

Issues and problems related to sewage sludge application in agriculture and forest land

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Prospects:

(1) **increase in volumes of sewage sludge produced by urban waste water treatment plants** (Council Directive 91/271/EEC, *concerning urban waste water treatment*)

(2) **reduction in the amount of biodegradable waste going to landfills** (Council Directive 1999/31/EC, *on the landfill of waste*).

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Necessity to find appropriate disposal or recycling routes for the sewage sludge produced

 European Commission - Agriculture Directorate-General 2

Concerns:

Sewage sludge is a waste, composed of by-products of waste water treatment processes.

It contains:

- **compounds of potential value for soil fertilisation** (organic matter, nitrogen, phosphorus and potassium, and to a lesser extent, calcium, sulphur and magnesium)
- **pollutants** (heavy metals, organic pollutants and pathogens).



Application of sludge to agricultural land

➤ **POTENTIAL BENEFITS:**

- **nitrogen and phosphorus** : partially replace conventional fertiliser application;
- **organic matter:** source of energy for activating the soil microbial population and its mineralisation capacity.

➤ **RISKS:**

- **Soil pollution** (➡ soil micro-organisms, absorption by plants, ingestion by animals)
- **Water and air pollution** (➡ transfer of pollutants or compounds derived from them into the air and water, and their introduction into the food chain).
- **Human exposure to sludge borne pollutants** (➡ consumption of contaminated animal and plant products and water, direct exposure during sludge handling and application to land).
- **Degradation of the agronomic value of agricultural land** (due to excessive accumulation of sludge borne pollutants).



Application of sludge to forest land

➤ SPECIFIC RISKS AND PROBLEMS:

- **accumulation of toxic substances** on forest soils ➤ possible transfer to groundwater, particularly in the areas of drinking water reservoirs;
- **change in balance in the very special and fragile forest habitats** ➤ problem of maintenance of forest biodiversity;
- **hygienic problems** in connection with both the free access into the forests and non-wood benefits (i.e. mushrooms, etc.) provided by forests;
- **lack of appropriate, nature friendly, techniques for sewage application** to forest soils ➤ soil erosion and compaction, damages on forest roads, etc.
- **high costs and difficulty of monitoring of effects**, due to the complexity of forest soil analysis.

➤ POTENTIAL BENEFITS ?

The regular atmospheric deposits and natural processes of fixation and mineralisation in the soils normally cover largely the nutrients needs of forests.



MANAGING RISKS

➤ Necessity to regulate sludge application to agricultural land, by a legislative initiative setting up:

- (1) **Strict technical requirements for admitting application** of sewage sludge to agricultural land, including:
 - (a) **minimum quality standards** for sewage sludge to be used for application to agricultural land.
 - (b) **Stringent operational conditions** (period and methods of application, type of crops, etc.) for such use;
- (2) **Appropriate provisions for monitoring** the application practices **and controlling**, with systematic analyses, both the compliance with quality standards and the effects of sludge application on soils.



MANAGING RISKS

THERE IS ROOM FOR INITIATIVE TO IMPROVE DIR. 86/278/EEC

- (1) **limit values concerning other types of pollutants** (i.e. organic chemicals and pathogens) that may be borne by sludge.
- (2) **certain Member States/regions have set more stringent rules** for application of sewage sludge to agricultural land, compared with the Community directive;
- (3) in practice, **different types of sewage sludge are currently spread on agricultural land** (domestic or industrial sewage sludge, treated or untreated, originated by different types of water waste treatment).
- (4) Apart from agricultural land, **there are other potential recycling outlets for sludge**, including especially urban green areas and reclaimed land.



MANAGING RISKS

Broadening the scope of the existing Directive?

Developing a new legislative framework concerning the use of sludge for purposes of fertilisation?

Taking on board concerns about sustainable management of agricultural land:

- Agricultural soil is a precious and limited resource to be appropriately protected and managed.
- Inexorable degradation of this resource involves not only ruining the farmer's main asset but also reducing the farming opportunities of future generations

Purposes of the new legislative framework:

- preventing harmful effects on soil, vegetation, animals and man.
- **safeguarding the fertility and, more generally, the agronomic value of agricultural land**



MANAGING RISKS

- **INFORMATION**

- Application of sewage sludge on agricultural land should be the result of an **informed decision of the farmer himself**

➤ Legislation should set up the obligation, for sludge suppliers, to provide appropriate **information about the origin and characteristics of the sludge produced**, and in particular about both its fertiliser value and its content of pollutants

- **CODES OF GOOD PRACTICE**

- At local level, there may be **site-specific problems and potentials** to be identified and appropriate **site-specific solutions** to be defined.

➤ Legislation should contribute to **favour the most suitable options to the local specific conditions** ➤ "regional" codes of good practice.



MANAGING RISKS

- **CERTIFICATION & LIABILITY**

- It is **to the sludge supplier to certify that the sludge supplied complies with requirements set up in legislation** and, thus, to guarantee its suitability for use, as neither the farmer nor the landowner has normally the means to guarantee the pollutant content of the sludge applied

➤ Legislation should clarify the **civil liability of sludge suppliers for possible damages**

- **TRACEABILITY & GLOBAL MONITORING**

- sludge spreading on agricultural land should occur in the framework of a contract established among the parties concerned

➤ **Obligatory registration of contracts** ➤ **traceability & monitoring at level of an appropriate geographical scale (for instance, at level of river basin district).**



MANAGING RISKS

• MONITORING AND CONTROLS

- It may be difficult to prove a causal connection between the sludge application and the resulting damage;
- Even if a causal connection could be established, damages might be irreversible and it might be difficult to define a compensation that is high enough to have a deterrent effect on future applications.

➤ **Establishing an appropriate monitoring and control system;**

➤ **Independent organisms ("third bodies") should handle the control of:**

- the characteristics of the sludge to be used for spreading on agricultural land, to verify that it complies with the **minimum quality standards** set up in the legislation
- the quality and vulnerability of soils (to be) spread with sludge.

