sewage sludge compost sample and one digestate sample.
- **Cd**: most samples meet the proposed 1.5 mg/kg dry matter limit value, except one green waste compost sample, one sewage sludge compost sample, four MBT compost samples and one digestate sample.

Furthermore, it can be derived that:

- compost produced from source separated collection (biowaste and green waste) nearly always meets the proposed limit values. At the same time, the few exceeding values also demonstrate that analysis of the output material is necessary to avoid possible problems related to e.g. contaminated input materials;

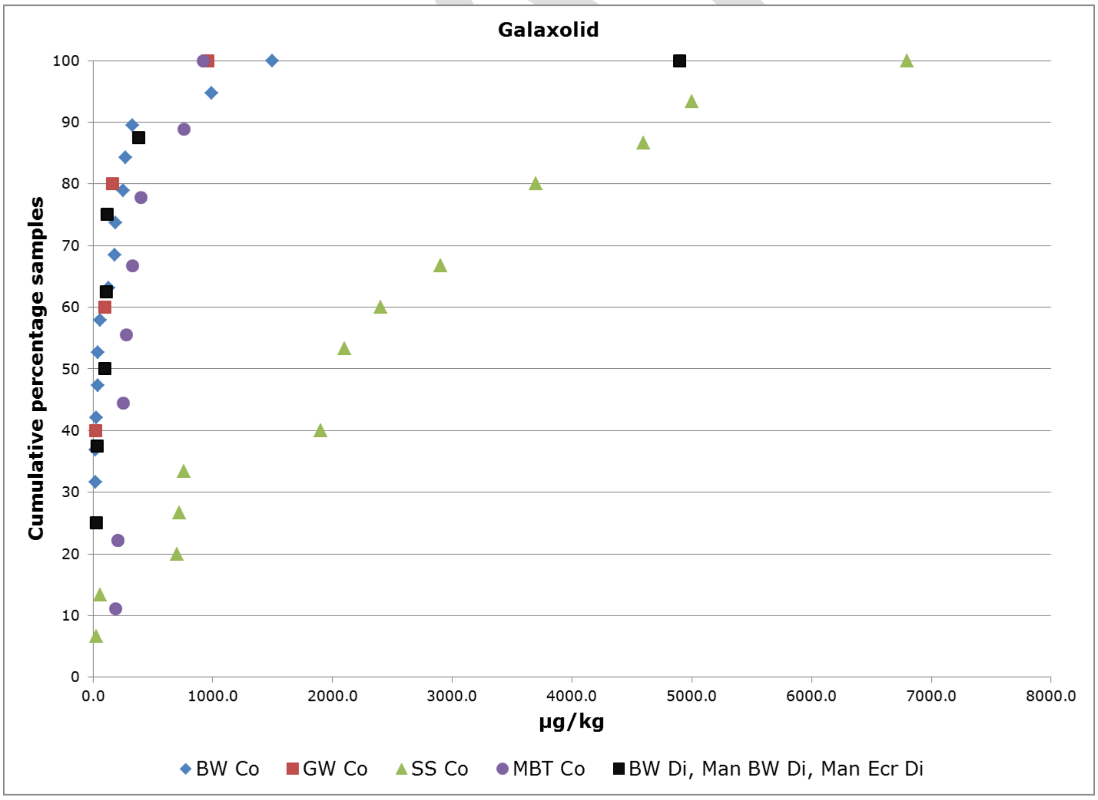
- sewage sludge compost generally meets the proposed limit values for Hg, Cr, Pb, Cd, Zn and Ni (with sporadic exceedings) but tends to have problems in meeting the proposed Cu limits;
- MBT compost generally meets the proposed limit values for Hg, Cr, Ni and Zn (with some sporadic exceedings) but tends to have problems in meeting the proposed limit values for Cu, Pb and Cd;
- digestate generally meets the proposed limit values for Hg, Cr, Pb, Cd, Zn and Ni (with sporadic exceedings) but tends to have problems in meeting the proposed Cu limit;
- there are not enough samples to make a sound judgement on MBT digestate, but the 2 samples analysed met all proposed limit values.

3.2. Polycyclic Musk Compounds

The results of PCM analysis are reported in Annex 1, and depicted in Figures 3.

The Figure 3 displays the distribution of concentration of galaxolid and tonalid per each category of COMDIG samples. The highest concentration encountered in any sample was 6.8 mg/kg for galaxolid and 0.95 mg/kg for tonalid.

No legal limits were found for these compounds in COMDIG materials at Member State level. There has been a proposal in Germany in 2006 to establish a limit of 10 or 15 mg/kg for these compounds in sewage sludge, but this has not been adopted in the end. In any case, the current study shows that the encountered concentrations are well below these suggested limit values. Therefore, it can be stated that these compounds are likely to be of very low concern for compost/digestate quality.



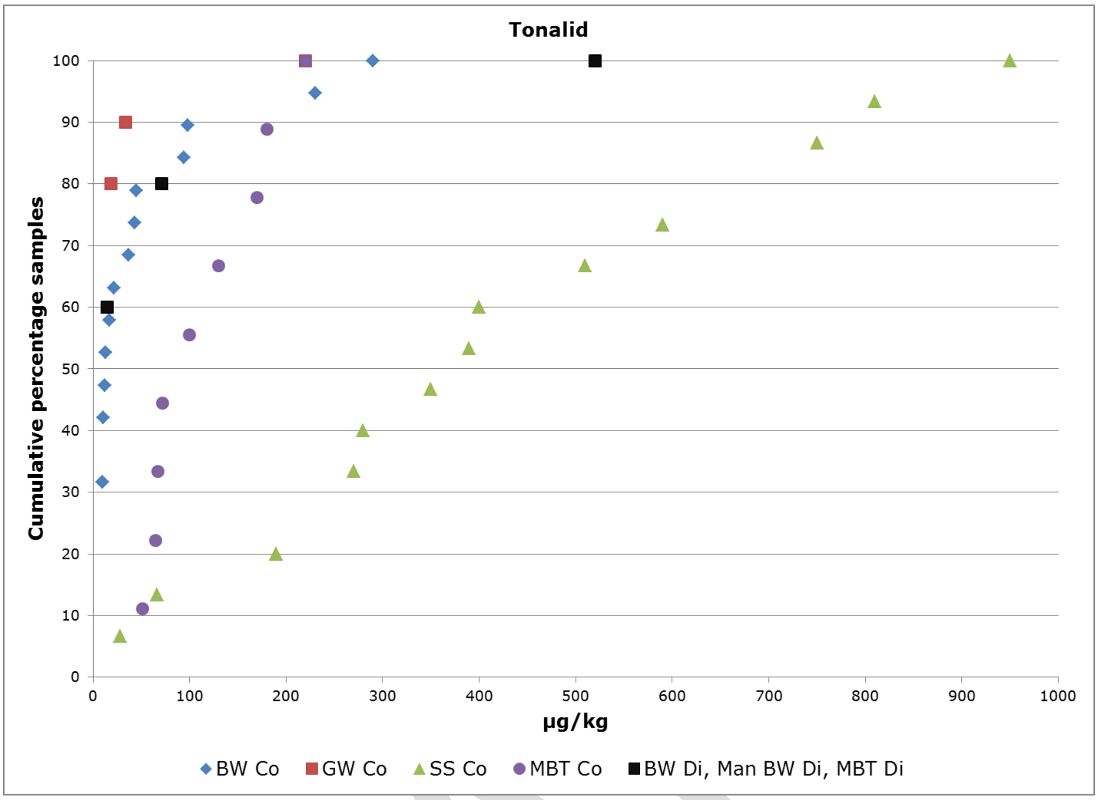


Figure 3: Cumulative percentage graphs for galaxolid and tonalid in different kind of compost. (Co=compost, Di=digestate, BW=source separated biowaste and green waste; GW=source separated green waste; SS=sewage sludge; MBT=mechanical biological treatment; Man=manure; Ecr=energy crops).

3.3. Siloxanes

The results for siloxanes analysis are reported in Annex 1, .
Concentrations in COMDIG materials ranged from 75 to 880 µg/kg for D4, from 110 to 1500 µg/kg for D5, from 8 to 20 for MD3M and from 240 to 1700 µg/kg for D6.

3.4. PAHs

The results for PAHs are reported in Annex 1, and depicted in Figure 4.

The figure 4 displays the distribution of the sum of 12 measured PAHs in different kind of analyzed compost.

12 of the 16 US EPA PAH compounds were measured (phenanthrene, anthracene, fluoranthene, pyrene, benzo[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, indeno[1,2,3- cd]pyrene, dibenzo[a,h]anthracene and benzo[ghi]perylene).

Naphthalene, acenaphtylene, acenaphtene and fluorene were not measured because of their high volatility therefore might have been lost through lyophilisation of the samples.

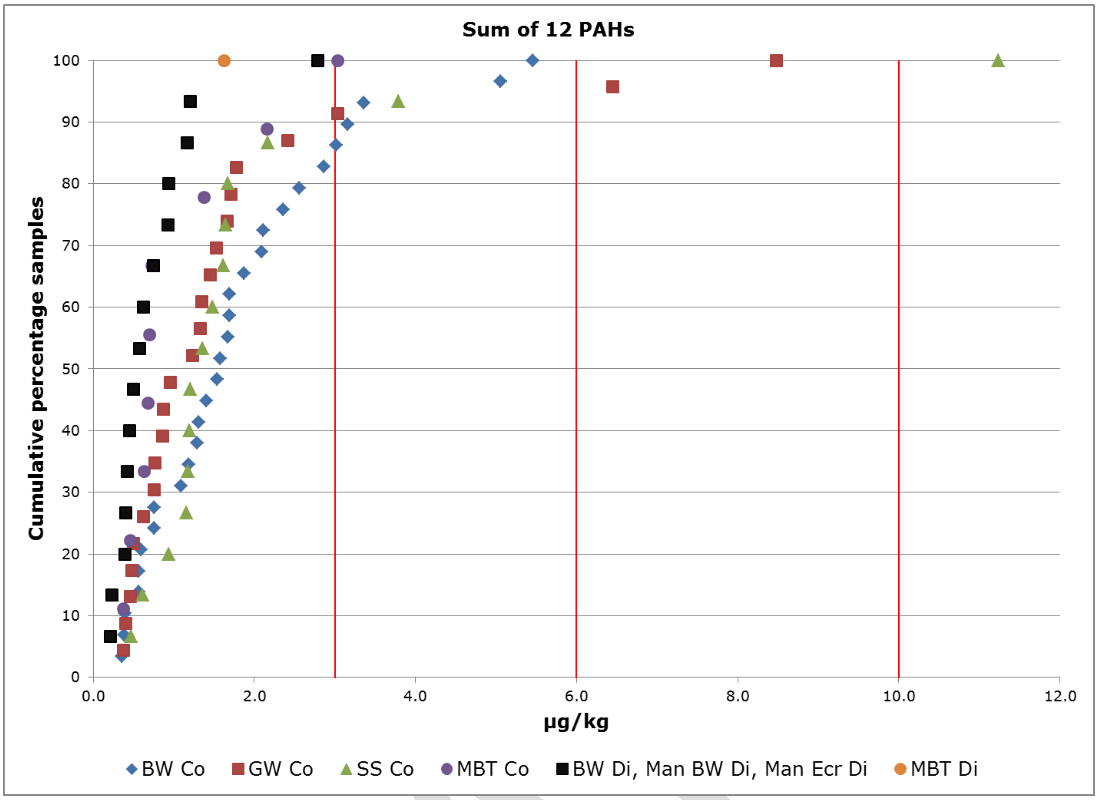


Figure 4: Cumulative percentage graph for the sum of 12 measured PAHs in different kind of COMDIG samples. The red bars represent existing limit values in European countries for similar materials. (Co=compost, Di=digestate, BW=source separated biowaste and green waste; GW=source separated green waste; SS=sewage sludge; MBT=mechanical biological treatment; Man=manure; Ecr=energy crops).

Most samples had relatively low PAHs values and in details:

- COMDIG samples produced from source separated collection (biowaste and green waste) exceed the stricter limit values of 3 µg/kg in only four and three cases, respectively.
- sewage sludge compost exceed the limits values in two samples.

3.5. AhR-active compounds

The AhR-active compounds bioassay gave a toxicity response that is induced through the binding of dioxins and dioxin like compounds to the aryl hydrocarbon receptor.

The results of AhR-active compounds analysis are reported in Annex 1, and depicted in Figures 5.

TEQbio values for crude (whole) extracts (all 98 samples) ranged from 0.05 ng TCDD-equivalents per g.d.w. up to 12.72 ng TEQbio/g.

For selected 20 compost samples both whole extracts and H_2SO_4-treated extracts were tested. After removal of less stable compounds (mostly PAHs) from the extract, the response in bioassay was only minor. At eight of the H_2SO_4-treated samples weak activation of AhR was observed, and it was quantifiable at two samples around the detection limit of the method (i.e. 0,007 and 0,01 ng TEQbio/g).

