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**STANDARDISATION MANDATE TO CEN FOR THE DEVELOPMENT OF  
HORIZONTAL STANDARDS  
IN THE FIELDS OF SLUDGE, BIOWASTE AND SOIL**

**I. MOTIVATION**

The objective of this Mandate is to develop horizontal European standards in the fields of sludge, biowaste and soil. These are needed to implement existing and upcoming EU Directives, such as

- The revision of the Sewage Sludge Directive 86/278/EEC;
- The upcoming Directive on the biological treatment of biodegradable waste foreseen for 2004;
- The initiative on soil monitoring to be proposed by 2004;

At a workshop on “Harmonisation of sampling and analysis methods for heavy metals, organic pollutants and pathogens in soil and sludge”<sup>1</sup> held in Stresa in February 2001 with participants active in a number of environmental CEN Technical Committees (TCs), it was stressed that horizontal standardisation is very much needed to avoid unnecessary duplication of work. It is the opinion of the Commission services that in future every attempt should be made to develop horizontal standards that are suitable for a wide range of materials and lead to equivalent results as far as this is technically feasible.

This Mandate considers standards on sampling and analytical methods for hygienic and biological parameters as well as inorganic and organic contaminants.

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<sup>1</sup> See the workshop proceedings in H Langenkamp, L Marmo (eds), Harmonisation of sampling and analysis methods for heavy metals, organic pollutants and pathogens in soil and sludge, JRC, EUR 19809 EN, 2001 (<http://europa.eu.int/comm/environment/waste/sludge/index.htm>).

Part of the work will focus on horizontal standardisation starting from existing standards developed for the same parameters in the fields of sludge, biowaste and soil. Another part of the work will focus on the development of standards not available at this point in time.

Any activity in testing of materials begins with sampling. This is an often undervalued aspect of analysis. In this Mandate, it will receive due attention as it may contribute significantly to the overall inaccuracy in the outcome of an analysis.

Compounds to be considered in this Mandate are selected according to their relevance as frequent contaminants in sludge, biowaste and soils in Europe. They are grouped into classes according to their physical/chemical properties, which in turn determine the methods needed to quantify the potential impact on human and animal health, plant uptake, soil function and groundwater quality. As the materials generally feature a mixture of different types of contaminants, it is important to provide an integral answer covering an evaluation of all relevant pollutants:

The Commission has supported the formation of a Consortium of research institutes, experts in standardisation, members of the academic world and regulatory agencies called "Horizontal". This Consortium is responsible for carrying out the background work that would allow the adoption of standards through the normal procedure of CEN. This background work includes the development of desk studies to evaluate state-of-the-art in standardisation for the different parameters concerned; harmonisation of existing standards with a view to developing horizontal protocols for sampling and analysis; and research for those parameters for which no standardisation method yet exists. This work is carried out under the guidance of a Steering Committee formed by representatives of the Commission and of the Member States financing the Consortium.

This work is being carried out in close co-operation with CEN/TCs (indeed, most of the members of the Consortium are active in the environmental TCs) and with the financial support of a large number of Member States, DG Environment, the Joint Research Centre of the Commission and, where possible, DG Research.

The objective of the Consortium is to carry out pre-normative research and develop draft texts in the form of prENs (draft European standards) for the parameters concerned. These draft texts in the form of prENs will then be validated prior to be submitted to CEN in order to be formally adopted under CEN rules.

This is an ambitious goal that requires the active participation of all concerned, in particular CEN. A Mandate to CEN is needed to develop horizontal standards to be employed for the purposes of the revision of Directive 86/278/EEC, the adoption of a Proposal for a Directive on the biological treatment of biodegradable waste and an initiative on soil monitoring. This will allow CEN to concentrate their work, thus speeding up the completion of the required standardised methods.

## **II. DESCRIPTION OF THE MANDATED WORK**

The Commission entrusts CEN to develop standards as described in the Annexes.

## **III. EXECUTION OF THE MANDATE**

The standards shall be developed in accordance with the requirements as stated in the Annexes. To the extent possible, existing standards for the acceptance of waste at landfills should be taken into consideration.

Within the overall frame of this Mandate, the Commission services may invite CEN to modify the Annex I and Annex II in order to ensure that the work to be carried out reflects the standardisation needs established by the progressive development of relevant Directives as well as the technical possibilities determined by research work.

These changes to the Annexes shall also be made with respect to the timeliness of the delivery of draft texts in the form of prENs by the Consortium.