➤ **The sludge supplier should bear the cost of these controls**



MANAGING RISKS

• COMMON RULES

- For a good operation of the single market, **common rules** are needed concerning:
- **minimum quality standards** for sludge to be used for application to agricultural land.
 - **Operational conditions for sludge application** (period and methods of application, types of crops)
 - **control procedures** on sludge and soils

➤ **Fixing common rules concerning control procedures involves availability of harmonised sampling and analysis methods for both sludge and soils** ➔ methods internationally recognised and able to produce comparable results concerning both nutrients and pollutants that may be borne by sludge.



MANAGING RISKS

- **HARMONISED SAMPLING AND ANALYSIS METHODS:**
 - **provide the technical base needed to link environmental and agronomic concerns,**
 - **are an essential building block of any future legislative initiative concerning sludge landspreading, as they contribute to:**
 - securing a uniform application of the control procedures
 - improving the average quality of these procedures
 - building a commonly agreed language, thus improving transparency concerning results of controls and risk communication
 - **contribute to a progressive improvement of risk assessment procedures concerning sludge landspreading** and, thus, also to improvement in the design of future risk management options



EU Policy on Contaminants in Foodstuffs

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Presentation

- Legal Framework?
- Commission Procedures?
- Current Legislation?
 - maximum limits
 - sampling and analysis
- New Proposals?
- Future Priorities?

Legal Framework

Council Regulation 315/93

‘Community procedures for contaminants in food’

- health protection
- toxicologically acceptable levels (Scientific Committee for Food)
- good working practice
- internal market & national provisions
- safeguard clause
- **Council Directive 85/591 – sampling & analysis**
Community methods of sampling and analysis for monitoring foodstuffs
- **Council Directive 89/397 – official control**
General principles for the performance of control of foodstuffs
- **Council Directive 93/99 – official control**
Quality standards for official control of foodstuffs

Commission Procedures

Setting Legislation on Contaminants

- hazard identified
- toxicity assessment: Scientific Committee for Food (SCF)
 - Acceptable or Tolerable Daily/ Weekly Intakes

- As Low As Reasonably Achievable (ALARA)
- Not present
- data from Member States
 - occurrence - consumption - exposure
- Standing Committee for Foodstuffs
- Commission draft
- consultations – internal and external
- Standing Committee for Foodstuffs

Ccurrent Legislation

EU maximum levels for certain contaminants

(Commission Regulation 194/97)

- nitrates – lettuce, spinach
- aflatoxins – nuts, cereals, milk

Methods of sampling & analysis for official control

(Commission Directive 98/53 - aflatoxins)

- sampling - lot & sub-lot sizes, number of incremental samples, aggregate sample size etc.
- analyses - performance criteria for methods of analyses, e.g. detection, quantification, precision, recovery, specificity

New Proposal

Commission regulation - maximum limits

For adoption:

- heavy metals - cadmium, lead, mercury
- 3-monochloropropane diol (3-MCPD)

Ongoing discussions:

- aflatoxins (in spices)
- ochratoxin A

Heavy metals – toxicity

Cadmium	kidney dysfunction Skeletal damage Reproductive deficiency
Lead	cognitive development, Intellectual performance (children) Blood pressure Cardiovascular disease (adults)

Commission Directive – sampling & analysis

Methods of sampling and analysis for official control of the levels of lead, cadmium and mercury

- lot & sub-lot sizes, incremental samples, aggregate sample size (1kg)
- two samples analysed, accept if mean conforms to maximum limit
- analyses performance criteria e.g. detection, quantification, precision, recovery, specificity.

Future Priorities

Heavy metals

- Arsenic, Tin, ...

Organic contaminants

- Dioxins + PCBs (Polychlorinated Biphenyls)
- PAHs (Polycyclic Aromatic Hydrocarbons)
- Organotins (e.g.tributyltin)
- Brominated flame retardants
- Musks, ...

Conclusions

- Contaminants in foods must be at toxicologically acceptable levels
- Regulation is used to limit levels of contaminants in foodstuffs where necessary to protect human health
- Harmonised methods for sampling & analysis are laid down for official control of certain contaminants in food
- Good working practice by industry should ensure levels of contamination in the environment and in food are as low as possible.

Extracted from Proposed Commission Regulation on Contaminants - Maximum Levels for Heavy Metals

Section 3: Heavy metals

3.1 Lead (Pb)

Product	Maximum level (mg/kg wet weight)	Performance criteria for sampling	Performance criteria for methods of analysis
3.1.1. Cow's milk (raw milk, milk for the manufacture of milk-based products and heat-treated milk as defined by Council Directive 92/46/EEC)	0,02	Directive 2000/..EC ¹	Directive 2000/..EC
3.1.2. Infant formulae and follow-on formulae as defined in Directive 91/321/EEC ²	0,02	Directive 2000/..EC	Directive 2000/..EC
3.1.3. Meat of bovine animals, sheep, pig and poultry as defined in Article 2 (a) of Council Directive 64/433/EEC ³ , as last amended by Council Directive 95/23/EC ⁴ , and Article 2 (1) of Council Directive 71/118/EEC ⁵ , as last amended by Council Directive 97/79/EC ⁶ , excluding offal as defined in Article 2 (e) of Council Directive 64/433/EEC and Article 2 (5) of Council Directive 71/118/EEC	0,1	Directive 2000/..EC	Directive 2000/..EC
3.1.3.1. Edible offal of cattle, sheep, pig and poultry as defined in Article 2 (e) of Council Directive 64/433/EEC and Article 2 (5) of Council Directive 71/118/EEC	0,5	Directive 2000/..EC	Directive 2000/..EC
3.1.4. Muscle meat of fish as defined in the category (a), (b) and (e) of the list of Article 1 of Council Regulation (EC) N° 104/2000 ⁷ , excluding fish species listed in 3.1.4.1.	0,2	Directive 2000/..EC	Directive 2000/..EC
3.1.4.1. Muscle meat of wedge sole (<i>Dicologlossa cuneata</i>), eel (<i>Anguilla anguilla</i>), spotted seabass (<i>Dicentrarchus punctatus</i>), horse mackerel or scad (<i>Trachurus trachurus</i>), grey mullet (<i>Mugil labrosus labrosus</i>), common two-banded seabream (<i>Diplodus vulgaris</i>), grunt (<i>Pomadasyb benneti</i>), european pilchard or sardine (<i>Sardina pilchardus</i>)	0,4	Directive 2000/..EC	Directive 2000/..EC
3.1.5. Crustaceans, excluding brown meat of crab	0,5	Directive 2000/..EC	Directive 2000/..EC

