



Project HORIZONTAL

Approach for validation studies

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Overview on materials

- 4 sludge materials from Düsseldorf, Germany;
- 1 agricultural soil material from Reading, United Kingdom;
- 1 compost material from Vienna, Austria;
- 1 compost material from Korschbroich, Germany;
- 1 sludge-amended, agricultural soil from Pavia, Italy;
- 1 sludge-amended soil from Barcelona, Spain;
- 1 sludge-amended soil from Essen, Germany;
- 1 long-term sludge exposed soil from Hohenheim, Germany

Overview on levels

Joint Research Centre

	Sludge 1	Sludge 2	Soil Reading	Compost Vienna	Soil Pavia	Soil Barcelona	Soil Essen	Soil Hohenhm.	Compost NRW
Trace elements	+++	+++	++	++	++	++	+	++	*/*
Hg	*/*	*/*	+	++	+	+	+	+++	*/*
AOX	++	++		+	*/*	*/*	*/*	*/*	*/*
PAH	++	+++	+++	++	*/*	*/*	+	*/*	*/*
PCB	++	++	-	+	*/*	*/*	*/*	*/*	*/*
NP+E	++	++	*/*	+	*/*	*/*	*/*	*/*	*/*
LAS	+++	+++	*/*	*/*	*/*	*/*	*/*	*/*	*/*
DEHP	+++	+++	*/*	+	+	+	+	++	*/*
PBDE	+++	+++	+	+	+	++	+	+	*/*
PCDD	+++	+++	*/*	+	+	*/*	*/*	*/*	*/*



Overview on participants

Parameter	Participants	Countries	Parameter	Participants	Countries
Dry Matter	43	14	PAH	30	11
LoI	30	11	PCB	21	11
TOC	35	10	NP+E	14	8
EC	27	11	LAS	8	6
pH	36	13	DEHP	13	8
Kjel.N	27	10	PBDE	11	9
P	32	12	PCDD	8	7
N	27	11	Weeds	6	5
NH4/NO3	27	11	Impurities	4	4
Aqua regia d.	36	13	GFAAS	16	8
Nitric acid d.	24	12	FCVAAS	27	12
ICP-AES	42	14	Fluorimetry	2	2
ICP-MS	19	8	AOX	15	8



PAH
PCB
NP+E
LAS
DEHP
PBDE
PCDD

Complexity group 1

Proposal for validation

- 1 Soil, 1 Compost, 1 Sludge;
- 2 individual units to be tested;
- 2 independent measurements per unit;
- $3 \times 2 \times 2 = 12$ measurement results per laboratory;
- Only 4 measurement results per material;



Aqua regia d.
Nitric acid d.
ICP-AES
ICP-MS
GFAAS
FCVAAS

Complexity group 2

Proposal for validation

- 2 Soils, 1 Compost, 2 Sludges;
- 2 individual units to be tested;
- 3 independent measurements per unit;
- $5 \times 2 \times 3 = 30$ measurement results per laboratory;
- 6 measurement results per material;



Complexity group 3

Dry Matter

LoI

TOC

EC

pH

Kjel.N

P

N

NH₄/NO₃

AOX

Proposal for validation

- 2 Soils, 2 Composts, 2 Sludges;
- 2 individual units to be tested;
- 3 independent measurements per unit;
- $6 \times 3 \times 2 = 36$ measurement results per laboratory;
- 6 measurement results per material;



Questions for discussions

- ➡ Selection of materials based on the available analytical information;
- ➡ Congeners/compound class members to be considered;
- ➡ Time available for participants to submit results;
- ➡ Groups with weak strength;
- ➡ Submission of instructions;
- ➡ Questions to be asked to the participants;
- ➡ Material needs per group (planning of dispatch);
- ➡ Miscellaneous.



Validation design

Round 1 (Attn. Contractual Obligations to be finished within Mar. 2007)

- Validation of Complexity Group 1;
- 1 Soil (Barcelona), 1 Compost (Vienna), 1 Sludge (Essen);
- 2 individual units to be tested, 2 independent measurements per unit, $3 \times 2 \times 2 = 12$ measurement results per laboratory
- Results to be submitted online using MILC Facility from IRMM;
- Statistical evaluation per guidance compound AND using outlier testing e.g. Cochran, Grubbs etc.
- Questionnaire to the participants.
- Samples to be dispatched within Oct. 2006.
- Time for analysis reserved: 2 Month (i.e. until End of Dec. 2006).
- First Evaluation Report Draft for consultation by CEN: End of Jan. 2007

Validation design

Round 2 (Attn. Contractual Obligations to be finished within Mar. 2007)

Validation of Complexity Group 2 and 3;

- 2 Soils, 2 Compost, 2 Sludges;
- 2 individual units to be tested, 3 independent measurements per unit, $5 \times 2 \times 3 = 30$ measurement results per laboratory in case of Complexity group 2, 6 measurement results per material; Results to be submitted online using MILC Facility from IRMM;
- Statistical evaluation per guidance compound AND using outlier testing e.g. Cochran, Grubbs etc.
- Questionnaire to the participants.
- Samples to be dispatched within Oct/Nov. 2006.
- Time for analysis reserved: 2 Month (i.e. until Jan. 2007).
- First Evaluation Report Draft for consultation by CEN: End of Feb. 2007



Overview on Material Dispatch Sequence

- **Sludge 1 (Dispatch 1 and 2);**
- **Sludge 2 (only Dispatch 2);**
- **Compost Vienna (Dispatch 1 and 2);**
- **Compost NRW (only Dispatch 2);**
- **Soil Reading (Only Dispatch 2);**
- **Soil Pavia (Only Dispatch 2);**
- **Soil Barcelona ((Dispatch 1 and 2);**
- **Soil Essen (Only Dispatch 2);**
- **Soil Hohenheim (Only dispatch 2).**

Dispatch 1: within Oct. 2006;

Dispatch 2: within Oct/Nov. 2006;