The Consortium should strive to combine parameters and properties mentioned in Annex I where technically possible<sup>2</sup> and unless there are specific reasons to the contrary, so that the number of standards can be reduced without reducing the number of parameters standardised.

Validation of the method contained in the draft texts in the form of prENs shall be carried out by the Consortium before transmission to CEN with a view to be adopted as ENs (European standards).

CEN shall send to the Commission, within three months of their adoption, the standards in three linguistic versions (English, French and German) and the titles of these standards in the other languages of the European Union. This information shall be submitted on paper and electronic support.

The standstill period referred to in Article 7 of Directive 98/34/EC (OJ L 217, 5.8.1998, p. 18) shall commence when CEN accepts this standardisation Mandate.

#### **IV. ANNEXES**

The two Annexes form an integral part of this Mandate.

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<sup>2</sup> For instance, potassium and phosphorus are mentioned separately in Annex I. However, they could be measured quite nicely with ICP together with a whole bunch of other parameters.

## ANNEX I

For the development of the standards listed in Annex II the following timetable is foreseen (parameters for which a European standard (EN) already exists):

|                                       | <b>Date of delivery</b>              |   |   |  |
|---------------------------------------|--------------------------------------|---|---|--|
|                                       | <b>Desk studies<br/>(Consortium)</b> | <b>Draft texts in<br/>the form of<br/>prEN or prTS<br/>(Consortium)</b> | <b>Validation of<br/>draft texts in<br/>the form of<br/>prEN<br/>(Consortium)</b> | <b>Publication<sup>1</sup><br/>(CEN)</b> |
| Sampling                              | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| pH                                    | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Dry matter                            | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Organic matter                        | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Bulk density                          | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Electrical conductivity               | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Extraction of trace elements          | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Determination of trace elements       | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Secondary nutrients & micro-nutrients | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Total nitrogen (Kjeldahl, Dumas, etc) | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Ammonium-nitrogen                     | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Nitrate-nitrogen                      | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Total phosphorus                      | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Bicarbonate extractable P             | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Potassium                             | 9/2003                               | 9/2004  | 9/2005  | 9/2006 (EN)                              |
| Leaching compliance                   | 9/2003                               | 9/2004  | -   | 6/2005 (TS)                              |
| Leaching characterisation             | 9/2003                               | 9/2004  | -   | 6/2005 (TS)                              |

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<sup>1</sup> As European standard (EN) or Technical Specification (TS).

For the development of the standards listed in Annex II the following timetable is foreseen (parameters for which a European standard (EN) does not yet exists):

|   | Date of delivery             |   |  |                                   |
|---|------------------------------|---|--|-----------------------------------|
|   | Desk studies<br>(Consortium) | Draft texts in<br>the form of<br>prEN or prTS<br>(Consortium) | Validation of<br>draft texts in<br>the form of<br>prEN<br>(Consortium) | Publication <sup>2</sup><br>(CEN) |
| Stability index                                       | 9/2003                       | 9/2004  | 9/2005   | 12/2007 (EN)                      |
| Germinating weed seeds                                | 9/2003                       | 9/2004  | 9/2005   | 12/2007 (EN)                      |
| Impurities<br>(plastic, glass etc.)                   | 9/2003                       | 9/2004  | 9/2005   | 12/2007 (EN)                      |
| <i>Salmonella senftenberg</i><br>W775                 | 9/2003                       | 9/2005  | -  | 6/2006 (TS)                       |
| <i>Salmonella spp.</i>                                | 9/2003                       | 9/2004  | 9/2005   | 12/2007 (EN)                      |
| <i>Escherichia coli</i>                               | 9/2003                       | 9/2004  | 9/2005   | 12/2007 (EN)                      |
| <i>Clostridium perfringens</i>                        | 9/2003                       | 9/2004  | 9/2005   | 12/2007 (EN)                      |
| AOX   | 9/2003                       | 9/2005  | -  | 6/2006 (TS)                       |
| PAH   | 9/2003                       | 9/2004  | 9/2005   | 12/2007 (EN)                      |
| PCB   | 9/2003                       | 9/2004  | 9/2005   | 12/2007 (EN)                      |
| LAS   | 9/2003                       | 9/2005  | -  | 6/2006 (TS)                       |
| DEHP  | 9/2003                       | 9/2005  | -  | 6/2006 (TS)                       |
| NPE   | 9/2003                       | 9/2005  | -  | 6/2006 (TS)                       |
| PCCD/F  | 9/2003                       | 9/2004  | 9/2005   | 12/2007 (EN)                      |
| Flowability   | 9/2003                       | 9/2005  | -  | 6/2006 (TS)                       |
| Solidity<br>Thixotropic behaviour<br>Piling behaviour | 9/2003                       | 9/2005  | -  | 6/2006 (TS)                       |

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<sup>2</sup> As European standard (EN) or Technical Specification (TS).