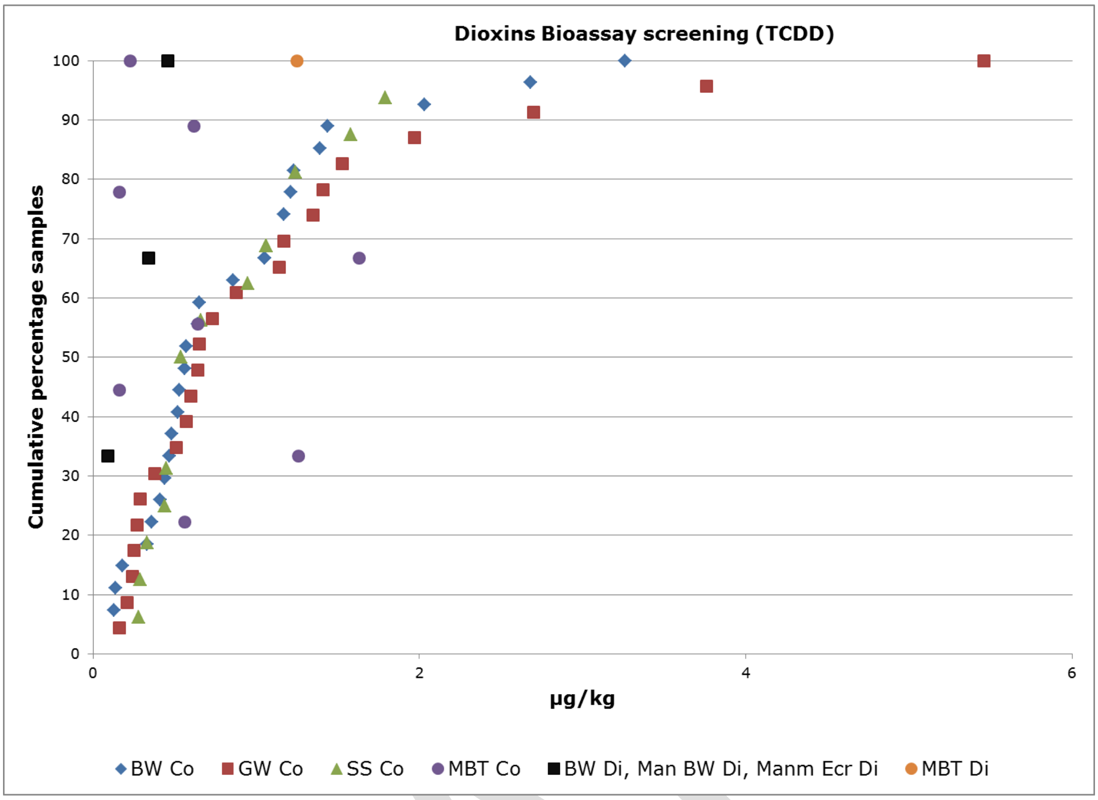


Figure 5: Cumulative percentage graph for of AhR-active compounds in different kind of COMDIG material. (Co=compost, Di=digestate, BW=source separated biowaste and green waste; GW=source separated green waste; SS=sewage sludge; MBT=mechanical biological treatment; Man=manure; Ecr=energy crops).

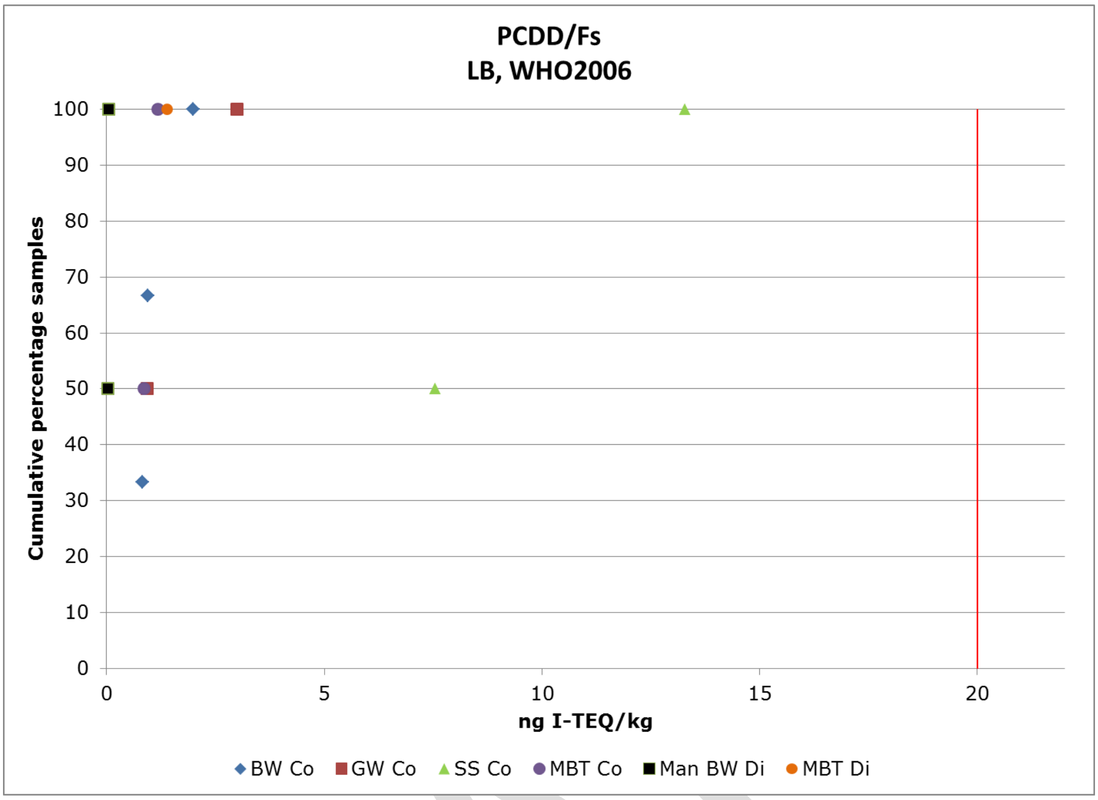
The Figure 5 put in evidence a similar trend for the bio-assay dioxin response as for the PAHs measurements displayed in Figure 4. These results indicate that dioxin-like effects observed could be mostly related to less persistent compounds, such as PAHs, present in the original whole extract but removed in the treated samples.

3.6. PCDD/Fs and PCBs chemical analysis of selected samples following AhR-active compound bioassay

Following the results obtained from AhR-active compound bioassay measurements, samples in each category exhibiting high TEQ values were subject to further chemical analysis on PCDD/Fs and PCBs.

In total 20 samples were selected: four BW Co, three GW Co, three SS Co, three MBT Co, three Man BW Di, two MBT Di and two belonging to the category "Other".

The PCDD/F analysis results are reported in Annex 1, **Error! Reference source not found.** and are given in Fig 6 as both lower and upper bound values, with actual values being between these two limits.



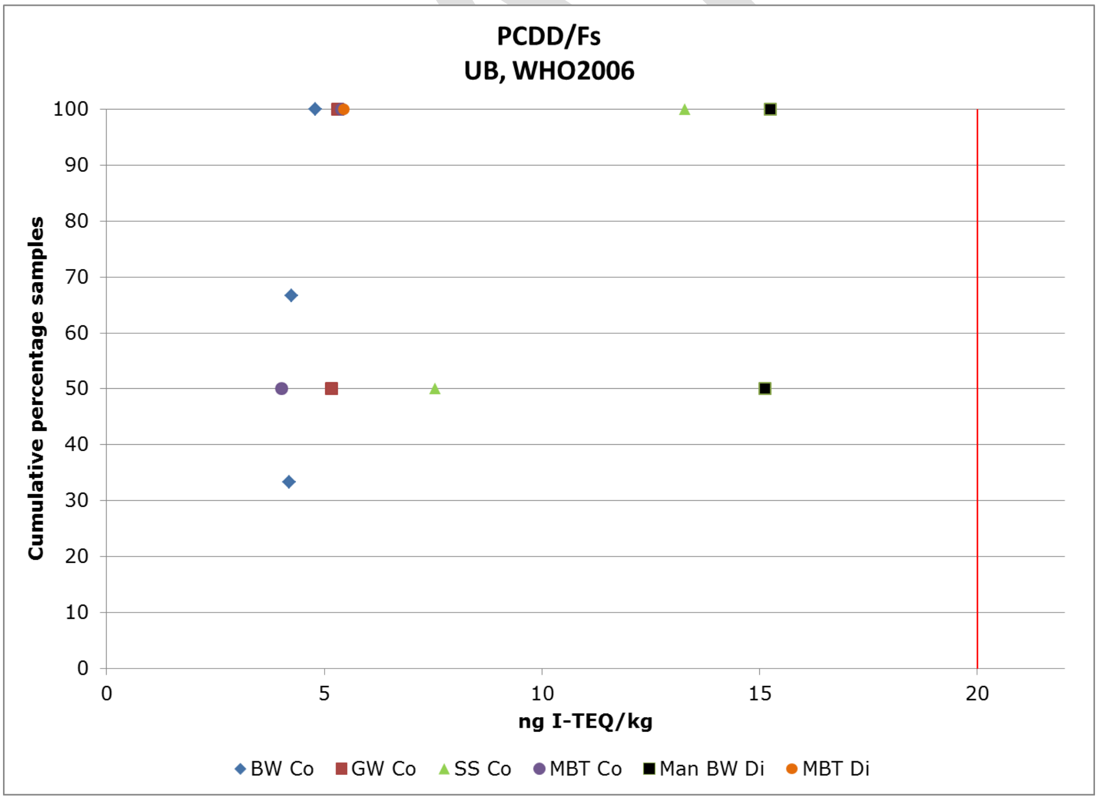


Figure 6: Cumulative percentage graph for of PCDD/Fs in selected COMDIG samples. Data represents lower bound (LB) and upper bound (UP) values. The red bar represents an existing limit value in different European countries. (Co=compost, Di=digestate, BW=source

separated biowaste and green waste; GW=source separated green waste; SS=sewage sludge; MBT=mechanical biological treatment; Man=manure; Ecr=energy crops).

The results generally indicate low to medium toxicity equivalents for all samples, with no upper bound value exceeding the strictest existing MS limit of 20 ng I-TEQ/ kg dm.

Again, no clear distinctions can be made between categories, especially when taking into account both the lower and upper bound levels.

The PCBs analysis results reported in Annex 1, **Error! Reference source not found.** and are depicted in Fig 7.

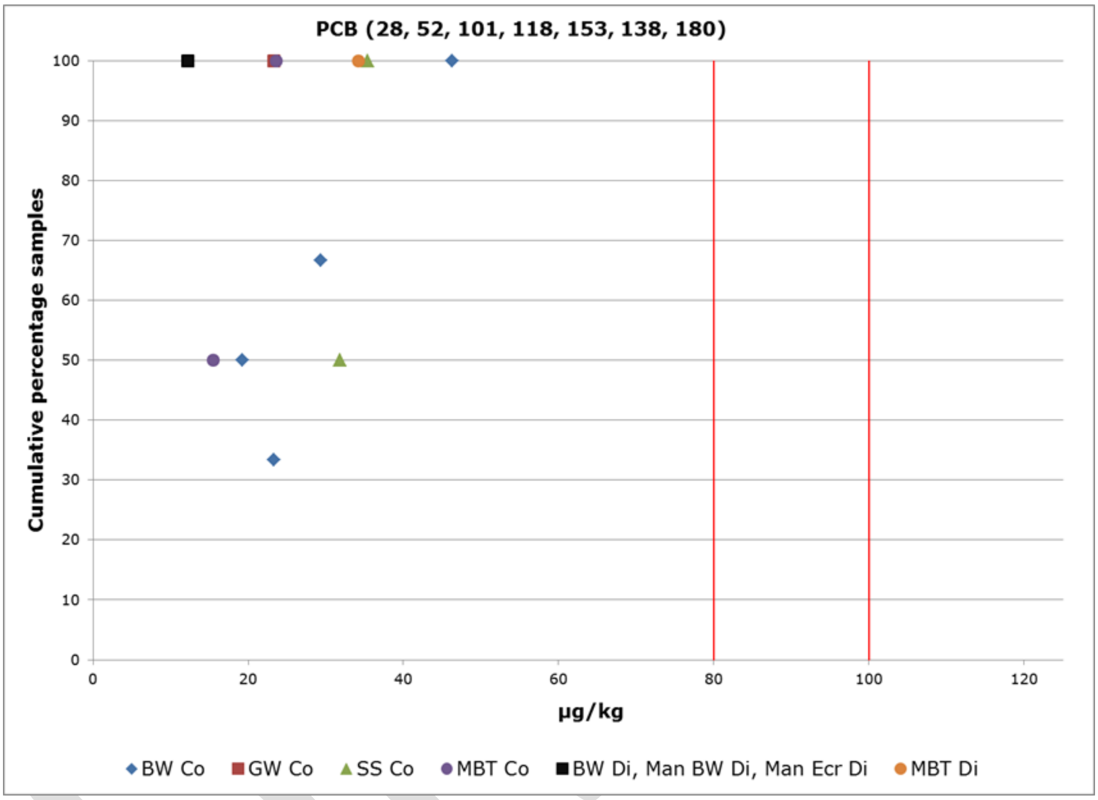


Figure 7: Cumulative percentage graph for sum of 7 PCBs (28, 52, 101, 118, 138, 153 and 180) in selected COMDIG samples. The red bar represents an existing limit value in different European countries. (Co=compost, Di=digestate, BW=source separated biowaste and green waste; GW=source separated green waste; SS=sewage sludge; MBT=mechanical biological treatment; Man=manure; Ecr=energy crops).

The COMDIG samples exhibit generally low PCB levels. None of the samples exceeded any of the existing national limits or guide values. Again, no clear distinctions can be made between the categories.

3.7. PCDD/Fs, PCBs and PBDEs measured by HRGC-HRMS in the framework of comparative sampling

3.7.1. PCDD/Fs measured by HRGC-HRMS

JRC-IES analysed samples for dioxins and furans in the framework of the comparative sampling exercise. The results, expressed as I-TEQs, are reported in Annex 1, .

3.7.2. EC-6PCBs and DL-PCBs measured by HRC-HRMS

The results for EC-6PCBs and DL-PCBs, expressed as I-TEQs, are reported in Annex 1, .