¹ [Proposal for a Draft Commission Directive laying down the sampling methods and the methods of analysis for the official control of the levels for certain contaminants in foodstuffs] See page... of this O.J.

² Maximum level applies to the product as proposed ready for consumption or as reconstituted according to the instructions of the manufacturer.

³ O.J. B 121, 29.7.1964, p. 2012.

⁴ O.J. L 243, 11.10.1995, p. 7.

⁵ O.J. L 55, 8.3.1971, p. 23.

⁶ O.J. L 24, 30.1.1998, p. 31.

⁷ O.J. L 17, 21.1.2000, p. 22.

Product	Maximum level (mg/kg wet weight)	Performance criteria for sampling	Performance criteria for methods of analysis
3.1.6. Bivalve molluscs	1,0	Directive 2000/./EC	Directive 2000/./EC
3.1.7 Cephalopods (without viscera)	1,0	Directive 2000/./EC	Directive 2000/./EC
3.1.8. Cereals (incl. buckwheat), legumes and pulses	0,2	Directive 2000/./EC	Directive 2000/./EC
3.1.9. Vegetables as defined in Article 1 of Council Directive 90/642/EEC ⁸ , as last amended by Directive 2000/48/EC ⁹ , excluding brassica, leafy vegetables, fresh herbs and all fungi. In case of potatoes the maximum level applies to peeled potatoes.	0,1	Directive 2000/./EC	Directive 2000/./EC
3.1.9.1 Brassica, leafy vegetables and all cultivated fungi	0,3	Directive 2000/./EC	Directive 2000/./EC
3.1.10. Fruits as defined in Article 1 of Council Directive 90/642/EEC, excluding berries and small fruits	0,1	Directive 2000/./EC	Directive 2000/./EC
3.1.10.1 Berries and small fruits as defined in Article 1 of Council Directive 90/642/EEC	0,2	Directive 2000/./EC	Directive 2000/./EC
3.1.11. Fats and oils, including milk fat	0,1	Directive 2000/./EC	Directive 2000/./EC
3.1.12. Fruit juices, concentrated fruit juices (for direct consumption) and fruit nectars as defined in Council Directive 93/77/EEC ¹⁰	0,05	Directive 2000/./EC	Directive 2000/./EC
3.1.13. Wines as defined in Council Regulation (EC) N° 1493/1999 ¹¹ (including sparkling wines and excluding liqueur wines), aromatized wines, aromatized wine-based drinks and aromatized wine-product cocktails as defined in Council Regulation (EEC) N° 1601/91 ¹² , and ciders, perry and fruit wines. Maximum level applies to products produced from the fruit harvest 2001 onwards.	0,2	Directive 2000/./EC	Directive 2000/./EC

⁸ O.J. L 350, 14.12.1990, p. 71.

⁹ O.J. L 197, 3.8.2000, p. 26.

¹⁰ O.J. L 244, 30.9.1993, p. 23.

¹¹ O.J. L 179, 14.7.1999, p. 1.

¹² O.J. L 149, 14.6.1991, p. 1.

3.2. Cadmium (Cd)

Product	Maximum level (mg/kg wet weight)	Performance criteria for sampling	Performance criteria for methods of analysis
3.2.1. Meat of bovine animals, sheep, pig and poultry as defined in Article 2 (a) of Council Directive 64/433/EEC and Article 2 (1) of Council Directive 71/118/EEC excluding offal as defined in Article 2 (e) of Council Directive 64/433/EEC and Article 2 (5) of Council Directive 71/118/EEC	0,05	Directive 2000/./EC ¹	Directive 2000/./EC
3.2.2. Meat of horse	0,2	Directive 2000/./EC	Directive 2000/./EC
3.2.3. Liver of cattle, sheep, pig and poultry	0,5	Directive 2000/./EC	Directive 2000/./EC
3.2.4. Kidney of cattle, sheep, pig and poultry	1,0	Directive 2000/./EC	Directive 2000/./EC
3.2.5. Muscle meat of fish as defined in the category (a), (b) and (e) of the list of Article 1 of Council Regulation (EC) N° 104/2000, excluding fish species listed in 3.2.5.1.	0,05	Directive 2000/./EC	Directive 2000/./EC
3.2.5.1. Muscle meat of wedge sole (<i>Dicologlossa cuneata</i>), eel (<i>Anguilla anguilla</i>), european anchovy (<i>Engraulis encrasicolus</i>), louvar or luvar (<i>Luvarus imperialis</i>), horse mackerel or scad (<i>Trachurus trachurus</i>), grey mullet (<i>Mugil labrosus labrosus</i>), common two-banded seabream (<i>Diplodus vulgaris</i>), european pilchard or sardine (<i>Sardina pilchardus</i>)	0,1	Directive 2000/./EC	Directive 2000/./EC
3.2.6. Crustaceans, excluding brown meat of crab	0,5	Directive 2000/./EC	Directive 2000/./EC
3.2.7. Bivalve molluscs	1,0	Directive 2000/./EC	Directive 2000/./EC
3.2.8. Cephalopods (without viscera)	1,0	Directive 2000/./EC	Directive 2000/./EC
3.2.9. Cereals, excluding bran, germ, wheat grain and rice	0,1	Directive 2000/./EC	Directive 2000/./EC
3.2.9.1 Bran, germ, wheat grain and rice	0,2	Directive 2000/./EC	Directive 2000/./EC
3.2.10. Soybeans	0,2	Directive 2000/./EC	Directive 2000/./EC