## HORIZONTAL STANDARDS IN THE FIELDS OF SLUDGE, BIOWASTE AND SOIL

### 1. BACKGROUND INFORMATION

Compounds to be considered in this Mandate are selected according to their relevance as frequent contaminants in sludge, biowaste and soils in Europe. They are grouped into classes according to their physical/chemical properties, which in turn determine the methods needed to quantify the potential impact on human and animal health, plant uptake, soil function and groundwater quality. As the materials generally feature a mixture of different types of contaminants, it is important to provide an integral answer covering an evaluation of all relevant pollutants:

- Inorganic compounds such as **heavy metals** (e.g. Cd, Cu, Pb, Zn), oxyanions (e.g. As, Mo, Se, Cr), and **nutrients** (N, P);
- Volatile to semi-volatile compounds (chlorinated compounds etc.);
- **Strongly sorbing, non-volatile compounds** with relatively low water solubility such as 3- to 6-ring polycyclic aromatic hydrocarbons, polychlorinated biphenyls, phthalates;
- **Soluble non-volatile organic compounds** such as oxygenated and heterocyclic compounds;
- The Mandate includes the most frequently occurring pollutants in sludge, treated biowaste and treated soils such as heavy metals and **PAHs** (polycyclic aromatic hydrocarbons). Procedures developed for the compounds investigated in the Mandate can also be applied to other compounds with similar physico-chemical properties such as **PCBs** (polychlorinated biphenyls) and endocrine disruptors such as phthalates.
- A list of **hygienic parameters** to be examined will also be needed to assure the sanitisation of sludge and treated biowaste. CEN/TC308/WG1 has actively started to draft methods for the hygienic parameters likely to be included in future Directives. The selected methods are adapted from standard methods for the examination of water. To ensure that fit for purpose microbiological results can be obtained for a wide range of materials co-normative research work is necessary to develop suitable standards. The validation of these standards will be achieved by carrying out interlaboratory trial(s) with participation of a number of experienced European laboratories. Such validation requires application of the draft standards to a wide range of real sludge and bio-solid samples.

It is not only necessary to make methods available to determine specific micro-organisms, but also to reach a detailed protocol for validating plant performance and end product specification in terms of hygienic microbiological parameters. This will include co-and pre-normative research, including method validation for complementary bacterial indicators (*Enterococci* and *Clostridium perfringens*), and parasites (helminth ova). For parameters likely to be included in future Directives (i.e.

*E. coli*, *Salmonella* and *Clostridium perfringens*), the selected methods will be assessed in large interlaboratory trials involving many European countries. For other parameters, there is a need to develop preliminary standards in order to carry out the relevant research.

In many Directives a reference to the physical consistency (liquid, solid, thixotropic, piling behaviour, etc.) is reported as a characteristic to be evaluated for fulfilling the regulations requirements. Further, in many analytical methods for sludges (pH, dry matter, leaching, etc.) different procedures are indicated depending on whether a sample is liquid or solid. However, no procedures are given for evaluating these **mechanical properties**. Some national methods and industrial methods are existing, which have been surveyed by the partners of WP7. Pre-normative studies and research will be done to draft harmonised European standards in the field of this Mandate.

In CEN TC 292 standards have been developed for the characterisation of waste. Many of these methods are implemented in the Council Decision 2003/33/EC establishing criteria and procedures for the acceptance of waste at landfills<sup>1</sup>. Tests at three levels have been defined – characterisation, compliance and on-site verification. Sludge and treated biowaste destined for landfill will have to be assessed by these methods in the future. These standards include methods for:

- extraction of trace elements and determination of trace elements from acid digested samples,
- dry matter/moisture content , organic matter (TOC),
- preparation of eluates,
- determination of pH, conductivity, ammonium, nitrite, TOC in eluates.