3.7.3. Indicators-PCBs (sum of PCB-28, PCB-52, PCB-101, PCB-138, PCB-153 and PCB-180) measured by HRC-HRMS

The results for Indicators-PCBs, expressed as I-TEQs, are reported in Annex 1, .

3.7.4. PBDEs

The results for PDBE analysis by HRGC-HRMS are reported in Annex 1, .

Furthermore, a total of 34 samples over all categories were selected and used to produce a pool sample for every category. This yielded 9 pool samples made up of 1 to 5 individual subsamples. Analytical results are reported in Annex 1, .

Pooled compost GW Co, SS Co and MBT Co were analysed in duplicate in order to evaluate the pool homogeneity (data not shown). Even considering the variability of the results, samples concentrations were always in the same order of magnitude. This showed that the same kind of compost coming from different plant has the same content of contamination.

3.8. Perfluoralkyl substances

The results for PFASs are reported in Annex 1, and are depicted in Figure 8 (as the sum of PFOA and PFOS).

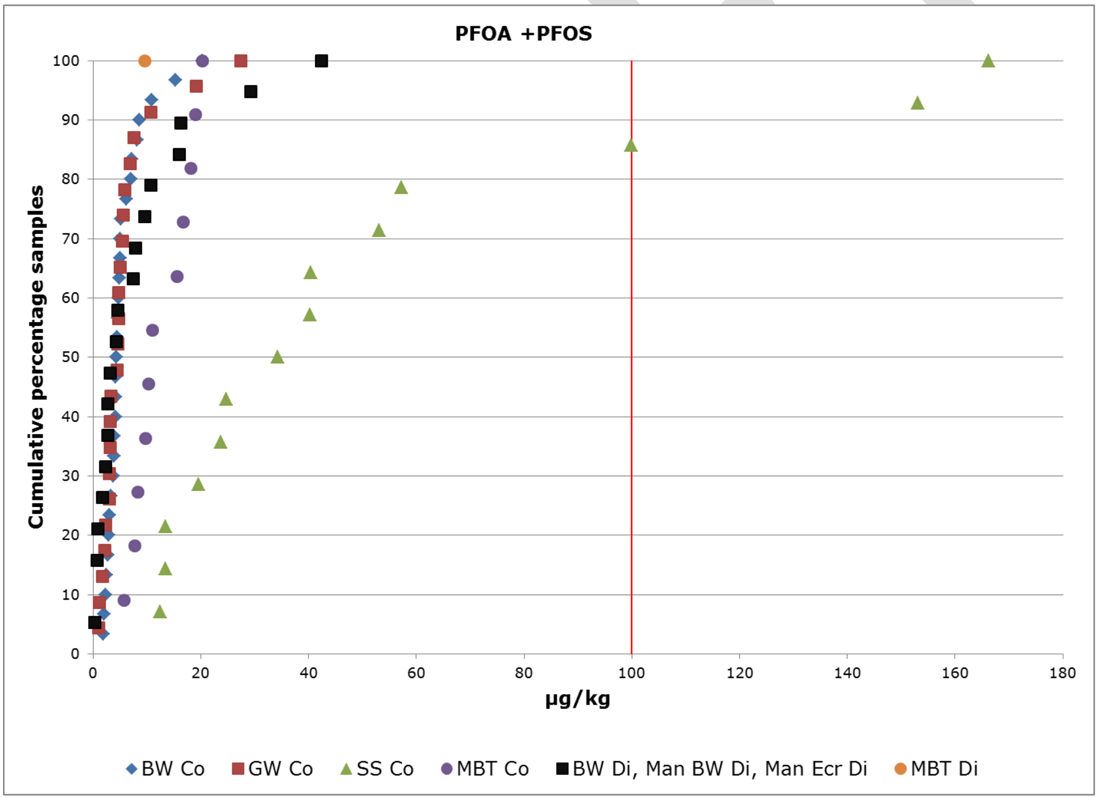


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Figure 8: Cumulative percentage graph for the sum of PFOA and PFOS concentrations in different kind of COMDIG materials. The red bars represent existing limit values in different European countries for similar materials. *(Co=compost, Di=digestate, BW=source separated biowaste and green waste; GW=source separated green waste; SS=sewage sludge; MBT=mechanical biological treatment; Man=manure; Ecr=energy crops).*

The measured concentrations ranged from 0.05 to 6.92 ng/g for PFNA, from 0.3 to 25.86 for PFOA and from 0.035 to 145.66 ng/g for PFOS.

Additional analytical data for PFDA, PFHpA and PFBS and descriptive statistics are reported in Annex 1, .

3.9. Comparative sampling exercise

In order to guarantee maximal objectivity, minimal variation and the smallest possible bias upon sampling an unannounced control sampling was performed by a single team composed of EC JRC staff only, at selected plants participating in the collaborative screening exercise.

A number of compost/digestate producing plants were selected from the list of participating producers, and visited during the last week of June 2011.

The JRC team took their own samples for measurement by JRC-IES. Nineteen different samples were taken during the sampling campaign, in Italy, France, Belgium, The Netherlands and Germany, belonging to the following categories: BW Co, GW Co, SS Co, MBT Co and BW Di.

A T-test at 95% confidence level did not indicate a significant difference between the data originating from the JRC samples and the plant samples (Annex 1, **Error! Reference source not found.**).

3.10. Non target screening

3.10.1. Pesticides

In the non-target screening of 78 compost samples belonging to different categories, seven pesticides were semi-quantitative determined.

The individual concentration of 2,4-D, Dichlorprop, Mecoprop, MCPA, 2,4,5-T, Bentazone and Imidacloprid in different kind of COMDIG material is reported in Annex 1, and the distribution of the sum of their concentration values is depicted in Figure 9.

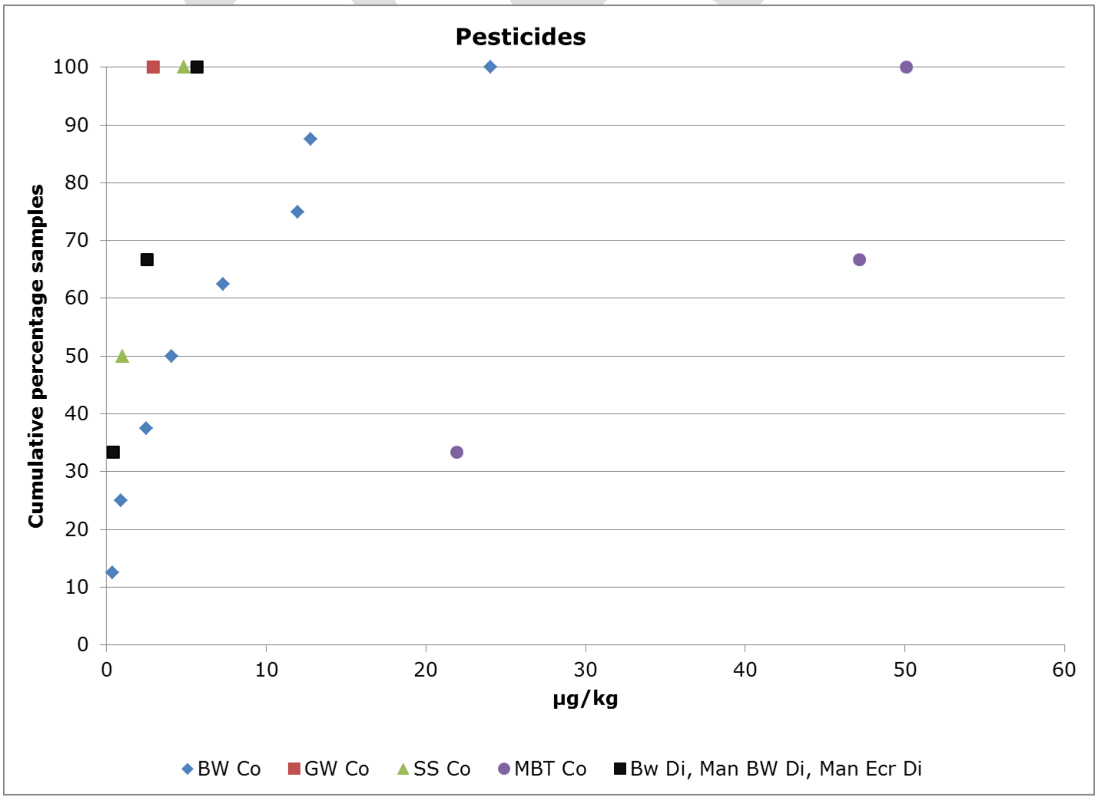

Figure 9: Cumulative percentage graph for the sum of 2,4-D, Dichlorprop, Mecoprop, MCPA, 2,4,5-T, Bentazone and Imidacloprid in different kind of COMDIG materials. (Co=compost, Di=digestate, BW=source separated biowaste and green waste; GW=source separated green waste; SS=sewage sludge; MBT=mechanical biological treatment; Man=manure; Ecr=energy crops).

The sum of the concentration values of these seven pesticides was in all type of compost lower than 50.1 ng/g.

3.10.2. Pharmaceuticals

In the non-target screening of 78 compost samples belonging to different categories, several pharmaceuticals were semi-quantitatively determined.
In detail:

- Diclofenac was semi-quantitative determined in 12 samples out of 78 (compost BW Co, C, D, E+F+G and I) with concentration values ranging from 6.58 to 782.8 ng/g.
- Ibuprofen was semi-quantitative determined in 17 samples out of 78 (compost types A, B, C, D, E+F+G and I) with concentration values ranging from 0.78 to 4275.4 ng/g.
- Ketoprofen was semi-quantitative determined in 4 samples out of 78 (compost types A, D and J) with concentration values ranging from 4.40 to 238.45 ng/g.
- Acetylsalicylic acid was semi-quantitative determined in 29 samples out of 78 (all compost types except type H) with concentration values ranging from 0.62 to 1178.7 ng/g.
- Naproxen was semi-quantitative determined in 7 samples out of 78 (compost types C, D, E+F+G and I) with concentration values ranging from 4.31 to 327.65 ng/g.
- Bezafibrate was semi-quantitative determined in 22 samples out of 78 (all compost types except H and J) with concentration values ranging from 0.06 to 80.86 ng/g.
- Gemfibrozil was semi-quantitative determined in 3 samples out of 78 (compost types D and E+F+G) with concentration values ranging from 20.09 to 130.86 ng/g.
- Cloramphenicol was semi-quantitative determined in only 1 sample out of 78 (MBT Co) with concentration of 1.11 ng/g.
- Clofibric acid was semi-quantitative determined in only 1 sample out of 78 (BW Di + Man Bw di + Man Ecr Di) with concentration of 3.89 ng/g.

3.10.3. Sweeteners

In the non-target screening of 78 compost samples belonging to different categories, sweeteners (saccharin, acesulfame and sucralose) were semi-quantitatively determined.
In particular:

- Saccharin was semi-quantitative determined in 41 samples out of 78 (all compost types except type J and in only one sample of types H and K, respectively) with concentration values ranging from 0.126 to 107.4 ng/g. The distribution of saccharin concentration in different compost types is depicted in Figure 10.
- Acesulfame was semi-quantitative determined in 20 samples out of 78 (all compost types except type H) with concentration values ranging from 0.05 to 125.5 ng/g.
- Sucralose was semi-quantitative determined in only 1 samples out of 78 (compost MBT Co) with concentration value of 0.632 ng/g.

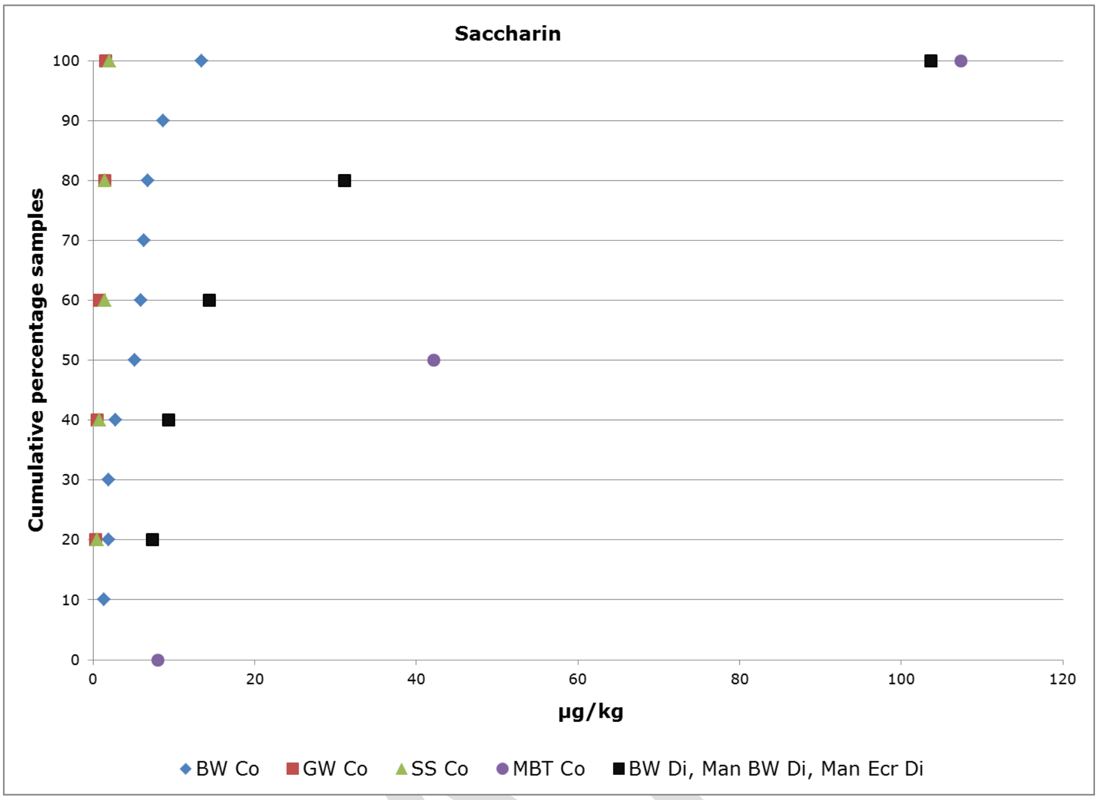

Figure 10: Cumulative percentage graph for saccharin in different kind of COMDIG material. (Co=compost, Di=digestate, BW=source separated biowaste and green waste; GW=source separated green waste; SS=sewage sludge; MBT=mechanical biological treatment; Man=manure; Ecr=energy crops).