Product	Maximum level (mg/kg wet weight)	Performance criteria for sampling	Performance criteria for methods of analysis
3.2.11. Vegetables and fruits as defined in Article 1 of Council Directive 90/642/EEC, excluding leafy vegetables, fresh herbs, all fungi, stem vegetables, root vegetables and potatoes	0,05	Directive 2000/./EC	Directive 2000/./EC
3.2.11.1 Leafy vegetables, fresh herbs, celeriac and all cultivated fungi	0,2	Directive 2000/./EC	Directive 2000/./EC
3.2.11.2 Stem vegetables, root vegetables and potatoes, excluding celeriac. In case of potatoes the maximum level applies to peeled potatoes.	0,1	Directive 2000/./EC	Directive 2000/./EC

3.3. Mercury

Product	Maximum level (mg/kg wet weight)	Performance criteria for sampling	Performance criteria for methods of analysis
3.3.1. Fishery products, except those in 3.3.1.1	0,5 mg/ kg	Directive 2000/./EC ¹	Directive 2000/./EC
3.3.1.1 Anglerfish (<i>Lophius spp.</i>), Atlantic catfish (<i>Anarhichas lupus</i>), Bass (<i>Dicentrarchus labrax</i>), Blue ling (<i>Molva dipterygia</i>), Bonito (<i>Sarda spp.</i>), Eel (<i>Anguilla spp.</i>), Halibut (<i>Hippoglossus hippoglossus</i>), Little tuna (<i>Euthynnus spp.</i>), Marlin (<i>Makaira spp.</i>), Pike (<i>Esox lucius</i>), Plain bonito (<i>Orcynopsis unicolor</i>), Portuguese dogfish (<i>Centroscymnes coelolepis</i>), Rays (<i>Raja spp.</i>), Redfish (<i>Sebastes marinus</i> , <i>S. mentella</i> , <i>S. viviparus</i>), Sail fish (<i>Istiophorus platypterus</i>), Scabbard fish (<i>Lepidopus caudatus</i> , <i>Aphanopus carbo</i>), Shark (all species), Snake mackerel (<i>Lepidocybium flavobrunneum</i> , <i>Ruvettus pretiosus</i> , <i>Gempylus serpens</i>), Sturgeon (<i>Acipenser spp.</i>), Swordfish (<i>Xiphias gladius</i>), Tuna (<i>Thunnus spp.</i>)	1,0 mg/ kg	Directive 2000/./EC	Directive 2000/./EC

Extracted from Proposed Commission Directive on Sampling & Analysis – Sampling Plans for Heavy Metals

Table 1: Minimum number of incremental samples to be taken from the lot.

Weight of lot (in kg)	Minimum number of incremental samples to be taken
< 50	3
50 to 500	5
> 500	10

If the lot consists of individual packages, then the number of packages which shall be taken to form the aggregate sample is given in Table 2.

Table 2: Number of packages (incremental samples) which shall be taken to form the aggregate sample if the lot consists of individual packages.

Number of packages or units in the lot	Number of packages or units to be taken
1 to 25	1 package or unit
26 to 100	about 5 %, at least 2 packages or units
> 100	about 5 %, at maximum 10 packages or units

Co-normative research in support to standardisation in the work programme of the Water Key Action

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Science, research and development European Commission

EU Research – FP5, a New Strategic Approach

- Address key European problems**
- Consult widely**
- Facilitate European-level mobilisation of resources/instruments**
- Maximise European added-value**

Stress
06/02/2001

ENERGY, ENVIRONMENT AND SUSTAINABLE DEVELOPMENT

2



The Importance of safe Practice

RTD Priorities

- **1.3.2 Waste water treatment and re-use (15/10/2001)**
 - **Industrial wastewater: towards a close loop operation**
 - **Municipal wastewater reuse**
 - **Sludge reduction and safe disposal**



1.7 Pre-normative, Co-normative Research and Standardisation

Area where there is a need:

- **Support for the water framework directive (15/2/2001)**
- **Support for the implementation of EU directives (15/10/2001)**
 - **Drinking water**
 - **Sludge reuse in agriculture**
 - **Priority chemicals**



Critical aspect of proposals for the Area 1.7.

➤ **Integrated Project:**

- ➔ **Should try to address a complete aspect**
 - ↳ e.g. should not develop a simple test for a single compound
- ➔ **Should be as wide as possible to get the widest support (EU + Associated States)**



Critical aspect of proposals for the Area 1.7.

- **Have the right End-Users**
 - ➔ i.e. International and/or National Normative organisations
- **1.4M_ Threshold?**



Energy, Environment and Sustainable Development

Information on calls for proposal procedures, projects:



- <http://www.cordis.lu/fp5/home.html>
- <http://www.cordis.lu/eesd/home.html>

For expert candidature:

- <http://www.cordis.lu/expert-candidature>



Important information

Minimum Threshold

- => seeking large project => FP VI

Deadline 15/2 and 15/10

- => Deadline for the reception

Pre-Proposal Check

- => Will be more active

Present situation of heavy metal background values in European Soils

Results of Preliminary Evaluations for 4 Member States

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HEAVY METAL IN EUROPEAN SOILS
In collaboration with the European Soil Bureau working group
The document was elaborated with the following partners:

• Denmark	Henrik Breuning-Madsen
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Use of Spatial information about Trace Element Contents in European Soils

Identify the current status of trace elements in European Soils in the context of the revision of the sewage Sludge Directive 86/278/CEE



Objectives of the study

- 1. Evaluate existing databases in Europe of trace elements & organic matter with special regard to:**

Comparability and availability of analytical data, sampling procedure, analytical methods, quality control, data validation

Identify areas or regions with data gaps

Preparation of Europe-wide spatial information (maps) at national level



Concept for a "Short Term Action"

1. Improve the knowledge about suitable databases, use of existing databases
2. Standardization of data to be analyzed
 - Sampling methods
 - Digestion methods
3. Map of soil parent material (Geographical database)
4. Map of main land use
5. Illustration of data sets by individual countries
6. Comparison between different countries



Evaluation related to Soil pH

- England/Wales and France pH based on water
(gives higher pH values)
- Germany and Denmark pH based solutions of 0.01 mol/l CaCl_2
or 1 mol/l KCl (gives lower pH values)
- What is the right pH ?