Recently, the validations of TOC (EN 13137), extraction of trace elements (EN 13657), and of a compliance leaching test (EN 12457 Parts 1 – 4) have been completed in a Mandate financed by Ministries of several Member States and national funding agencies as a joint effort between CEN/TC 292 and CEN/TC 308. In the process of horizontal standardisation the existing methods for digestion, leaching and analysis need to be evaluated for their suitability for biowaste. Because of the wide variety of waste materials and the results of the validation experiments where different matrices, including sludge, were tested, it may be supposed that these standards will also be applicable for biowaste.

## **2. STRUCTURE OF THE WORKPLAN**

The work is split up in coherent Work Packages (WPs), which each address a main aspect of all relevant standards required or likely to be required in the development of Directives. Relevant samples for the experimental work will be centrally prepared.

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<sup>1</sup> Council Decision 2003/33/EC of 19 December 2002 establishing criteria and procedures for the acceptance of waste at landfills pursuant to Article 16 of and Annex II to Directive 1999/31/EC (OJ L 11, 16.1.2003, p. 27).

Draft standards will be prepared for the following:

- sampling of sludge, treated biowastes and soils;
- enumeration of *E. coli*, *Salmonella*, *Enterococci*, *Clostridium perfringens*, and viable helminth ova in sewage sludge and bio-waste and comparable matrices;
- physical impurities in treated biowaste, weed seeds and plant propagules, the stability of treated biowaste (respiration method);
- determination of AOX, PCB, PAH, Linear Alkyl Sulfonates (LAS), Nonyl Phenol (NP) and endocrine disrupters like phthalates and compounds such as Diethylhexylphthalate (DEHP) and Dibutylphthalate (DBP);
- pH, dry matter/moisture content, organic matter/loss on ignition, total nitrogen, ammonium nitrogen, total phosphorus; extraction of trace elements and determination of trace elements from acid digested samples;
- flowability, solidity, thixotrophy, stacking behaviour.

All of these methods will contain the results of international inter-laboratory performance evaluation according to ISO 5725. Inter-comparison studies are centrally organised.

### **3. DETAILED DESCRIPTION OF THE WORKPLAN**

#### **3.1. Work package 1: Sample collection and preparation**

The materials to be studied will be different types of sludges, compost and sewage sludge treated soils. The main work in this WP will be to sample, prepare and analyse the materials for suitability in the different WP's. When materials prove to be suitable, they will be dispatched to the participating organisations.

##### *3.1.1. Identification of suitable testing sites for sludge and treated biowaste (soils are identified by work package 2)*

Materials to be tested must be selected such that the materials can be considered sufficiently representative for sludges and treated biowaste as produced in Europe. Relevant materials are:

##### Sludge material:

- Wet sewage sludge, part a) de-watered sludge, part b) liquid fraction
- Air-dried sewage sludge (same plant as the wet sewage sludge)
- Wet water works sludge from the drinking water industry
- De-watered water works sludge (same plant as 3)
- Sludge from the paper and pulp industry



- Lime-stabilised sludge
- Synthetic Sludge (specification will be given by work package 6)

Compost material:

- Mixed biowaste including gardening and household-derived biowaste
- Mixed biowaste from gardening (we had concluded at least two would be needed)

Sewage sludge treated soils:

- Organic rich, low clay content soil
- Calcareous, low organic content soil
- Clay rich, low organic and high iron content soil
- *Medium clay content (10-25%), low organic and low iron content*
- Coarse textured (low clay content) low organic content soil

*3.1.2. Characterisation by analyses at these testing sites for relevant properties to fall in a suitable range*

Prior to making a large effort to prepare materials for ruggedness testing and validation, it must be ensured that the relevant parameters for which the materials will be analysed fall in the measuring range as well as in the range of interest.

*3.1.3. Sampling of the necessary material from the selected sites (typically several 100 kg)*

Once the proper materials have been identified fairly large amounts of material need to be collected and stored properly. Storage is a key issue for “unstable” materials such as sludge and treated biowaste.

*3.1.4. Production of test material for work packages 4, 5 and 6*

Once the proper materials have been identified large amounts of material need to be processed to provide a sufficient amount of reasonably homogeneous material for the required experimental validation work. Rigorous homogenisation is not anticipated as the results obtained from the validation work shall reflect as much as possible normal operation procedures and not the thorough effort of preparing a standard reference material. Large portions of material are prepared for experimental development work and ruggedness testing.

Note: For some parameters samples must be analysed directly after collection as such parameters may not be stable. Material for such studies (e.g. hygienic parameters) will be collected with the programme of the WP.