The descriptive statistics for sweeteners is reported in Annex 1, .

3.11. Phenols

A screening was done on 29 compost samples throughout nitrophenol, phenol, all categories for 2,4,6-trichlorophenol, pentachlorophenol, 2-chlorophenol, 2,4-dichlorophenol, 2,4,5-trichlorophenol and 2,3,4,6-tetrachlorophenol, octylphenol, nonylphenol and bisphenol A.
The highest concentration encountered was 0.08 mg/kg.

3.12. Physical impurities

For organizational reason only 16 samples could be analysed for physical impurities. The results are reported in Annex 1, .

4. Conclusions

The results from the JRC Sampling and Analysis Campaign presented in this report provide many new insights.

Overall, the results indicate that:

- No single technology provides an absolute barrier to inorganic or organic pollutants, so regular testing of certain pollutants is recommended for all types of materials.
- The use of source-separated bio-waste and green-waste material inputs tends to lead to better results for heavy metal concentrations than when mixed municipal waste or sewage sludge is used.
- MBT composts tend to have very high physical impurity levels, and a large majority of the MBT composts would fail the proposed end-of-waste physical impurities criteria.

- On average, all materials show comparable concentration levels of PAHs, PCBs, PCDD/Fs and PFASs, with the sole exception of sewage sludge compost that tends to have higher PFAS levels. For PAHs, existing national limit and guidance values appeared to most probably be exceeded in all material categories. Exceedences of existing national limit and guidance values of PFASs were limited to materials derived from sewage sludge, where they appeared quite probable. Other organic pollutants showed very low concentration levels in all the materials studied and/or are currently not widely considered as compounds of concern in Member States' national legislations.

However, it is important to note the following limitations:

- Participation in the campaign was on a voluntary basis, and therefore it cannot be excluded that other COMDIG installations produce materials with very different qualities to those sampled within the FATE-COMES framework.
- Due to the set up and time limitations of the campaign, temporal variations could not be considered, although the data seem to be confirmed by external studies that cover longer periods and therefore take into account seasonal variations and possible spikes of contamination. Moreover, Brändli et al. [18] report that the highest concentrations of persistent organic pollutants were observed in summer compost samples. So given that most FATE-COMES samples were acquired during the 2011 summer period, there appears to be no particular reason to assume that the organic pollutant measurements would systematically underrepresent actual POP concentrations in compost and digestate.
- Due to its limited size, the present dataset generally provides trend information rather than elucidate statistically significant differences between different COMDIG types.

In summary, following conclusions and recommendations regarding end-of-waste criteria for COMDIG can be derived from the scientific data presented in this report:

- End-of-waste product quality requirements should provide an additional safeguard against undesired pollutants that cannot be avoided or removed solely through the selection and processing of input material.
- When establishing end-of-waste criteria, testing requirements and limit values for heavy metals and physical impurities should be included for all COMDIG categories, as no technology or input material type can fully safeguard against the presence of heavy metals.
- When establishing end-of-waste criteria, testing requirements and limit values for certain organic pollutants should be included, especially for PAH (for all possible COMDIG materials) and PFAS (only if sewage sludge derived materials were to be allowed), as no technology or input material type provides a full safeguard against the presence of organic pollutants.

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Supplementary information

Non detectable (n.d) data were treated as <LoQ data. In the computation of statistic parameters, when no uncensored data were available for <LOQ and n.d., they were replaced with the most used estimated, LoD/2.

However, when more than 50% of data in the analyzed chemicals is <LoQ (or n.d.) only minimum and maximum values were listed in the following tables.

Table SI 1: Descriptive statistic for metals in COMDIG samples

Uncensored data ((ICP / AES true values) were used for statistical analyisis. However when negative data occurred, they were replaced with LoD/2 estimate.

Number of samples 105

BW Co	***Hg (mg/kg)***	***Ag (mg/kg)***	***Mg (%)***	***Cd (mg/kg)***	***K (%)***	***P (%)***	***Ba (mg/kg)***	***Cu (mg/kg)***	***Mn (mg/kg)***
average	0.07	0.34	0.37	0.39	0.97	0.74	125.63	57.46	309.74
std.dev	0.04	0.35	0.21	0.35	1.73	1.49	67.37	23.63	127.28
min	0.02	< DL	0.13	< DL	0.06	0.14	42.19	18.16	117.68
max	0.16	1.68	1.26	1.32	9.87	8.32	352.49	121.80	583.78
25° percentile	0.04	0.08	0.26	0.12	0.30	0.26	79.15	45.10	215.92
90° percentile	0.14	0.60	0.49	0.79	1.12	0.77	206.85	89.80	460.71
CV(%)	54%	102%	57%	91%	179 %	200 %	54%	41%	41%
number of samples	32	30	30	30	30	30	30	30	30
BDL (%)	0%	33%	0%	30%	0%	0%	0%	0%	0%

GW Co	***Hg (mg/kg)***	***Ag (mg/kg)***	***Mg (%)***	***Cd (mg/kg)***	***K (%)***	***P (%)***	***Ba (mg/kg)***	***Cu (mg/kg)***	***Mn (mg/kg)***
average	0.07	0.31	0.32	0.31	0.46	0.38	111.61	36.80	321.82
std.dev	0.05	0.46	0.21	0.33	0.33	0.71	60.32	25.38	155.91
min	0.02	< DL	0.08	< DL	0.02	0.07	28.37	< DL	99.16
max	0.18	2.02	0.95	1.52	1.32	3.71	245.95	125.42	667.95
25° percentile	0.04	0.04	0.20	0.04	0.21	0.15	60.12	19.22	231.14
90° percentile	0.13	0.84	0.52	0.60	0.84	0.45	198.91	58.52	515.36
CV(%)	66%	149%	65%	105%	70%	185 %	54%	69%	48%
number of samples	25	25	25	25	25	25	25	25	25
BDL (%)	0%	44%	0%	40%	0%	0%	0%	4%	0%

SS Co	***Hg (mg/kg)***	***Ag (mg/kg)***	***Mg (%)***	***Cd (mg/kg)***	***K (%)***	***P (%)***	***Ba (mg/kg)***	***Cu (mg/kg)***	***Mn (mg/kg)***
average	0.24	1.81	0.41	0.47	0.47	1.12	159.05	146.14	483.30
std.dev	0.13	1.69	0.23	0.44	0.33	0.57	82.24	70.43	528.44
min	0.04	< DL	0.16	< DL	< DL	0.28	< DL	61.37	138.05
max	0.52	6.15	1.05	1.56	1.30	2.28	298.75	349.39	2532.33
25° percentile	0.17	0.44	0.27	0.04	0.23	0.72	99.68	89.39	247.61
90° percentile	0.43	3.88	0.66	0.91	0.83	1.96	269.69	204.69	602.40
CV(%)	53%	93%	57%	92%	70%	51%	52%	48%	109%
number of samples	18	18	18	18	18	18	18	18	18
BDL (%)	0%	6%	0%	39%	6%	0%	6%	0%	0%

MBT Co	***Hg (mg/kg)***	***Ag (mg/kg)***	***Mg (%)***	***Cd (mg/kg)***	***K (%)***	***P (%)***	***Ba (mg/kg)***	***Cu (mg/kg)***	***Mn (mg/kg)***
average	0.36	1.60	0.38	1.04	0.85	0.39	119.59	130.54	250.53
std.dev	0.16	1.55	0.16	0.88	1.52	0.15	45.78	53.22	120.64
min	0.11	0.25	0.11	< DL	0.12	0.17	23.45	49.97	107.07
max	0.58	5.33	0.68	2.77	5.39	0.76	166.62	208.29	528.65
25° percentile	0.23	0.38	0.29	0.42	0.29	0.29	91.76	96.68	181.95
90° percentile	0.55	3.13	0.55	2.03	0.88	0.48	162.09	202.67	395.87
CV(%)	43%	97%	42%	85%	180%	39%	38%	41%	48%
number of samples	11	11	11	11	11	11	11	11	11
BDL (%)	0%	0%	0%	18%	0%	0%	0%	0%	0%

BW Di + Man Bw di + Man Ecr Di	***Hg (mg/kg)***	***Ag (mg/kg)***	***Mg (%)***	***Cd (mg/kg)***	***K (%)***	***P (%)***	***Ba (mg/kg)***	***Cu (mg/kg)***	***Mn (mg/kg)***
average	0.09	0.88	0.40	0.50	3.28	0.98	53.70	146.18	256.91
std.dev	0.05	2.27	0.23	0.63	2.82	0.50	48.03	104.54	156.16
min	0.01	< DL	0.04	< DL	0.13	0.36	0.40	17.82	61.42
max	0.20	8.41	0.75	2.43	8.93	2.35	184.07	325.86	516.88
25° percentile	0.06	0.19	0.22	0.24	0.93	0.56	5.50	13.41	35.21
90° percentile	0.46	2.63	0.95	1.78	6.66	1.85	73.26	275.72	429.00
CV(%)	55%	257%	58%	125%	86%	51%	89%	72%	61%
number of samples	13	13	13	13	13	13	13	13	13
BDL (%)	0%	46%	0%	15%	0%	0%	0%	0%	0%

Other	***Hg (mg/kg)***	***Ag (mg/kg)***	***Mg (%)***	***Cd (mg/kg)***	***K (%)***	***P (%)***	***Ba (mg/kg)***	***Cu (mg/kg)***	***Mn (mg/kg)***
average	0.38	1.04	0.53	0.80	1.08	0.60	277.44	186.88	448.46
std.dev	0.46	1.15	0.21	0.67	0.82	0.37	286.80	173.38	219.93
min	0.04	< DL	0.28	< DL	0.08	0.21	42.86	5.51	26.95
max	1.28	3.04	0.89	2.00	2.64	1.37	835.30	131.70	487.74
25° percentile	0.06	0.13	0.40	0.36	0.64	0.42	99.04	27.25	46.21
90° percentile	0.88	2.39	0.74	1.59	1.87	0.95	624.82	104.70	387.47
CV(%)	122%	110%	39%	84%	76%	62%	103%	93%	49%
number of samples	7	7	7	7	7	7	7	7	7
BDL (%)	0%	29%	0%	14%	0%	0%	0%	0%	0%

SS Di + BW	***Hg* (mg/kg)**	***Ag* (mg/kg)**	***Mg* (%)**	***Cd* (mg/kg)**	***K* (%)**	***P* (%)**	***Ba* (mg/kg)**	***Cu* (mg/kg)**	***Mn* (mg/kg)**
average	-	-	-	-	-	-	-	-	-
std.dev									
min									
max									
25° percentile									
90° percentile									
CV(%)									
number of samples	2	1	1	1	1	1	1	1	1
BDL (%)	0%	-	-	-	-	-	-	-	-

BW Co	***Ti***	***Zn***	***Cr***	***Ni***	***Pb***	***V***	***As***	***Co***	***Mo***	***Al***	***Fe***
	(mg/kg)									**(%)**	
average	399.04	117.66	27.25	25.55	38.05	26.59	4.36	6.11	1.30	0.92	1.12
std.dev	210.49	78.39	11.29	29.78	19.80	14.22	2.27	10.43	0.94	0.85	1.20
min	158.25	< DL	8.39	7.14	4.46	8.34	< DL	1.87	< DL	0.24	0.29
max	1276.64	256.96	57.09	155.73	75.70	65.09	12.46	60.48	4.08	5.05	5.87
25° percentile	259.12	65.12	19.29	11.04	23.68	16.56	2.88	2.96	0.61	0.59	0.63
90° percentile	540.18	232.86	38.81	61.39	70.39	45.25	6.44	6.32	2.25	1.34	1.32
CV(%)	53%	67%	41%	117%	52%	53%	52%	171%	73%	92%	108%
number of samples	30	30	30	30	30	30	30	30	30	30	30
BDL (%)	0%	7%	0%	0%	0%	0%	73%	0%	27%	0%	0%

GW Co	***Ti***	***Zn***	***Cr***	***Ni***	***Pb***	***V***	***As***	***Co***	***Mo***	***Al***	***Fe***
	(mg/kg)									**(%)**	
average	542.44	149.90	17.88	19.93	29.84	19.92	3.76	4.14	0.67	0.89	0.76
std.dev	370.43	183.08	7.13	48.08	16.98	11.81	2.57	2.08	0.72	0.81	0.35
min	136.57	16.70	6.09	3.04	4.42	4.92	< DL	1.07	< DL	0.23	0.25
max	1644.02	988.75	32.67	249.99	69.74	53.76	12.73	10.46	2.56	4.39	1.37
25° percentile	296.79	80.30	13.58	8.12	16.56	10.41	1.73	2.97	0.14	0.39	0.45
90° percentile	942.07	189.79	28.04	17.87	51.32	34.45	5.82	5.97	1.67	1.25	1.20
CV(%)	68%	122%	40%	241%	57%	59%	68%	50%	106%	91%	46%
number of samples	25	25	25	25	25	25	25	25	25	25	25
BDL (%)	0%	0%	0%	0%	0%	0%	72%	0%	56%	0%	0%