Summary of the Country-wide available Data in the 4 Member States

Country	All Data	Land use	Soil parent Material	Soil pH Classes	Soil texture classes
Denmark	Yes	Yes	No	Yes	Yes
England/Wales	Yes	No	No	Yes	Yes
France	Yes	No data for grassland	No	Yes	Yes
Germany	Yes	Yes	Yes	Yes/No data for grassland	Yes



Characterization of the data sets

Country	Sampling strategy (small scale)	Georeferenced sampling	Elimination of contaminated samples	Digestion procedure
Denmark	Grid sampling	Yes	No	50 % HNO ₃
France	Random sampling	Not all	No	Aqua Regina HF/HClO ₄
Germany	Random/Grid Sampling	Yes	Yes	Aqua Regina HF/HClO ₄
England/Wales	Grid Sampling	Yes	No	Aqua Regina



Summary of the preliminary Evaluation for 4 Member states

1. From the results obtained it is obvious that at the present stage of data harmonization, a qualified comparison of data in Europe is not yet possible.
2. Country specific evaluations are possible to a certain extent.
3. Europe-wide data collection about heavy metal and organic matter content for all 15 Member States is under way.



Conclusions and further evaluation work

Europe-wide harmonization is requested in:

- to find an agreement on a common measurement procedure for pH
- to find an agreement on common analytical methods for heavy metal analysis and digestion
- to find an agreement to apply Europe-wide sampling strategy
- to find an agreement for the elimination of contaminated soil samples
- to find an agreement on criteria to ensure a minimum level of spatial coverage



Conclusions and further evaluation work

Europe-wide evaluation in:

- **Soil parent material, using Geographical data in Europe**
- **Land use units, based on CORINE land cover**
- **Soil texture classes, based on FAO reference data base**



La strategia Comunitaria per la gestione dei rifiuti

- **Il Centro Comune di ricerca**
- **La situazione dei rifiuti in Italia**
- **Legislazione Europea di gestione dei rifiuti,**
- **Emissioni verso aria, suoli e acque**
- **L'impatto sulla qualità del cibo, protezione dei consumatori**



Results of the working groups on current ISO, CEN and national standards

Results of the Working Group A1

Sampling methods for Soils

Chairman: Dr. Stephen Nortcliff

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Participants from:

Belgium (Wallonia), Germany, Italy, Portugal, Spain, United Kingdom
EC representatives: DG Agriculture; JRC Ispra.

Task: To address Annex VII – ‘Analysis and Sampling’ of the 3rd Draft of the Working Document on Sludge, April 2000

Conclusions and Observations

1. A number of the references to ISO standards are incorrect or not precise enough
2. It was important to recognise that the proposed directive will apply only to conditions relating to the preventative/precautionary aspects.
3. Following from the guidance in the current draft of Annex VII we recommend that *Representative Soil Samples (RSS)* for analysis shall be produced by mixing together 25 core samples for a given area. The number of RSS to be taken will vary with the size of the area (see 5 below) and the homogeneity of the area. No sample patten for the core sampling is prescribed, but it is expected one will be chosen from the designs suggested in ISO-DIS 10381-1.
4. The number of RSS to be collected for each area will be determined by the size of each area (see 5 below). Where the are exhibits marked heterogeneity in terms of soil properties (for example soil texture or soil depth), soil forming factors (for example Soil Parent Material, Soil Drainage, Slope angle or Slope Form) or land use, it may be appropriate to divide the larger area into homogeneous sub-areas. The size of the sub-areas will determine the number of RSS per sub-area.
5. The number of Representative Soil samples for areal units of uniform land use, soil quality, environment and soil management should follow the following guidance (based on ISO-DIS 10381-1).
6. The depth of sampling should take account of both the mode of incorporation (e.g. injection or ploughing) and the depth of incorporation. Land management subsequent to the application of sludge may also influence distribution within the

Table: Representative Soil Sample (RSS) numbers for homogeneous areal units.

Area (ha)	RSS (n)
0 - 2	1
>2 - 5	2
>5 - 10	3
>10 - 15	4
>15 - 20	5
>20 - 30	6

For areas greater than 30 ha, n is determined by the following formula; $n = 1 + A^2$ (where A is the area in hectares).

profile and hence must be taken into account when determining the sampling depth. Examples are given below:

- a. If sludge is incorporated by ploughing to 30 cm, or if subsequent management following application is by ploughing to 30 cm, the sample should comprise a core taken from 0 to 30 cm depth.
- b. If sludge is injected to 25 cm and subsequent land management does not involve incorporation by ploughing, the core should be taken around the point of injection at 20 to 30 cm.

Land use types listed in the Table on Page 4 of the Draft document will determine whether sludge application is by ploughing or injection.

Note: The Working Group had strongly polarised views on whether sludge application was appropriate for forests (excluded in the Draft). Colleagues from Spain and Portugal strongly supported the use of sludge on forested lands.

7. Sampling implements and their use are defined in ISO-DIS 10381-2
8. Sampling personnel should be suitably qualified and validated by the appropriate responsible administrative body.
9. These recommendations provide the minimum standard of sampling
10. No consideration has been given to sampling of manufactured soils.