3.1.5. *Production of a set of sludge, compost and soil materials to be used in the validation studies (WPs 3 to 8) – limited validation in phase I*

Preparation of laboratory samples for validation studies on the respective parameters in the programme. For some parameters sample collection and preparation may need to be done shortly before dispatch to participating laboratories and the window for validation testing (e.g hygienic parameters, biological parameters, some organic contaminants) may have to be set quite narrow to ensure comparable results.

3.1.6. *Characterisation of the material according to specified quality criteria with respect to stability and homogeneity of the analyses between different laboratory samples (bottles)*

Prior to dispatch of materials, a quality check on laboratory samples by the lab dispatching the samples will be necessary to identify the variability within charges sent out in validation.

3.1.7. *Distribution of the samples to the laboratories as agreed by the Mandate research and validation programmes*

Timely dispatch of laboratory samples to laboratories participating in validation work after a draft standard has been agreed by relevant CEN and ISO bodies.

## **3.2. Work package 2: Sampling**

In this phase the experimental work will be very limited. The main emphasis will be on the literature review of the latest developments in sampling of soil, sludge, treated biowaste and related fields. In addition, evaluating the existing sampling protocols for the different purposes of use of materials aimed at preparing draft protocols for sampling of soil, sludge and treated biowaste from different practical situations (pile, belt, field) and for different purposes. Irrespective of the precision of laboratory methods, the overall precision of the final analytical results can be no better than the precision associated with sampling and sample handling.

3.2.1. *Literature review and critical evaluation of work on sampling protocols and standards resulting in draft sampling protocols for discussion in relevant CEN and ISO bodies*

This work will consist of a critical review of sampling standards as available today in the field of soil, sludge, treated biowaste and waste to derive draft sampling protocols for the different sampling situations (in-situ sampling and sampling from tanks, piles or belts) relevant to soil, sludge and treated biowaste. In order that data are comparable it is essential that there is harmonisation of the way in which soils and biomaterials are sampled. Treated biomaterials may occur as heaps or as flowing streams (in pipes or on conveyers, etc.). The properties of soils (in the landscape) might vary across the sampled area and also with depth. The aim is to develop protocols for sampling and sample-handling that are practicable and that do not impose a financial or administrative burden that is disproportionate to risk against the benchmark of the most representative samples that can be obtained irrespective of cost.

### 3.2.2. *Experimental design for comparing a limited selection of sampling strategies for sludges*

To facilitate the discussion in CEN and ISO bodies on sampling needs and issues, an experimental design will be provided for the sampling of sludge using a limited number of sampling strategies. Based on such examples the need for additional research in the next phase of the work can be focussed.

### 3.2.3. *Experimental design for comparing a limited selection of sampling strategies for treated biowastes*

To facilitate the discussion in CEN and ISO bodies on sampling needs and issues, an experimental design will be provided for the sampling of treated biowaste using a limited number of sampling strategies. Based on such examples the need for additional research in the next phase of the work can be focussed.

### 3.2.4. *Experimental design for comparing a limited selection of sampling strategies for soils*

To facilitate the discussion in CEN and ISO bodies on sampling needs and issues, an experimental design will be provided for the sampling of sludge using a limited number of sampling strategies. Based on such examples the need for additional research in the next phase of the work can be focussed.

### 3.2.5. *Research report on the relationship of sampling protocols to the analytical results obtained for a limited selection of soils and treated biomaterials (biowastes and sludges) in England and environmental significance as measured by uptake of trace elements into cereal grains*

The only way to evaluate the performance of a sampling is to analyse the samples. To provide a basis for discussion actual experimental work is needed to illustrate possibilities and limitations in drafting sampling protocols. Samples will be extracted with aqua-regia (EN 13650:2001), nitric acid (Nordic method) or calcium chloride/DTPA (CAT) (EN 13651:2001) and the extracts analysed for Cu, Ni, Zn, Cd, Pb, and Cr using ICP. These elements have been chosen because they are regulated by the Sludge Directive and can be analysed relatively inexpensively by multi-element analysis using ICP. Soil pH will be measured in water and in calcium chloride solution; these are the predominant alternative methods for arable soils in the EU (see report of Stresa Workshop, February 2001). An assessment of the environmental relevance of soil samples will also be investigated by multi-element analysis of grain samples taken at the same time and places as some of the soil samples.