SS Co	***Ti***	***Zn***	***Cr***	***Ni***	***Pb***	***V***	***As***	***Co***	***Mo***	***Al***	***Fe***
					(mg/kg)					**(%)**	
average	428.92	292.27	34.39	21.65	37.40	27.64	5.95	5.27	4.36	0.93	1.96
std.dev	253.02	164.31	23.85	13.01	20.39	13.41	2.66	2.00	7.97	0.56	1.61
min	134.32	32.31	14.93	8.16	16.12	10.58	< DL	2.80	< DL	0.30	0.61
max	1210.77	657.70	106.98	65.03	103.10	66.33	14.42	10.53	34.29	1.90	7.33
25° percentile	294.88	188.31	17.96	14.09	25.44	20.22	4.56	4.06	1.15	0.48	0.87
90° percentile	647.97	487.72	58.53	33.15	56.70	40.06	8.18	7.33	7.17	1.74	3.35
CV(%)	59%	56%	69%	60%	55%	49%	45%	38%	183%	61%	82%
number of samples	18	18	18	18	18	18	18	18	18	18	18
BDL (%)	0%	0%	0%	0%	0%	0%	61%	0%	11%	0%	0%

MBT Co	***Ti***	***Zn***	***Cr***	***Ni***	***Pb***	***V***	***As***	***Co***	***Mo***	***Al***	***Fe***
					(mg/kg)					**(%)**	
average	257.17	325.21	54.82	32.53	113.48	20.87	4.41	4.06	1.88	0.85	0.96
std.dev	135.71	108.31	61.14	33.85	58.88	21.02	3.63	1.87	1.48	0.26	0.42
min	11.90	160.65	3.01	4.84	2.69	4.05	< DL	1.31	< DL	0.41	0.39
max	442.69	500.88	225.51	130.10	227.48	76.08	14.04	8.14	5.54	1.21	1.69
25° percentile	197.04	247.99	23.59	18.29	100.52	11.71	1.69	3.44	1.10	0.74	0.73
90° percentile	429.93	448.14	82.43	39.10	158.99	43.01	5.66	5.95	3.03	1.13	1.52
CV(%)	53%	33%	112%	104%	52%	101%	82%	46%	79%	31%	44%
number of samples	11	11	11	11	11	11	11	11	11	11	11
BDL (%)	0%	0%	0%	0%	0%	0%	91%	0%	18%	0%	0%

BW Di + Man Bw di + Man Ecr Di	***Ti***	***Zn***	***Cr***	***Ni***	***Pb***	***V***	***As***	***Co***	***Mo***	***Al***	***Fe***
					(mg/kg)					**(%)**	
average	289.91	282.86	26.82	18.38	14.56	14.08	6.49	5.76	2.01	0.66	0.87
std.dev	476.59	167.24	20.20	9.32	17.36	11.43	12.23	5.44	1.46	0.54	0.83
min	24.47	28.12	6.80	5.32	< DL	1.87	< DL	1.32	< DL	0.07	0.15
max	1795.43	625.58	72.52	40.90	56.02	42.54	40.29	16.32	4.99	1.92	2.47
25° percentile	16.74	18.56	7.77	6.75	2.03	2.31	0.89	1.92	0.80	0.25	0.30
90° percentile	490.77	448.13	44.41	22.10	27.11	19.91	8.84	12.34	3.89	1.77	2.20
CV(%)	164%	59%	75%	51%	119%	81%	188%	95%	73%	81%	95%
number of samples	13	13	13	13	13	13	13	13	13	13	13
BDL (%)	0%	0%	0%	0%	8%	0%	77%	0%	15%	0%	0%

Other	***Ti***	***Zn***	***Cr***	***Ni***	***Pb***	***V***	***As***	***Co***	***Mo***	***Al***	***Fe***
					(mg/kg)					**(%)**	
average	336.97	393.14	52.71	33.20	110.29	24.36	3.89	6.38	2.19	0.85	1.35
std.dev	185.49	416.61	43.38	28.97	119.09	11.12	1.77	4.20	1.74	0.53	0.76
min	165.18	141.78	5.51	6.22	< DL	11.76	< DL	1.92	< DL	0.28	0.24
max	701.38	1304.87	131.70	81.62	270.10	42.82	6.43	12.11	4.70	1.83	2.35
25° percentile	213.44	155.00	27.25	14.38	22.27	18.66	3.13	3.28	0.94	0.45	0.80
90° percentile	526.64	789.11	104.70	72.02	262.21	39.07	5.98	12.04	4.28	1.37	2.07
CV(%)	55%	106%	82%	87%	108%	46%	46%	66%	79%	62%	56%
number of samples	7	7	7	7	7	7	7	7	7	7	7
BDL (%)	0%	0%	0%	0%	14%	0%	86%	0%	29%	0%	0%

SS Di + BW	***Ti***	***Zn***	***Cr***	***Ni***	***Pb***	***V***	***As***	***Co***	***Mo***	***Al***	***Fe***
					(mg/kg)					**(%)**	
average	-	-	-	-	-	-	-	-	-	-	-
std.dev											
min											
max											
25° percentile											
90° percentile											
CV(%)											
number of samples	1	1	1	1	1	1	1	1	1	1	1
BDL (%)	-	-	-	-	-	-	-	-	-	-	-

Table SI 2: Descriptive statistic for analyzed PCM in COMDIG samples

When observed values were below the LoD, LoD/2 was used as an estimate for statistical analysis.

Number of samples: 100

BW Co	**Galaxolid**	**Tonalid**	**Cashmeran**	**Celestolid**	**Phantolid**	**Traesolid**
				(µg/kg)		
average	145.05	34.72				
std.dev	325.13	67.48				
min	< DL	< DL				
max	1500.00	290.00				
25° percentile	10.00	5.00				
90° percentile	282.00	94.80				
CV(%)	224%	194%				
number of samples	29	29	29	29	29	29
BDL (%)	52%	52%	100%	100%	100%	76%

GW Co	**Galaxolid**	**Tonalid**	**Cashmeran**	**Celestolid**	**Phantolid**	**Traesolid**
				(µg/kg)		
average	58.04	15.32				
std.dev	191.00	43.11				
min	< DL	< DL				
max	960.00	220.00				
25° percentile	10.00	5.00				
90° percentile	68.40	13.40				
CV(%)	329%	281%				
number of samples	25	25	25	25	25	25
BDL (%)	84%	88%	100%	100%	100%	96%

SS Co	**Galaxolid**	**Tonalid**	**Cashmeran**	**Celestolid**	**Phantolid**	**Traesolid**
				(µg/kg)		
average	2280.00	398.94				
std.dev	1978.49	282.20				
min	< DL	< DL				
max	6800.00	950.00				
25° percentile	715.00	190.00				
90° percentile	4800.00	780.00				
CV(%)	87%	71%				
number of samples	16	16	16	16	16	16
BDL (%)	6%	0%	63%	81%	81%	19%

MBT Co	**Galaxolid**	**Tonalid**	**Cashmeran**	**Celestolid**	**Phantolid**	**Traesolid**
			(µg/kg)			
average	367.00	109.30				
std.dev	263.65	62.43				
min	< DL	< DL				
max	920.00	220.00				
25° percentile	210.00	65.50				
90° percentile	776.00	184.00				
CV(%)	72%	57%				
number of samples	10	10	10	10	10	10
BDL (%)	0%	0%	90%	90%	100%	30%

BW Di + Man Bw di + Man Ecr Di	**Galaxolid**	**Tonalid**	**Cashmeran**	**Celestolid**	**Phantolid**	**Traesolid**
			(µg/kg)			
average	440.62	50.46	-	-	-	-
std.dev	1343.79	142.25				
min	< DL	< DL				
max	4900.00	520.00				
25° percentile	9.50	5.00				
90° percentile	538.93	69.47				
CV(%)	305%	282%				
number of samples	13	13	13	13	13	13
BDL (%)	46%	77%	85%	92%	100%	92%

Other	**Galaxolid**	**Tonalid**	**Cashmeran**	**Celestolid**	**Phantolid**	**Traesolid**
			(µg/kg)			
average	424.00	66.00				
std.dev	579.98	65.71				
min	< DL	< DL				
max	1400.00	140.00				
25° percentile	10.00	5.00				
90° percentile	1036.00	136.00				
CV(%)	137%	100%				
number of samples	5	5	5	5	5	5
BDL (%)	40%	40%	80%	80%	100%	80%

SS Di + BW	**Galaxolid**	**Tonalid**	**Cashmeran**	**Celestolid**	**Phantolid**	**Traesolid**
			(µg/kg)			
average	-	-	-	-	-	-
std.dev						
min						
max						
25° percentile						
90° percentile						
CV(%)						
number of samples	2	2	2	2	2	2
BDL (%)	0%	0%	50%	100%	100%	0%

Table SI 3: Siloxanes concentration in analysed COMDIG samples

Number of samples: 8

GW Co	**Sampling at plant**	**Control sampling by JRC staff**
	µg/kg	
MDM	nd	nd
D4	75	91
MD2M	nd	nd
D5	120	110
MD3M	nd	nd
D6	1100	1200

SS Co	**Sampling at plant**	**Control sampling by JRC staff**
	µg/kg	
MDM	nd	nd
D4	860	880
MD2M	nd	nd
D5	1500	1300
MD3M	20	18
D6	410	370

MBT Co	**Sampling at plant**	**Control sampling by JRC staff**
	µg/kg	
MDM	nd	nd
D4	200	290
MD2M	nd	nd
D5	1000	1300
MD3M	< 10	nd
D6	1700	1300

BW Di	**Sampling at plant**	**Control sampling by JRC staff**
	µg/kg	
MDM	nd	nd
D4	nd	nd
MD2M	nd	nd
D5	110	130
MD3M	< 10	nd
D6	240	250

Table SI 4: Descriptive statistic for analyzed PAHs in COMDIG samples

When observed values were below the LoD, LoD/2 was used as an estimate for statistical analysis.

Number of sample: 105

BW Co	**Antracene**	**Benzo(a) antracene**	**Benzo(b) fluoranthene**	**Benzo(k) fluoranthene**	**Benzo(a) pyrene**	**Benzo(e) pyrene**	**Benzo(g,h,i) perylene**	**Chrysene**	**Coronene**
					(ng/g)				
average	24.06	101.64	122.50	59.48	68.48	88.80	82.42	297.46	22.67
std.dev	23.00	116.77	118.96	54.40	81.25	81.49	79.14	802.67	25.84
min	< DL	10.10	21.60	11.60	< DL	< DL	< DL	12.90	< DL
max	104.00	636.90	550.90	254.30	399.10	365.50	369.00	4506.00	107.70
25° percentile	9.75	29.39	40.65	21.53	19.02	30.26	35.88	72.64	5.70
90° percentile	42.38	195.43	256.40	118.14	132.33	198.26	136.98	358.18	60.68
CV(%)	96%	115%	97%	91%	119%	92%	96%	270%	114%
number of samples	30	30	30	30	30	30	30	30	30
BDL (%)	3%	0%	0%	0%	10%	3%	7%	0%	53%

GW Co	**Antracene**	**Benzo(a) antracene**	**Benzo(b) fluoranthene**	**Benzo(k) fluoranthene**	**Benzo(a) pyrene**	**Benzo(e) pyrene**	**Benzo(g,h,i) perylene**	**Chrysene**	**Coronene**
					(ng/g)				
average	31.20	98.13	168.86	81.27	107.98	125.40	140.58	168.08	31.92
std.dev	37.87	144.37	246.22	129.73	199.36	184.57	194.54	195.97	46.82
min	< DL	19.00	20.30	10.30	< DL	15.10	22.40	24.20	< DL
max	171.90	568.36	1097.28	596.28	898.83	845.14	820.30	839.91	188.98
25° percentile	12.10	33.54	69.90	29.97	32.97	55.02	45.69	78.75	5.70
90° percentile	75.51	135.25	321.80	139.19	186.93	240.11	419.57	244.10	87.87
CV(%)	121%	147%	146%	160%	185%	147%	138%	117%	147%
number of samples	24	24	24	24	24	24	24	24	24
BDL (%)	4%	0%	0%	0%	4%	0%	0%	0%	54%

SS Co	Antracene	Benzo(a) antracene	Benzo(b) fluoranthene	Benzo(k) fluoranthene	Benzo(a) pyrene	Benzo(e) pyrene	Benzo(g,h,i) perylene	Chrysene	Coronene
					(ng/g)				
average	27.17	129.23	186.22	92.06	119.64	138.62	145.08	199.15	51.78
std.dev	20.91	217.20	305.99	151.04	190.37	231.70	212.19	333.88	92.24
min	< DL	19.50	35.50	22.30	19.80	25.40	26.80	34.10	< DL
max	85.30	929.80	1289.20	638.20	770.40	963.00	871.10	1426.00	357.70
25° percentile	13.55	61.84	77.59	31.65	46.35	52.08	58.90	85.63	5.70
90° percentile	47.50	144.76	281.35	131.65	227.23	239.55	266.45	247.00	113.10
CV(%)	77%	168%	164%	164%	159%	167%	146%	168%	178%
number of samples	16	16	16	16	16	16	16	16	16
BDL (%)	6%	0%	0%	0%	0%	0%	0%	0%	50%