Results of the Working Group A2 Sampling Methods for Sludge

Chairman: Dr. TC White

Convener of -ISO TC147 SC6 WG11 - The Sampling of Sediments and Sludges

Participants from:

The Working Group (WG) was attended by four delegates from four Member States. The meeting was Chaired by the Convener of the International Standards Organisation (ISO) working group on the Sampling of Sediments and Sludges, ISO TC 147 SC6 WG11.

Those participating were:

- Mr B. Esch from ATV-OVVK Germany
- Mr S.E. Jebesen from the Danish Environmental Protection Agency Denmark
- Ms A. Rodreiguez Cruz Ministerio de Medio Ambienbte Spain
- Ms M.L. Carrreira Da Silva Ministry of the Environment Portugal

In attendance:

- Mr H. Langenkamp JEC (Ispra) Soil and Waste Unit, European Commission.

Objectives

The WG defined its objective as follows.

To arrive at a consensus of opinion as to what it should advise the European Commission are the appropriate requirements for the prescription of guidance for the sampling of sludge with respect to any future revisions of the document:

ENV.E.3/LM - 27 April 2000 concerning an EU imitative to improve the present situation for sludge management based on Treaty Article 175 EC

WORKING DOCUMENT ON SLUDGE 3RD DRAFT.

Resolutions

- 1) The WG advises the Commission that reference in the current, and any future, draft to the required sampling protocols for sludge should be drawn from the guidance document:

ISO EN 5667-13: 1988 Water Quality - Sampling

Part 13. Guidance on sampling of sludges from sewage and water treatment works.

- 2) The WG advises the Commission that the Convener of ISO TC 147 SC6 WG11 has been requested to suggest a revision of ISO EN 5667-13: 1998 to take into account a need for greater clarity of suggested procedural requirements for samples taken for regulatory purposes.
- 3) Notwithstanding the action implicit in the previous two resolutions the WG agreed to make representation, through appropriate ISO member body organisations, for a revision of ISO EN 5667-13: 1998 to the effect that clarification can be made of appropriate protocols for sampling for regulatory purposes. This coordinated action was agreed with the objective of achieving more effective harmonisation of procedures which could be adopted by member states.

Results of the Working Group B1

Analytical methods for pH, dry matter, organic matter and nutrients

Chairman: Dr. Per Jennische

ISO/TC 190, Swedish University of Agricultural Science

Participants from:

Finland, Greece, Ireland, and Sweden. Mr. Marmo and Gawlik from the Commission took part in most of discussions.

General Procedure for Selecting Methods

The group agrees to discuss according to the following scheme:

- What do we want to know about the soil and the sludge?
- Which methods can give us that information?
- Which one of the available methods should we select?
- If none is available & shy; how do we proceed to find a method?

A) pH

This parameter provides information on mobility of pollutants, availability of nutrients, sludge impact on microbial activity and water quality.

A1) Soils

ISO method 10390 is acceptable. Three extractants are described: water, 1 M potassium chloride and 0,05 M calcium chloride. Use of potassium chloride is not to be recommended in the proposed directive. The directive should be designed to accommodate the differences in pH in the extracts of calcium chloride and water, the pH in the calcium chloride extractant usually being 0,5 lower than in water. The working group (WG) has noted that a variation of $\pm 0,2$ for the ISO method has been found in the interlaboratory trial carried out in the production of soil Certified Reference Materials which are now available from JRC.

A) 2 Sludge

EN 12176 is acceptable

B) DRY MATTER

This parameter provides information on the amount of substance -other than water- that is supplied to the soil.

B1) *Soil*

ISO 11465 is acceptable

B2) *Sludge*

EN 12880 is acceptable. The WG has questions about the specification of heavy metals with regard to dry matter as the method for dry matter is based on a determination of water content which is an overestimation. The drying process in which the water content is determined will release other components than water. A more exact method, for example Karl Fischer titration, could be used but the WG would hesitate to recommend a very sophisticated method. Also the error caused by the drying method will result in a slight overestimation of the metal contents in the sludge which is acceptable from a safety point of view.

C) **ORGANIC MATTER**

This determination is useful for the farmer's estimation of the possible benefits of sludge application.

C1) *Soil*

The WG finds that ISO 10694, based on elementary analysis, is acceptable and recommends that the term organic Carbon is always used as the calculation of organic matter requires the introduction of an estimated conversion factor, 1,7 - 2, which will introduce an unnecessary error.

The WG notes that this method may be troublesome for calcareous soils in which the inorganic carbon must either be measured separately or removed before the measurement of organic carbon. An alternative method, ISO 14235, based on sulfochromic oxidation, may be used but it is not recommended because of its environmental load and work hazard. It is suggested that the method based on elementary analysis is recommended in the directive. It should be noted that no limit value is based on this parameter.

C2) *Sludge*

EN 12879 is acceptable

D) **NUTRIENTS**

D1) *Soil*

The WG finds that the main reason for requiring determination of N and P would be to have the ability to estimate the risk of leaching of the nutrients to ground and surface waters. The measurements also give the farmer assistance in judging the benefits of sludge application for the nutrient status of his land. The WG finds that further studies will be required to determine which methods would be suitable to estimate the leaching potential. The present ISO methods 14255 and 11263 have not been developed for this purpose.

Further work will also be necessary to determine which methods are most suitable to give the farmer information on the nutrient status of the land.

The ISO methods are not generally acceptable for all soil types and conditions. The WG suggests that national methods may be used if it is not possible to reach an agreement on common methods.

E) EXTRACTION METHODS

Although this discussion is assigned to another group the WG has some suggestions.

Northern Europe and Ireland - and possibly other countries - use nitric acid (50%) for extraction and have based their national regulations on this.

Germany, France and other countries use aqua regia. As the group understands from available investigations only small differences between the methods are expected for sludge. For soils further investigations are required to see if the differences between the methods are so small or so regular that both methods can be referred to in a directive.