## **3.3. Work package 3: Hygienic parameters**

In the Sludge Directive *E. Coli*, *Salmonella* and *Clostridium perfringens* are specifically mentioned. This leads to the logical choice to start the work on these parameters in phase I. For the other parameters desk studies to prepare draft protocols for CEN and ISO discussion are prepared.

### 3.3.1. *Desk studies on feasibility of horizontal standards for E. coli and Salmonella*

In a desk study, the latest developments on assessing E.coli and Salmonella in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### 3.3.2. *Draft horizontal standard for E. coli enumeration in sewage sludge and bio-waste after comparison of methods and ruggedness testing*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on *E. coli* enumeration will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation.

### 3.3.3. *Draft standards for Salmonella enumeration in sewage sludge and bio-waste with ruggedness testing*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on Salmonella enumeration will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation.

### 3.3.4. *Desk study for a draft horizontal standard for Clostridium perfringens enumeration*

In a desk study, the latest developments on assessing Clostridium perfringens in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### 3.3.5. *Experimental work in preparation of a draft horizontal standard for Clostridium perfringens enumeration*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on *Clostridium perfringens* enumeration will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation.

### 3.3.6. *Interlaboratory trial on E. Coli, Salmonella and Clostridium*

European validation of the draft horizontal standards for *E. coli*, *Salmonella* and *Clostridium* will be carried out after consultation of the relevant CEN and ISO bodies using materials centrally prepared and dispatched. In view of the limited stability of samples of sludge and treated biowaste, special measures in terms of sample dispatch and timing of the experimental work by participating laboratories are required to ensure a proper validation.

### 3.3.7. *Desk study on a draft horizontal standard for Helminth ova*

In a desk study, the latest developments on assessing Helminth ova in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### 3.3.8. *Desk studies on feasibility of horizontal standard for Enterococci*

In a desk study, the latest developments on assessing *Enterococci* in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### 3.3.9. *Desk study on rapid methods for hygienic parameters*

In a desk study, the suitability of rapid methods for assessing hygienic parameters in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### 3.3.10. *Literature review on levels of pathogens and abatements*

In a desk study, a literature review is provided on the level of pathogens and abatements for them in the field of sludge, soil and treated biowaste.

## **3.4. Work package 4: Biological parameters**

In the Sludge Directive, germinating weed seeds are identified as parameters for which a standard is required. This topic is therefor taken as priority item. For the other two issues originally identified under biological parameters the work in this phase is limited to desk study and draft protocol preparation.

### 3.4.1. *Desk study with an update on methods for impurities in sludge and treated bio-waste including a draft standard for discussion in relevant CEN and ISO*

In a desk study, the latest developments on assessing impurities in the fields of sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### 3.4.2. *Desk study with an update on methods for measuring the stability index in sludge and treated bio-waste including a draft standard for discussion in relevant CEN and ISO bodies*

In a desk study, the latest developments on assessing degradability using a stability index in the fields of sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between

different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

*3.4.3. Desk study to prepare a draft horizontal standard for germinating weed seeds in sludge and treated bio-waste for discussion in relevant CEN and ISO bodies*

In a desk study, the latest developments on assessing germinating weed seeds in the fields of sludge, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

*3.4.4. Experimental work for, preparation and justification of a draft European standard method for viable weed seed and plant propagules in sludge and treated biowaste and a draft method for testing for the presence of phytotoxic factors*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on viable weed seed and plant propagules will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation after consultation of the relevant CEN and ISO bodies. The validation itself will not be part of phase I.

### **3.5. Work package 5: Organic parameters**

Several organic contaminants are important in sludge, soil and biowaste. For this reason, all relevant parameters were selected to prepare draft standards for discussion in CEN and ISO bodies.

*3.5.1. Desk study for a horizontal European standard method for determining (7) selected PCB in sludge, soil and solid waste*

In a desk study, the latest developments on assessing PCB in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation. Based on discussions in CEN/TC 292, CEN TC 308 and ISO 190 it was decided to focus on seven congeners: 28, 52, 101, 118, 138, 153 and 180 for the determination of PCB. In an annex six additional congeners (PCB 18, 31, 44, 149, 170, 194) are entered.

*3.5.2. Draft horizontal standard method for determining (7) selected PCB in sludge, soil and solid waste for discussion in relevant CEN and ISO bodies*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on PCB congeners will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European

validation after consultation of the relevant CEN and ISO bodies. The European validation is not part of Phase I.