MBT Co	Antracene	Benzo(a) antracene	Benzo(b) fluoranthene	Benzo(k) fluoranthene	Benzo(a) pyrene	Benzo(e) pyrene	Benzo(g,h,i) perylene	Chrysene	Coronene
					(ng/g)				
average	13.42	60.95	77.82	42.39	64.05	87.06	75.58	94.89	23.75
std.dev	10.90	66.26	64.11	32.94	61.39	89.87	62.39	74.84	39.34
min	< DL	10.20	22.60	13.30	17.80	20.00	29.60	32.90	< DL
max	33.10	181.28	194.92	102.24	195.46	304.93	187.50	214.30	125.52
25° percentile	5.20	24.07	34.30	15.20	25.32	35.80	40.40	40.20	5.70
90° percentile	26.33	173.06	173.78	91.25	149.41	171.63	181.10	211.15	50.70
CV(%)	81%	109%	82%	78%	96%	103%	83%	79%	166%
number of samples	9	9	9	9	9	9	9	9	9
BDL (%)	11%	0%	0%	0%	0%	0%	0%	0%	67%

BW Di + Man Bw di + Man Ecr Di	**Antracene**	**Benzo(a) antracene**	**Benzo(b) fluoranthene**	**Benzo(k) fluoranthene**	**Benzo(a) pyrene**	**Benzo(e) pyrene**	**Benzo(g,h,i) perylene**	**Chrysene**	**Coronene**
					(ng/g)				
average	28.80	39.01	47.27	23.00	46.36	45.87	36.86	48.46	7.87
std.dev	49.73	26.11	37.36	17.08	48.65	33.05	28.28	37.25	8.68
min	< DL	5.70	< DL	< DL	< DL	< DL	< DL	< DL	< DL
max	205.43	107.18	151.70	61.20	176.60	108.60	85.90	153.95	40.40
25° percentile	4.55	7.90	9.93	4.15	6.25	6.25	3.35	10.03	5.70
90° percentile	54.24	59.95	69.00	44.10	77.93	79.08	64.17	69.95	11.32
CV(%)	173%	67%	79%	74%	105%	72%	77%	77%	110%
number of samples	16	16	16	16	16	16	16	16	16
BDL (%)	25%	0%	6%	6%	13%	6%	31%	6%	94%

Other	**Antracene**	**Benzo(a) antracene**	**Benzo(b) fluoranthene**	**Benzo(k) fluoranthene**	**Benzo(a) pyrene**	**Benzo(e) pyrene**	**Benzo(g,h,i) perylene**	**Chrysene**	**Coronene**
					(ng/g)				
average	83.50	431.97	388.88	202.34	337.14	360.37	327.79	493.77	147.57
std.dev	150.80	827.98	681.48	363.85	610.10	643.66	553.39	897.03	198.91
min	9.50	20.30	65.62	32.86	52.00	65.19	73.50	44.82	28.30
max	353.00	1911.30	1607.80	853.10	1428.20	1511.70	1317.70	2096.30	500.70
25° percentile	10.01	34.22	87.30	34.80	52.50	68.30	81.10	65.80	42.90
90° percentile	221.71	1197.26	1001.64	531.84	892.45	941.48	824.62	1324.62	335.16
CV(%)	181%	192%	175%	180%	181%	179%	169%	182%	135%
number of samples	5	5	5	5	5	5	5	5	5
BDL (%)	0%	0%	0%	0%	0%	0%	0%	0%	0%

SS Di + BW	Antracene	Benzo(a) antracene	Benzo(b) fluoranthene	Benzo(k) fluoranthene	Benzo(a) pyrene	Benzo(e)pyrene	Benzo(g,h,i) perylene	Chrysene	Coronene
					(ng/g)				
average	14.83	95.38	75.72	60.36	103.59	116.09	124.66	92.12	23.33
std.dev	7.77	96.85	65.40	57.11	95.06	133.19	155.75	43.08	30.96
min	< DL	15.74	34.02	17.77	47.65	34.91	42.98	60.53	< DL
max	22.28	232.95	189.16	159.44	272.89	351.78	402.68	153.86	77.19
25° percentile	8.98	25.03	39.06	30.99	61.23	45.51	51.24	60.67	5.70
90° percentile	21.84	204.44	143.35	118.22	192.77	245.67	270.04	140.82	55.25
CV(%)	52%	102%	86%	95%	92%	115%	125%	47%	133%
number of samples	5	5	5	5	5	5	5	5	5
BDL (%)	20%	0%	0%	0%	0%	0%	0%	0%	0%

BW Co	Dibenz(a,h) antracene	Fluoranthene	Indeno (1,2,3-cd) pyrene	Perylene	Phenantrene	Pyrene	Dibenzo(a,e) pyrene	Dibenzo(a,h) pyrene	Dibenzo(a,i) pyrene	Dibenzo(a,l) pyrene
					(ng/g)					
average	33.57	323.73	97.52	30.44	172.58	216.84				
std.dev	37.54	246.96	125.89	27.98	168.80	145.98				
min	< DL	39.07	< DL	< DL	9.84	34.10				
max	457.70	1017.30	614.10	157.80	601.71	592.70				
25° percentile	28.75	138.50	33.87	16.78	45.95	109.10				
90° percentile	212.35	677.63	177.03	48.35	421.65	440.34				
CV(%)	112%	76%	129%	92%	98%	67%				
number of samples	30	30	30	30	30	30	0	8	0	2
BDL (%)	23%	0%	3%	10%	0%	0%	100%	73%	100%	93%

GW Co	Dibenz(a,h)antracene	Fluoranthene	Indeno (1,2,3-cd) pyrene	Perylene	Phenantrene	Pyrene	Dibenzo(a,e) pyrene	Dibenzo(a,h)pyrene	Dibenzo(a,i) pyrene	Dibenzo(a,l) pyrene
					(ng/g)					
average	58.39	206.86	112.95	38.04	89.35	126.77				
std.dev	93.89	173.55	175.48	53.72	110.03	98.08				
min	< DL	26.20	12.40	< DL	< DL	18.50				
max	402.08	659.30	815.27	210.54	445.42	336.30				
25° percentile	11.30	86.02	35.64	15.96	19.09	40.73				
90° percentile	131.73	458.14	255.74	66.36	197.49	260.79				
CV(%)	161%	84%	155%	141%	123%	77%				
number of samples	24	24	24	24	24	24	24	24	24	24
BDL (%)	13%	0%	0%	4%	4%	0%	100%	71%	100%	100%

SS Co	Dibenz(a,h) antracene	Fluoranthene	Indeno(1,2,3-cd) pyrene	Perylene	Phenantrene	Pyrene	Dibenzo(a,e) pyrene	Dibenzo(a,h) pyrene	Dibenzo(a,i) pyrene	Dibenzo(a,l) pyrene
					(ng/g)					
average	57.63	285.12	122.60	42.01	125.06	219.46				
std.dev	99.58	243.62	175.51	63.16	140.44	198.24				
min	< DL	29.30	24.70	< DL	17.60	44.60				
max	408.80	1020.10	720.10	254.00	551.40	869.00				
25° percentile	19.63	159.83	42.37	11.58	33.38	108.57				
90° percentile	99.05	499.60	221.55	80.35	271.40	352.60				
CV(%)	173%	85%	143%	150%	112%	90%				
number of samples	16	16	16	16	16	16	16	16	16	16
BDL (%)	19%	0%	0%	13%	0%	0%	94%	63%	94%	88%

MBT Co	**Dibenz(a,h) antracene**	**Fluoranthene**	**Indeno (1,2,3-cd) pyrene**	**Perylene**	**Phenantrene**	**Pyrene**	**Dibenzo(a,e) pyrene**	**Dibenzo(a,h) pyrene**	**Dibenzo(a,i) pyrene**	**Dibenzo(a,l) pyrene**
					(ng/g)					
average	25.95	179.60	58.33	27.79	131.29	151.96				
std.dev	25.09	161.92	42.44	16.84	59.13	151.70				
min	< DL	34.20	18.70	5.80	48.50	41.60				
max	76.40	511.20	134.00	56.20	221.50	521.94				
25° percentile	11.70	84.55	23.60	19.10	96.27	65.50				
90° percentile	62.32	360.80	123.39	53.20	217.82	285.03				
CV(%)	97%	90%	73%	61%	45%	100%				
number of samples	9	9	9	9	9	9	9	9	9	9
BDL (%)	22%	0%	0%	0%	0%	0%	100%	89%	100%	89%

BW Di + Man Bw di + Man Ecr Di	**Dibenz(a,h)antracene**	**Fluoranthene**	**Indeno(1,2,3-cd)pyrene**	**Perylene**	**Phenantrene**	**Pyrene**	**Dibenzo(a,e)pyrene**	**Dibenzo(a,h)pyrene**	**Dibenzo(a,i)pyrene**	**Dibenzo(a,l)pyrene**
						(ng/g)				
average	36.45	82.37	22.04	20.24	152.52	93.81	-	-	-	-
std.dev	79.63	71.51	18.71	13.79	247.45	92.06				
min	< DL	12.45	< DL	< DL	26.80	14.22				
max	329.83	243.97	79.70	42.70	1021.92	384.07				
25° percentile	3.15	13.51	3.46	3.58	28.00	15.34				
90° percentile	68.04	155.15	29.80	35.86	278.98	150.58				
CV(%)	218%	87%	85%	68%	162%	98%				
number of samples	16	16	16	16	16	16	16	16	16	16
BDL (%)	38%	0%	19%	19%	0%	0%	100%	100%	100%	100%

Other	Dibenz(a,h)anthracene	Fluoranthene	Indeno (1,2,3-cd) pyrene	Perylene	Phenantrene	Pyrene	Dibenzo(a,e) pyrene	Dibenzo(a,h) pyrene	Dibenzo(a,i) pyrene	Dibenzo(a,l) pyrene
					(ng/g)					
average	141.61	1098.82	235.64	100.56	831.34	771.48	-	-	-	-
std.dev	246.42	2063.29	376.56	161.83	1717.88	1367.87				
min	23.60	50.20	44.90	24.10	32.90	21.90				
max	582.30	4778.40	908.80	390.00	3904.10	3197.20				
25° percentile	30.21	63.05	70.30	27.60	53.40	53.64				
90° percentile	365.23	3047.20	578.35	246.24	2380.85	2106.72				
CV(%)	174%	188%	160%	161%	207%	177%				
number of samples	5	5	5	5	5	5	5	5	5	5
BDL (%)	0%	0%	0%	0%	0%	0%	80%	20%	100%	40%

SS Di + BW	Dibenz(a,h)anthracene	Fluoranthene	Indeno (1,2,3-cd) pyrene	Perylene	Phenantrene	Pyrene	Dibenzo(a,e) pyrene	Dibenzo(a,h) pyrene	Dibenzo(a,i)pyrene	Dibenzo(a,l)pyrene
					(ng/g)					
average	63.32	82.18	76.43	78.25	113.74	132.99	-	-	-	-
std.dev	86.76	39.46	91.21	103.34	32.44	18.39				
min	< DL	15.11	23.16	19.52	61.11	115.74				
max	214.73	112.12	239.03	261.72	142.96	162.81				
25° percentile	20.37	82.43	37.75	19.94	105.54	120.76				
90° percentile	151.18	111.03	160.54	175.34	138.84	151.94				
CV(%)	137%	48%	119%	132%	29%	14%				
number of samples	5	5	5	5	5	5	5	5	5	5
BDL (%)	20%	0%	0%	0%	0%	0%	80%	100%	100%	80%

Table SI 5 Descriptive statistic for AhR-active compounds in COMDIG samples

When observed values were below the LoD, LoD/2 was used as an estimate for statistical analysis.

Number of samples: 98

BW Co	
	TEQbio (ng/g) *
average	0.83
std.dev	0.77
min	< DL
max	3.26
25° percentile	0.36
90° percentile	1.56
CV(%)	93%
number of samples	29
BDL (%)	3%

GW Co	
	TEQbio(ng/g) *
average	1.13
std.dev	1.27
min	0.13
max	5.46
25° percentile	0.29
90° percentile	2.48
CV(%)	113%
number of samples	24
BDL (%)	0%

SS Co	
	TEQbio(ng/g) *
average	0.90
std.dev	0.99
min	< DL
max	4.37
25° percentile	0.36
90° percentile	1.64
CV(%)	110%
number of samples	18
BDL (%)	6%

MBT Co	
	TEQbio (ng/g) *
average	0.67
std.dev	0.49
min	0.16
max	1.63
25° percentile	0.29
90° percentile	1.30
CV(%)	73%
number of samples	10
BDL (%)	0%

BW Di + Man Bw di + Man Ecr Di	
	TEQbio (ng/g) *
average	0.16
std.dev	0.20
min	< DL
max	0.46
25° percentile	0.04
90° percentile	1.19
CV(%)	125%
number of samples	6
BDL (%)	33%

Other	
	TEQbio (ng/g) *
average	4.58
std.dev	4.76
min	0.26
max	12.72
25° percentile	0.47
90° percentile	10.02
CV(%)	104%
number of samples	7
BDL (%)	0%

SS Di + BW	
	TEQbio (ng/g) *
average	-
std.dev	
min	
max	
25° percentile	
90° percentile	
CV(%)	
number of samples	0
BDL (%)	-

Table SI 6: I-TEQ for PCDD/Fs in COMDIG samples following bioassay

	LB WHO2006	UB WHO2006
BW Co	**ng I-TEQ/kg**	
	1.9875	4.25
	0.9375	4.1875
	0.825	3.375
	0.5875	4.8
GW Co		
	0.8875	3.7125
	2.9875	5.1625
	0.925	5.3
SS Co		
	0.1	13.2875
	2.2375	7.55
	0.525	7.3875
MBT Co		
	1.175	5.3875
	0.85	4.0125
	0.075	3
Man BW Di		
	0.025	15.125
	0.0375	12.6375
	0	15.25
MBT Di		
	0.625	2.975
	1.3875	5.45
Other		
	2.075	13.425
	2.725	6.8625