Some comments made by Per Jennische during the discussion

ON pH

Although there is a difference between the two extraction media, water and calcium chloride, the difference is quite small. It is very difficult to define which method is the best for all conditions. If one method would be the best it would not have been necessary for ISO to use three different extraction media. In most cases a difference of about 0,5 would generally have little consequence. It should be mentioned that a long tradition of interpreting pH, for example for liming purposes, have developed within the countries, based on the method used in that country. It should be possible to design the directive in such a way that it can accommodate both extractants.

It could be possible to find another construction of a guide than a table with strict limit values.

ON EXTRACTION MEDIA FOR HEAVY METALS

Determination of heavy metals in sludge in Scandinavia and some other countries have been based on extraction with nitric acid. In other countries extraction with aqua regia has been used. The nitric acid method is simple to use, requires no concentrated acids and leads to no addition of chloride which can cause problems in later steps in the determination (as was also confirmed by Dr Gomez). If the purpose of a choice of methods, is to choose the best method this method is a good candidate.

By the best method one could mean the method that

- gives the desired information
- gives that information simply and efficiently
- is friendly to the staff and to the environment

Note on the measurement of soil pH

Dr. Peter Loveland

Professor of Applied Soil Chemistry, Soil Survey and Land Research Centre,
Cranfield University, Silsoe UK

This note arises from a discussion of analytical methods held during this meeting. It represents a personal view of the issue and is submitted as a contribution to reaching an informed decision on which methodology to adopt. It necessarily simplifies a considerable amount of complex physical chemistry and there could be endless discussion about points of detail. However, a pragmatic solution needs to be reached, which all can generally accept.

- A) Some assumptions have to be made in order to arrive at a consensus:
1. The method has to be *practical* and be *suitable for a wide range of soil types*;
 2. The assumption is that we are dealing *mostly with agricultural soils*;
 3. The pH range of interest is *from pH5 to about pH8.5*;
 4. The soils are largely within a *temperate or Mediterranean climate*;
 5. That the aim is to *protect the soil using the precautionary principle*, if no better evidence is available.
- B) Although this might seem very elementary, it has to be remembered that the application of the concept of pH to soils is an approximation of the original concept. The latter was developed for solutions with relatively simple chemistry. In contrast, *and in general terms*, soil pH is the result of an interaction between the electrical properties of soil particle surfaces, their ion exchange reactions, and the presence of substances dissolved in the soil solution. These factors can differ considerably between soils because of: their inherent properties, the addition of materials such as fertilisers or other agro-chemicals (which can dissolve in the soil solution), changes in soil moisture status (which will affect the concentration of substances in the soil solution), the effects of growing crops (for example, as they might affect the concentration of substances in the soil), the kind of substances in solution, the soil gas chemistry (which is often a reflection of soil biological activity), and so on. **In practical terms, the measurement of pH attempts to estimate the sum of these effects in a single measurement.**
- C) In about 1950, Schofield addressed this problem in *agricultural, temperate soils* and concluded that the best approach was to measure the range over which soil pH might vary during the growing season. He developed a standardised approach to this by recommending that pH be measured in a fixed soil:water ratio, and also in the same ratio in 0.01M calcium chloride solution. The latter was based on the finding that calcium was by far the *commonest cation present in temperate, agricultural soils*, and that the variation in its concentration during the growing cycle did not exceed the concentration represented by 0.01M calcium chloride solution *most of the time and in*

most situations. By adding a relatively large volume of a weak solution of a simple electrolyte to the soil, Schofield's method, in effect, smoothed out all the sources of variation, which might themselves be difficult to measure individually, yet represented the maximum variation which one might reasonably expect. The addition of a solution of a simple salt (an electrolyte - in this case 0.01M calcium chloride solution) to most temperate soils in agricultural use will cause pH to fall, as the calcium will displace protons (H^+) from the soil particle surfaces and/or from anions in the soil solution. The extent to which this happens depends on the composition of the particles, the exchange complex of their surfaces, the soil solution and so on.

- D) In practice, the measurement of pH of thousands of samples of temperate, agricultural soils has shown that *in general pH will fall slightly in 0.01M calcium chloride solution* compared with that measured in water. The fall is less at lower pH, because release of a further quantity of protons into an already acid medium does not lower the pH much further, whilst at higher pH (greater than about 7) the fall in pH is also relatively small because the system is buffered by the presence of increasing amounts of carbonate. The equilibrium pH of the calcium carbonate - water - carbon dioxide system (at partial pressures of carbon dioxide about equal to those found under growing crops) is about pH8.2. Thus, the effect of release of protons is small at or close to this pH. The greatest lowering of pH occurs in the middle range - from about pH5.5 to about pH6.5/pH7, but the lowering of pH *rarely exceeds 0.5 of a unit in most temperate, agricultural soils*.
- E) In the application of pH to the protection of soil, one needs to consider that many of the trace elements of interest become more mobile at a lower pH. It is arguable whether a lowering of soil pH by 0.5 pH units would be significant in most situations, but the precautionary principle would suggest that the lower value, i.e. that measured in 0.01M calcium chloride solution, offers the greatest protection against potential mobility, i.e. if you assume that the soil is more acid, you add less pollutant to it. **Before accepting this view, however, the European Commission might wish to review the literature on element mobility to gain an expert view on the magnitude of this issue.**
- F) The ISO standard (ISO 10390) also allows the use of 1M potassium chloride solution as a background electrolyte for the measurement of soil pH. This is a much more concentrated electrolyte and lowers soil pH considerably more than occurs in 0.01M calcium chloride solution, although the mechanisms are broadly the same. However, the ISO standard was developed for International, i.e. global, use. Generally speaking, highly weathered soils have exchange complexes which are less saturated with cations such as calcium, and more so with hydrogen, iron and aluminium - all 'acid' cations. A number of soils in warmer climates also contain far larger quantities of soluble salts than most temperate agricultural soils. In these circumstances, the potential for greater annual or inter-annual variation in soil pH is larger than in temperate agricultural soils. The potassium chloride approach to soil pH measurement was developed for this situation. It is not generally thought to be applicable to temperate, agricultural soils in the range pH5 to pH8. It is, however, entirely appropriate to include the method in a standard (ISO 10390) intended to meet the requirements of soil pH measurement under all conditions.