*3.5.3. Desk study for a horizontal European standard method for determining 16 PAH in sludge, soil and solid waste*

In a desk study, the latest developments on assessing PAH (16) in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

*3.5.4. Draft European standard method for determining 16 PAH in sludge, soil and solid waste for discussion in relevant CEN and ISO bodies*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on 16 PAH will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation after consultation of the relevant CEN and ISO bodies. The European validation is not part of Phase I.

*3.5.5. Desk study for a horizontal European standard for determination of LAS in sewage sludge and comparable matrices*

In a desk study, the latest developments on assessing LAS (Linear Alkyl Sulfonates) in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

*3.5.6. Experimental work to prepare a draft horizontal European standard for determination of LAS in sewage sludge and comparable matrices for discussion in relevant CEN and ISO bodies*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on the determination of LAS will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation after consultation of the relevant CEN and ISO bodies. The European validation is not part of Phase I.

*3.5.7. Desk study for a horizontal European standard for determination of AOX in sewage sludge and comparable matrices*

In a desk study, the latest developments on assessing AOX in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

3.5.8. *Draft European standard method for determination of AOX in sewage sludge and comparable matrices for discussion in relevant CEN and ISO bodies*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on AOX will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation after consultation of the relevant CEN and ISO bodies. The European validation is not part of Phase I.

3.5.9. *Desk study for a horizontal European standard for determination of NP in sewage sludge and comparable matrices*

In a desk study, the latest developments on assessing NP (NonylPhenol) in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

3.5.10. *Experimental work to prepare a horizontal European standard for determination of NP in sewage sludge and comparable matrices*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on the determination of NP will be carried out to evaluate critical aspects in the draft protocol. This work will lead to a first draft horizontal standard that may require further work before it can be carried forward for European validation after consultation of the relevant CEN and ISO bodies. The European validation is not part of Phase I.

3.5.11. *Desk study for a horizontal European standard for determination of DEHP in sewage sludge and comparable matrices*

In a desk study, the latest developments on assessing DEHP (Di-ethylhexylphthalate) in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

3.5.12. *Experimental work to prepare a draft horizontal European standard method for determination of DEHP in sewage sludge and comparable matrices*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on the determination of DEHP will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation after consultation of the relevant CEN and ISO bodies. The European validation is not part of Phase I.

### **3.6. Work package 6: Inorganic parameters**

The inorganic parameters are all listed in the Sludge Directive and will be relevant for the draft Biowaste directive. This implies that all aspects need to be covered.



### *3.6.1. Desk study to assess the feasibility of a draft horizontal standard for pH*

In a desk study, the latest developments on assessing pH in eluates derived from sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### *3.6.2. Experimental work on robustness/comparability – pH, consultation of CEN and ISO bodies and validation*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on the determination of pH will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European consultation of the relevant CEN and ISO bodies.

### *3.6.3. Desk study to assess the feasibility of a draft horizontal standard for DM/LOI/TOC*

In a desk study, the latest developments on assessing DM(dry matter), LOI (loss on ignition) and TOC (total organic carbon) in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### *3.6.4. Desk study to assess the feasibility of a draft horizontal standard for nutrients (Total P and N)*

In a desk study, the latest developments on assessing nutrients in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### *3.6.5. Ruggedness work to evaluate methods and develop a draft horizontal standard for nutrients (total P and N)*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on the determination of nutrients will be carried out to evaluate critical aspects in the draft protocol. This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation after consultation of the relevant CEN and ISO bodies. The European validation is not part of Phase I.

### *3.6.6. Report on feasibility for harmonisation of standards for determination of trace elements*

In a desk study, the latest developments on assessing total elements in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between methods and use in different fields, evaluate

to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

### *3.6.7. Evaluation of methods and robustness/comparability of -“total” trace elements including chemical analysis aspects*

Based on the evaluation in the desk study and the results of consultation of the relevant CEN and ISO bodies experimental work on the determination of total trace elements including the chemical analysis will be carried out to evaluate critical aspects in the draft protocol(s). This type of ruggedness testing shall lead to a draft horizontal standard that can be carried forward for European validation after consultation of the relevant CEN and ISO bodies. The European validation is not part of Phase I.

## **3.7. Work package 7: Mechanical properties**

Although of importance to sludge soil and biowaste handling, the mechanical properties were not considered highest priority for Phase I. To facilitate the development of horizontal standards in this field desk studies are foreseen to evaluate existing information and ongoing work and prepare draft standards for discussion.