Table SI 7: PCBs in COMDIG samples following bioassay

	Sum of 7 PCB (PCBs 28, 52, 101, 118, 138, 153 and 180)
	µg/kg
BW Co	23.295
	29.381
	18.961
	46.341
GW Co	19.263
	15.526
	23.353
SS Co	10.544
	31.867
	35.387
MBT Co	15.452
	23.576
	6.655
Man BW Di	1.037
	12.214
Man Ecr Di	8.574
MBT Di	10.686
	34.187
Other	30.348
	103.844

Table SI 8: I-TEQ for PCDD/Fs in analysed COMDIG samples from comparative sampling

Number of samples: 2 per type of compost

BW Co		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
I-TEQ	5.76	4.90
1998 WHO-TEQ	5.57	4.71
2005 WHO-TEQ	5.26	4.23
Middle-bound		
I-TEQ	5.76	4.90
1998 WHO-TEQ	5.57	4.71
2005 WHO-TEQ	5.26	4.23
Lower-bound		
I-TEQ	5.76	4.90
1998 WHO-TEQ	5.57	4.71
2005 WHO-TEQ	5.26	4.23

GW Co		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
I-TEQ	12.3	15.0
1998 WHO-TEQ	12.0	14.9
2005 WHO-TEQ	11.4	13.9
Middle-bound		
I-TEQ	12.3	15.0
1998 WHO-TEQ	12.0	14.9
2005 WHO-TEQ	11.4	13.9
Lower-bound		
I-TEQ	12.3	15.0
1998 WHO-TEQ	12.0	14.9
2005 WHO-TEQ	11.4	13.9

SS Co		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
I-TEQ	5.56	4.58
1998 WHO-TEQ	5.27	4.54
2005 WHO-TEQ	4.92	4.18
Middle-bound		
I-TEQ	5.56	4.58
1998 WHO-TEQ	5.27	4.54
2005 WHO-TEQ	4.92	4.18
Lower-bound		
I-TEQ	5.56	4.58
1998 WHO-TEQ	5.27	4.54
2005 WHO-TEQ	4.92	4.18

MBT Co		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
I-TEQ	9.29	9.08
1998 WHO-TEQ	7.44	7.33
2005 WHO-TEQ	7.62	7.52
Middle-bound		
I-TEQ	9.29	9.08
1998 WHO-TEQ	7.44	7.33
2005 WHO-TEQ	7.62	7.52
Lower-bound		
I-TEQ	9.29	9.08
1998 WHO-TEQ	7.44	7.33
2005 WHO-TEQ	7.62	7.52

BW Di		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
I-TEQ	9.04	12.9
1998 WHO-TEQ	8.18	11.1
2005 WHO-TEQ	7.89	10.8
Middle-bound		
I-TEQ	9.04	12.9
1998 WHO-TEQ	8.18	11.1
2005 WHO-TEQ	7.89	10.8
Lower-bound		
I-TEQ	9.04	12.9
1998 WHO-TEQ	8.18	11.1
2005 WHO-TEQ	7.89	10.8

Table SI 9: I-TEQ for DL-PCBs (HRGC-HRMS) in COMDIG samples from comparative sampling

Number of samples: 2 per type of compost

BW Co

Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
1998 WHO-TEQ	2.86	2.53
2005 WHO-TEQ	2.26	2.03

GW Co

Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
1998 WHO-TEQ	3.4701	6.9308
2005 WHO-TEQ	3.0352	4.9653

SS Co

Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
1998 WHO-TEQ	3.64	3.54
2005 WHO-TEQ	3.05	2.97

MBT Co

Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
1998 WHO-TEQ	6.5904	5.0274
2005 WHO-TEQ	3.6908	2.8430

BW Di

Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(pg/g)	
1998 WHO-TEQ	5.49	6.26
2005 WHO-TEQ	4.43	5.23

Table SI 10: Indicators-PCBs in COMDIG samples from comparative sampling

Number of samples: 2 per type of compost

Sum of PCB-28, PCB-52, PCB-101, PCB-138, PCB-153 and PCB-180 analysed by HRGC-HRMS.

BW Co		
	Sampling at plant	**Control sampling by JRC staff**
	(ng/g)	
ΣIndicator-PCBs	30.5	26.4

GW Co		
	Sampling at plant	**Control sampling by JRC staff**
	(ng/g)	
ΣIndicator-PCBs	25.9	94.2

SS Co		
	Sampling at plant	**Control sampling by JRC staff**
	(ng/g)	
ΣIndicator-PCBs	33.5	32.5

MBT Co		
	Sampling at plant	**Control sampling by JRC staff**
	(ng/g)	
ΣIndicator-PCBs	94.2	78.2

BW Di		
	Sampling at plant	**Control sampling by JRC staff**
	(ng/g)	
ΣIndicator-PCBs	58.1	56.3

Table SI 11: I-TEQ for PBDE in analysed COMDIG samples from comparative sampling

Number of samples: 2 per type pf compost

BW Co		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(ng/g)	
Total WFD PBDEs/Penta Formulated *(ΣBDE-28, BDE47, BDE-99, BDE-101, BDE-153 and BDE-154)*	9.39	8.07
Total OCTA Formulated *(ΣBDE-183, BDE-196, BDE-197 and BDE-203)*	0.27	0.25
Total DECA Formulated *(ΣBDE-206, BDE-207, BDE-208 and BDE-209)*	14.1	11.1

GW Co		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(ng/g)	
Total WFD PBDEs/Penta Formulated *(ΣBDE-28, BDE47, BDE-99, BDE-101, BDE-153 and BDE-154)*	4.44	47.5
Total OCTA Formulated *(ΣBDE-183, BDE-196, BDE-197 and BDE-203)*	0.14	0.21
Total DECA Formulated *(ΣBDE-206, BDE-207, BDE-208 and BDE-209)*	6.04	7.02

SS Co		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(ng/g)	
Total WFD PBDEs/Penta Formulated *(ΣBDE-28, BDE47, BDE-99, BDE-101, BDE-153 and BDE-154)*	10.8	10.5
Total OCTA Formulated *(ΣBDE-183, BDE-196, BDE-197 and BDE-203)*	1.99	1.59
Total DECA Formulated *(ΣBDE-206, BDE-207, BDE-208 and BDE-209)*	123	115

MBT Co		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(ng/g)	
Total WFD PBDEs/Penta Formulated *(ΣBDE-28, BDE47, BDE-99, BDE-101, BDE-153 and BDE-154)*	67.3	67.4
Total OCTA Formulated *(ΣBDE-183, BDE-196, BDE-197 and BDE-203)*	69.5	54.7
Total DECA Formulated *(ΣBDE-206, BDE-207, BDE-208 and BDE-209)*	958	1004

BW Di		
Upper-bound	**Sampling at plant**	**Control sampling by JRC staff**
	(ng/g)	
Total WFD PBDEs/Penta Formulated *(ΣBDE-28, BDE47, BDE-99, BDE-101, BDE-153 and BDE-154)*	9.93	13.2
Total OCTA Formulated *(ΣBDE-183, BDE-196, BDE-197 and BDE-203)*	1.03	1.44
Total DECA Formulated *(ΣBDE-206, BDE-207, BDE-208 and BDE-209)*	22.4	38.4

Table SI 12: PBDEs concentration in pooled COMDIG samples

Pool samples	**Category**							
	Bw Co	**GW Co**	**SS Co**	**MBT Co**	**BW Di**	**Man Bw Di**	**Man BW Di/Man Ecr Di**	**Other**
Total WFD PBDEs/Penta Formulated *(ΣBDE-28, BDE47, BDE-99, BDE-101, BDE-153 and BDE-154)*	4.372	1.656	34.889	39.428	3.802	9.676	1.073	10.989
Total OCTA Formulated *(ΣBDE-183, BDE-196, BDE-197 and BDE-203)*	2.666	0.279	3.110	24.645	1.275	2.185	0.117	5.798
Total DECA Formulated *(ΣBDE-206, BDE-207, BDE-208 and BDE-209)*	25.145	4.130	112.103	488.375	13.250	38.555	3.605	663.992

Table SI 13: Descriptive statistic for analyzed PFASs in COMDIG samples

When observed values were below the LoD, LoD/2 was used as an estimate for statistical analysis.

Number of samples:111

BW Co

	PFNA	PFOA	PFOS	PFDA	PFHpA	PFBS
			(ng/g)			
average	0.75	3.37	2.19			
std.dev	0.44	1.72	3.48			
min	< DL	0.77	0.56	< DL	< DL	
max	1.74	7.50	18.59	152.80	0.24	
25° percentile	0.57	1.99	0.85	7.85	0.13	
90° percentile	1.33	6.02	3.45	54.58	0.23	
CV(%)	59%	51%	159%			
number of samples	30	30	30	20	20	20
BDL (%)	13%	0%	0%	35%	45%	100%

GW Co

	PFNA	PFOA	PFOS	PFDA	PFHpA	PFBS
			(ng/g)			
average	0.57	3.47	2.48			
std.dev	0.49	2.37	4.50			
min	< DL	0.66	< DL	< DL	< DL	
max	2.28	11.10	21.57	81.54	0.16	
25° percentile	0.18	1.52	0.75	1.42	0.10	
90° percentile	0.89	5.82	4.07	65.19	0.15	
CV(%)	86%	68%	182%			
number of samples	23	23	23	10	10	10
BDL (%)	22%	0%	4%	50%	60%	100%

SS Co

	PFNA	PFOA	PFOS	PFDA	PFHpA	PFBS
			(ng/g)			
average	2.46	11.20	39.35			
std.dev	1.87	6.78	46.30			
min	< DL	2.54	1.77	< DL	< DL	
max	6.30	25.86	145.66	866.82	0.61	
25° percentile	1.23	7.32	10.15	55.03	0.20	
90° percentile	5.15	19.84	117.15	779.21	0.48	
CV(%)	76%	61%	118%			
number of samples	15	15	15	12	12	12
BDL (%)	13%	0%	0%	50%	25%	100%

MBT Co

	PFNA	PFOA	PFOS	PFDA	PFHpA	PFBS
			(ng/g)			
average	1.89	6.67	5.71			
std.dev	0.97	3.69	2.87			
min	0.31	2.15	2.24	< DL		
max	3.19	13.17	11.04	149.77		
25° percentile	1.22	4.25	3.17	56.26		
90° percentile	2.90	11.59	8.62	137.30		
CV(%)	51%	55%	50%			
number of samples	12	12	12	5	5	5
BDL (%)	0%	0%	0%	60%	80%	100%

BW Di+Man BW Di + Man Ecr Di

	PFNA	PFOA	PFOS	PFDA	PFHpA	PFBS
			(ng/g)			
average	1.42	2.23	6.42	-	0.33	-
std.dev	2.24	1.75	9.87		0.40	
min	< DL	< DL	< DL	< DL	< DL	
max	6.92	5.58	37.37	92.99	0.92	
25° percentile	0.05	0.79	0.33	#DIV/0!	#DIV/0!	
90° percentile	5.94	5.18	12.41	#DIV/0!	#DIV/0!	
CV(%)	157%	79%	154%		121%	
number of samples	19	19	19	9	9	9

Other

	PFNA	PFOA	PFOS	PFDA	PFHpA	PFBS
			(ng/g)			
average	1.72	6.69	5.72	48.95	0.27	
std.dev	0.85	3.80	3.09	34.41	0.09	
min	0.54	1.47	1.15	< DL	< DL	
max	3.18	13.70	11.10	106.29	0.38	
25° percentile	1.30	5.04	4.37	34.73	0.21	
90° percentile	2.62	10.32	8.93	83.49	0.36	
CV(%)	49%	57%	54%	70%	36%	
number of samples	7	7	7	6	6	6
BDL (%)	0%	0%	0%	17%	17%	100%

BDL (%)	42%	21%	32%	78%	56%	100%

SS Di + BW

	PFNA	**PFOA**	**PFOS**	**PFDA**	**PFHpA**	**PFBS**
	(ng/g)					
average	2.49	5.65	14.39	-	-	-
std.dev	2.00	3.15	10.85			
min	0.51	2.82	3.79	5.92		
max	5.45	9.92	27.60	94.59		
25° percentile	1.28	3.03	6.63	28.09		
90° percentile	4.69	9.13	26.32	85.72		
CV(%)	80%	56%	75%			
number of samples	5	5	5	2	2	2
BDL (%)	0%	0%	0%	0%	50%	50%

Table SI 14: Comparative statistics

t-Test: Paired Two Sample for Means

	Plant Sample data	*JRC sample data*
Mean	216.1333961	233.3486846
Variance	322887.9884	386131.3506
Observations	75	75
Pearson Correlation	0.973755608	
Hypothesized Mean Difference	0	
df	74	
t Stat	-1.020107412	
P(T<=t) one-tail	0.155500515	
t Critical one-tail	1.665706893	
P(T<=t) two-tail	0.31100103	
t Critical two-tail	1.992543495	

Table SI 15: Descriptive statistic for detected pesticides in COMDIG samples

The dataset contained not available (n.a.) data, which were treated like missing values (MV). No substitution was made and statistical parameters were computed using the available number of true data only, which varies between every analyzed compound.

Descriptive statistic was not always computed because the number of positive detection was not significant.

In the following table the available statistical parameters are reported.

BW Co	**2,4-D**	**Dichlorprop**	**Mecoprop**	**MCPA**	**2,4,5-T**	**Bentazone**	**Imidacloprid**
				(ng/g)			
average							
std.dev							
min	< DL	< DL	< DL	< DL	< DL	< DL	< DL
max	6.47	7.32	19.93	6.29	0.37	0.00	2.42
25° percentile	0.71	2.37	10.47	3.33	0.37	#NUM!	2.42
90° percentile	5.28	6.66	18.67	5.80	0.37	#NUM!	2.42
CV(%)							
number of samples	20	20	20	20	20	20	20
BDL (%)	80%	90%	90%	85%	95%	100%	95%

GW Co	**2,4-D**	**Dichlorprop**	**Mecoprop**	**MCPA**	**2,4,5-T**	**Bentazone**	**Imidacloprid**
				(ng/g)			
average							
std.dev							
min	< DL						
max	2.03						
25° percentile	0.73						
90° percentile	1.86						
CV(%)							
number of samples	11	11	11	11	11	11	11
BDL (%)	82%	100%	91%	100%	100%	100%	100%

SS Co	**2,4-D**	**Dichlorprop**	**Mecoprop**	**MCPA**	**2,4,5-T**	**Bentazone**	**Imidacloprid**
				(ng/g)			
average							
std.dev							
min	< DL	< DL	< DL	< DL	< DL	< DL	< DL
max	0.97	0.00	4.85	0.00	0.25	0.00	0.00
25° percentile							
90° percentile							
CV(%)							
number of samples	12	12	12	12	12	12	12
BDL (%)	92%	100%	92%	100%	92%	100%	100%

MBT Co	**2,4-D**	**Dichlorprop**	**Mecoprop**	**MCPA**	**2,4,5-T**	**Bentazone**	**Imidacloprid**
				(ng/g)			
average							
std.dev							
min	< DL	< DL	< DL	< DL	< DL		
max	12.20	5.73	14.95	45.09	0.31		
25° percentile	6.53	3.19	6.69	33.22	0.23		
90° percentile	11.44	5.39	13.84	43.51	0.30		
CV(%)							
number of samples	5	5	5	5	5	5	5
BDL (%)	60%	60%	60%	60%	60%	100%	80%

BW Di + Man Bw di + Man Ecr Di	**2,4-D**	**Dichlorprop**	**Mecoprop**	**MCPA**	**2,4,5-T**	**Bentazone**	**Imidacloprid**
				(ng/g)			
average	-		-	-	-	-	-
std.dev							
min	< DL		< DL			< DL	
max	0.40		5.68			0.41	
25° percentile	0.40		0.88			0.41	
90° percentile	1.00		4.10			1.00	
CV(%)							
number of samples	9	9	9	9	9	9	9
BDL (%)	89%	100%	78%	100%	100%	89%	100%

Other	**2,4-D**	**Dichlorprop**	**Mecoprop**	**MCPA**	**2,4,5-T**	**Bentazone**	**Imidacloprid**
				(ng/g)			
average							
std.dev							
min							
max							
25° percentile							
90° percentile							
CV(%)							
number of samples	6	6	6	6	6	6	6
BDL (%)	100%	100%	83%	83%	100%	100%	100%

SS Di + BW	**2,4-D**	**Dichlorprop**	**Mecoprop**	**MCPA**	**2,4,5-T**	**Bentazone**	**Imidacloprid**
				(ng/g)			
average	-		-	-	-	-	-
std.dev							
min							
max							
25° percentile							
90° percentile							
CV(%)							
number of samples	1	1	1	1	1	1	1
BDL (%)	100%	100%	83%	83%	100%	100%	100%

Table SI 16: Descriptive statistic for analyzed sweeteners in COMDIG samples

The dataset contained not available (n.a.) data, which were treated like missing values (MV). No substitution was made and statistical parameters were computed using the available number of true data only, which varies between every analyzed compound.

BW Co	**Saccharin**	**Acesulfame K**	**Sucralose**
		(ng/g)	
average	2.73		
std.dev	3.78		
min	< DL		
max	13.43		
25° percentile	0.00		
90° percentile	6.96		
CV(%)	139%		
number of samples	20	20	20
BDL (%)	45%	75%	100%

GW Co	**Saccharin**	**Acesulfame K**	**Sucralose**
		(ng/g)	
average	0.45		
std.dev	0.59		
min	< DL		
max	1.59		
25° percentile	0.00		
90° percentile	1.46		
CV(%)	132%		
number of samples	11	11	11
BDL (%)	45%	73%	100%

SS Co	**Saccharin**	**Acesulfame K**	**Sucralose**
		(ng/g)	
average	0.56		
std.dev	0.73		
min	< DL		
max	2.02		
25° percentile	0.00		
90° percentile	1.49		
CV(%)	130%		
number of samples	12	12	12
BDL (%)	42%	92%	100%

MBT Co	Saccharin	Acesulfame K	Sucralose
		(ng/g)	
average	31.51		
std.dev	45.86		
min	< DL		
max	107.41		
25° percentile	0.00		
90° percentile	81.30		
CV(%)	146%		
number of samples	5	5	5
BDL (%)	40%	40%	80%

BW Di + Man Bw di + Man Ecr Di	Saccharin	Acesulfame K	Sucralose
		(ng/g)	
average	18.60	-	-
std.dev	33.47		
min	< DL		
max	103.70		
25° percentile	0.70		
90° percentile	59.87		
CV(%)	180%		
number of samples	9	9	9
BDL (%)	33%	44%	100%

Other	Saccharin	Acesulfame K	Sucralose
		(ng/g)	
average	8.74		
std.dev	12.14		
min	< DL		
max	28.91		
25° percentile	0.14		
90° percentile	23.64		
CV(%)	139%		
number of samples	6	6	6
BDL (%)	33%	67%	100%

SS Di + BW	Saccharin	Acesulfame K	Sucralose
		(ng/g)	
average	-	-	-
std.dev			
min			
max			
25° percentile			
90° percentile			
CV(%)			
number of samples	2	2	2
BDL (%)	100%	50%	100%

Table SI 17: Descriptive statistic for physical impurities in COMDIG samples

Type	>20 mm plastic rigid	>20 mm plastic light	> 5 mm stones	> 2 mm plastic rigid	> 2 mm plastic light	> 2 mm stones	> 2 mm glass	> 2 mm metals
BW Co				**Impurities (g)**				
	0	0	2.8±0.326	0.050±0.010	0.162±0.020	1.493±0.363	0.025±0.005	0
				Impurities (%dm)				
	0	0	3.027	0.053	0.175	1.618	0.022	0
				Surface (cm2)				
	0	0	-	0.75	20.8	-	-	-
BW Co				**Impurities (g)**				
	0	0	0.676±0.024	0.469±0.064	0.629±0.091	2.291±0.0334	1.107±0.026	0
				Impurities (%dm)				
	0	0	0.564	0.39	0.526	1.904	0.924	0
				Surface (cm2)				
	0	0	-	5.18	134	-	-	-
BW Co				**Impurities (g)**				
	0	0	0.319±0.006	0.049±0.023	0.053±0.006	4.102±0.728	0.207±0.073	0.005±0.007
				Impurities (%dm)				
	0	0	0.197	0.03	0.033	2.532	0.127	0.003
				Surface (cm2)				
	0	0	-	2	9.8	-	-	-
BW Co				**Impurities (g)**				
	0	0	0.307±0.068	0.002±0.001	0.057±0.069	3.695±0.795	0.309±0.080	0
				Impurities (%dm)				
	0	0	0.183	0.001	0.034	2.204	0.184	0
				Surface (cm2)				
	0	0	-	0	16.4	-	-	-
Type	**>20 mm plastic rigid**	**>20 mm plastic light**	**> 5 mm stones**	**> 2 mm plastic rigid**	**> 2 mm plastic light**	**> 2 mm stones**	**> 2 mm glass**	**> 2 mm metals**
GW Co	0	0	0.925±0.266	0.014±0.00	0.005±0.003	5.583±0.363	0.083±0.039	0
				Impurities (%dm)				
	0	0	0.86	0.013	0.005	5.281	0.077	0
				Surface (cm2)				
	0	0	-	0.019	0.06	-	-	-
GW Co				**Impurities (g)**				
	0	0.102±0.030	2.697±0.776	0.148±0.034	0.010±0.002	1.493±0.558	0.121±0.053	0
				Impurities (%dm)				
	0	0.078	2.059	0.111	0.007	1.113	0.093	0
				Surface (cm2)				
	0	19.55	-	1	1.44	-	-	-

SS Co	**Impurities (g)**							
	0	0.004±0.002	0.956±0.751	0	0.008±0.001	1.435±0.265	0.051±0.036	0
	Impurities (%dm)							
	0	0.004	0.824	0	0.007	1.275	0.047	0
	Surface (cm2)							
	0	0	-	0	1.6	-	-	-
SS Co	**Impurities (g)**							
	0	0	8.067±0.714	0	0.007±0.003	2.398±0.357	0	0
	Impurities (%dm)							
	0	0	8.751	0	0.008	2.604	0	0
	Surface (cm2)							
	0	0	-	-	1.2	-	-	-
MBT Co	**Impurities (g)**							
	0	0	0	0.877±0.348	0.408±0.217	1.519±0.526	1.353±0.218	0
	Impurities (%dm)							
	0	0	0	0.867	0.401	1.528	1.355	0
	Surface (cm2)							
	0	0	-	18.8	6	-	-	-
MBT Co	**Impurities (g)**							
	0	0	0.774±0.258	0.902±0.191	0.117±0.029	1.467±0.145	0.190±0.063	0
	Impurities (%dm)							
	0	0	0.891	1.038	0.135	1.691	0.22	0
	Surface (cm2)							
	0	0	-	10.6	18	-	-	-
D	**Impurities (g)**							
	0	0	2.427±0.956	0.478±0.341	0.280±0.058	1.208±0.175	2.642±0.421	0
	Impurities (%dm)							
	0	0	2.753	0.529	0.316	1.355	2.963	0
	Surface (cm2)							
	0	0	-	6	37	-	-	-
MBT Co	**Impurities (g)**							
	0	0	0.425±0.045	0.723±0.213	0.332±0.081	2.584±0.100	0.886±0.258	0
	Impurities (%dm)							
	0	0	0.336	0.57	0.301	2.039	0.698	0
	Surface (cm2)							
	0	0	-	64	8	-	-	-
D	**Impurities (g)**							
	0	0	0	0.592±0.048	0.598±0.095	0	0	0
	Impurities (%dm)							
	0	0	0	1.086	1.376	0	0	0
	Surface (cm2)							
	0	0	-	5.95	62.8	-	-	-
MBT Co	**Impurities (g)**							
	0	0	0	0.924±0.199	0.259±0.076	0.232±0.047	0.823±0.280	0
	Impurities (%dm)							
	0	0	0	0.832	0.235	0.211	0.74	0
	Surface (cm2)							
	0	0	-	11.2	59.4	-	-	-

MBT Co	**Impurities (g)**							
	0	0	0.811±0.227	1.045±0.045	0.248±0.046	3.276±0.386	11.413±4.440	0
	Impurities (%dm)							
	0	0	0.828	1.083	0.261	3.37	11.56	0
	Surface (cm2)							
	0	0	-	15.8	19.5	-	-	-
??	**Impurities (g)**							
	0	0	0.45±0.088	0	0.004±0.002	1.161±0.136	0	0
	Impurities (%dm)							
	0	0	0.396	0	0.004	1.021	0	0
	0	0	-	-	1.1	-	-	-

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Authors: Simona Tavazzi, Giovanni Locoro, Sara Comero, Elzbieta Sobiecka, Robert Loos, Peter Eder, Hans Saveyn, Ludek Blaha, Martin Benisek, Oliver Gans, Stefan Voorspoels, Michela Ghiani, Gunther Umlauf, Giulio Mariani, Gert Suurkuusk, Bruno Paracchini, Carmen Cristache, Isabelle Fissiaux, Augustín Alonso-Ruiz and Bernd M. Gawlik

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Abstract

This report describes work conducted by the European Commission's Joint Research Centre (JRC) in the context of an Administrative Arrangement between DG Environment and the JRC.

This work aimed at the generation, within a limited timeframe, of a large amount of analytical data, with high scientific and statistical value, for a number of compost and digestate types (afterwards referred to as COMDIG samples), to help provide a general overview and estimation of that possible variability within and between different COMDIG materials.

The report includes the results of a targeted and independent screening of typical European situations of COMDIG materials with regard to the occurrence and levels of compounds of concern, many of which have never been assessed at a pan-European level.

In total, 139 samples, mostly taken as grab samples and originating from 15 countries, were assessed for 22 minor and trace elements and 92 organic compounds including ingredients of personal care products and pharmaceuticals.

The underlying analytical methods are carefully documented with regard to their performance characteristics. Where available, the so-called "horizontal" standards were followed.

The results obtained are assessed statistically.

Although the analysed single samples are insufficient to make any statement on the performance of the treatment processes leading to COMDIG samples, this collective of data provide a glimpse of the pan-European situation as regards the studied compounds.optatur, ium accaerc ipsapicatiis inima cume sapersp edipis.

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As the Commission's in-house science service, the Joint Research Centre's mission is to provide EU policies with independent, evidence-based scientific and technical support throughout the whole policy cycle.

Working in close cooperation with policy Directorates-General, the JRC addresses key societal challenges while stimulating innovation through developing new standards, methods and tools, and sharing and transferring its know-how to the Member States and international community.

Key policy areas include: environment and climate change; energy and transport; agriculture and food security; health and consumer protection; information society and digital agenda; safety and security including nuclear; all supported through a cross-cutting and multi-disciplinary approach.

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