### *3.7.1. Desk study with update of methods on Physical consistency, Solidity, Thixotrophy, Piling behaviour*

In a desk study, the latest developments on assessing Physical consistency, Solidity, Thixotrophy, Piling behaviour in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome.

### *3.7.2. Preparation of first draft standards on Physical consistency, Solidity, Thixotrophy, Piling behaviour for discussion in relevant CEN and ISO TCs and WGs*

Preparation of first draft standards on Physical consistency, Solidity, Thixotrophy, Piling behaviour for discussion in relevant CEN and ISO TCs and WGs to facilitate horizontal standardisation. Identification of research needs.

### *3.7.3. Desk study with update of methods on Flowability*

In a desk study, the latest developments on assessing Flowability in sludge, soil, treated biowaste and neighbouring fields are evaluated. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome.

### *3.7.4. Preparation of first draft standards on Flowability for discussion in relevant CEN and ISO TCs and WGs*

Preparation of first draft standards on Flowability for discussion in relevant CEN and ISO TCs and WGs to facilitate horizontal standardisation. Identification of research needs.

### **3.8. Work package 8: Leaching**

In relation to impact of treated soil, sludge and biowaste on sub-soil and groundwater quality as well as in relation to such materials not meeting requirements and thus destined for landfill, leaching test are needed according to the relevant regulatory framework. The experience gained in leaching of waste can be largely adopted.

#### *3.8.1. Feasibility study on horizontal standardisation for leaching of soil, sludge and bio-waste*

In a desk study, the latest developments on assessing leaching from in sludge, soil, treated biowaste and neighbouring fields in case such materials destined for disposal are evaluated. Validation of contaminated soil has already been carried out in connection with methods development in CEN/TC 292 and CEN/TC 308. Identification of key points where possible differences exist between different fields, evaluate to what extent such perceived differences can be overcome and a draft standard prepared with annotations to facilitate horizontal standardisation.

#### *3.8.2. Preparation of a work plan for validation of a leaching test for sludge, soil and biowaste*

Based on previous experience a work plan for validation of a leaching test for sludge and biowaste can be drafted in line with horizontal standards development between CEN/TC 292, CEN/TC 308 and ISO/TC190/SC7/WG6.

### **3.9. Work package 9: Data handling & interpretation**

For the validation work to be carried out, a uniform approach is needed. This aim is best served by dealing with validation studies centrally. In addition, guidance drafting horizontal standards is needed.

#### *3.9.1. Preparation of electronic data reporting sheets and reports*

To facilitate uniform data handling, easy access and statistical evaluation data reporting sheets for validation in different parts of the experimental program are foreseen. The presentation of statistical data in standards to be developed can be harmonised in this manner.

#### *3.9.2. Prepare and ensure a basis for internal quality of data within the Mandate*

To ensure quality of analysis and quality of standards minimum requirements in reporting and guidance for writing horizontal standards shall be provided. The drafting of this new category of standards in CEN will require adjustments to the existing system, particularly with respect to referencing other normative standards and to standards forming a logical chain (e.g. sampling, pre-treatment, extraction, analysis). To ensure quality of analysis between laboratories, standard reference materials or similar internal quality control materials will be provided along with the laboratory samples to be tested in validation trials.

### 3.9.3. *Preparation, co-ordination and harmonisation of the different types of validation work needed within the Mandate*

Assistance will be given to WP leaders responsible for validation of methods in their area in preparation, co-ordination and harmonisation of validation approaches within the programme.

## **4. VALIDATION OF STANDARDS**

Validation of any method contained in a draft text in the form of a prEN is a crucial element of standardisation in order to be able to judge the performance characteristics of that method and to accept, reject or qualify test data in an objective and consistent manner. Validation is a final critical check to be carried out on the technical specifications agreed in the standardisation working groups to decide whether the method satisfies quality objectives and criteria that can be agreed on by the consensus process in standardisation bodies.

The different methods contained in draft texts in the form of prENs developed and agreed in the working groups have to be tested in an inter-laboratory test (round robin test) with a statistically viable number of laboratories (usually around 15). The results obtained from these tests are necessary to guaranty robustness, repeatability and reproducibility of each draft text. The planning of the validation exercise, the preparation of the test material for the inter-laboratory tests (round robin tests), the distribution to the laboratories and the evaluation of the results will be carried out in a close co-operation with the work package leaders and relevant standardisation bodies.

The validation should provide the repeatability and reproducibility of the method covering the entire chain: sampling – sample preparation – analysis.