Results of the working Group B2 Analytical methods for the measurement of pathogens and micro-organisms

Chairman: Dr. Marie-Renée de Roubin

Expert CEN/TC 308, Anjou Recherche, Vivendi Water

Participants from:

Marie-Renée de Roubin (convenor CEN/TC308/WG1/TG5),

Linda Bagge (DK)

Reinhard Böhm (D)

Luc Debaene (B)

Isabelle Deportes (F)

Tim Evans (UK)

Marie Rosado (P)

- The group agreed that it is impossible to select between the various microbiological methods of test without doing interlaboratory assessment work. That this work is also essential to define the precision of methods and that it is a suitable candidate for co-normative research funding.
- The group discussed the practicality of distributing samples across national boundaries and considered this is achievable but that a courier service would be preferred to ensure rapid delivery in controlled conditions.
- The group considered that the following existing methods for Salmonella spp. are worth developing, all of them have validation data:
 - AFNOR proposed method CEN/TC 308 WG1 TG5 working document;
 - UKWIR membrane resuscitation method;
 - Method proposed by Professor Böhm;
 - Italy has had a standard for Salmonella in sludge since 1992 and therefore may have a standardised method with validation data?
- The most suitable method should be selected by interlaboratory comparison using untreated and treated sludge samples. It was considered that it is important to check that methods do not produce false positive results.
- The group discussed whether the standardised methods should include a requirement for laboratories to consider whether an appropriate pre-treatment step should be applied when necessary.
- The group considered that the following existing methods for E. coli are worth developing, all of them have validation data:
 - MLGA filtration method for E. coli used in the UK national survey;
 - UKWIR membrane resuscitation method;
 - ISO 9308-2 multiple tube MNP method;
 - ISO 9308-3 MPN microplate method.

It was considered that the proposed limit of 500cfu E. coli /g fresh weight is consistent with analytical capability.

- The group felt that *Enterococci* and viable *Ascaris* eggs are worthy of consideration for validating processes and perhaps for end product standards. Members agreed that *Clostridium perfringens* is not suitable as an indicator organism. There was division about the necessity of an *E. coli* method's ability to detect VTEC strains.
- The group questioned and debated the protocols for process validation and considered that if they are to be used there should be well defined, standardised procedures that are applicable to every type of process.
- There was division amongst the group about the practicability of validating every full-scale advanced sludge treatment plant by using an introduced organism. Where it has been practised the organism is introduced in a recoverable captive form, in a metal canister for liquid processes and impregnated in cloth for other systems.
- The group was uncertain about whether the basis for expressing *E. coli* is dry weight or wet weight.

Results of the Working Group C 1

Sample preparation, extraction and analytical methods for heavy metals and nutrients

Chairman: Dr. Alain Gomez
CEN/TC 308. INRA - USRAVE

Participants from:

- 12 participants
- 9 countries: United Kingdom, Netherlands, Italy, Sweden, Denmark, Portugal, Belgium, Austria, France + European Commission.
- Experts from:
 1. ISO/TC 190 Soil Quality
 2. CEN/TC 223 Soil improver and growing media
 3. CEN/TC 292 Characterisation of wastes
 4. CEN/TC 308 Characterisation of sludges.

Work has been conducted on the basis of the following documents:

Harmonisation of methods in soil and sludge WG/C1

Sample preparation, extraction and analytical methods for heavy metals and nutrients

What elements?

- Nutrients: N, P, K, Ca, Mg.
- Micro-nutrients: S, Fe, Mn, B, Zn, Cu
Trace elements: Cd, Cr, Co, Pb, Cu, Mn, Ni, Zn, Hg, As, Se, Sb, Tl...

	ISO TC 190		CEN TC 308	
	Ref.	Title	Ref.	Title
Sample preparation	ISO 11464	Pretreatment of samples for physico-chemical analysis	EN 13346	Characterization of sludges: Aqua regia extraction methods- Determination of trace elements and phosphorus
Analytical methods	ISO 11466	Extraction of trace elements by aqua regia	EN 13342	
	ISO 11047	Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc: Flame and electrothermal atomic absorption spectrometric methods		
	ISO CD 16772	Determination of mercury in aqua regia soil extracts.		
	ISO N 373	Determination of arsenic, antimony and selenium in aqua regia soil extracts by atomic absorption spectrometry.		
	ISO DIS 14869-1	Determination of total trace element content: Part 1: Digestion with hydrofluoric and perchloric acids.		
	ISO DIS 14869-2	ISO DIS 14869-2 Determination of total trace element content: Part 2: Dissolution by alkaline fusion.		
	ISO 11260	Determination of effective cation exchange capacity and base saturation level using barium chloride solution		
	ISO 13536	Determination of the potential cation exchange capacity and exchangeable cations using barium chloride solution buffered at pH = 8.1		
	ISO 11261	Determination of total nitrogen: Modified Kjeldahl method.		
ISO 13878	Determination of total nitrogen content by dry combustion ("elemental analysis").			
ISO 14255	Determination of nitrate nitrogen, ammonium nitrogen and total soluble nitrogen in air-dry soils using calcium chloride solution as extractant.			

	ISO TC 190		CEN TC 308	
	Ref.	Title	Ref.	Title
	ISO DIS 14256-1	Determination of nitrate, nitrite and ammonium in field moist soils by extraction with potassium chloride solution: Part 1: Manual method	308.012	Characterisation of sludges - Determination of ammoniac nitrogen
			308.013	
	ISO CD 14256-2	Determination of nitrate, nitrite and ammonium in field moist soils by extraction with potassium chloride solution: Part 2: Automated method.		Characterisation of sludges - Determination of nitrate nitrogen
	ISO 11263	Determination of phosphorus: Spectrometric determination of phosphorus soluble in sodium hydrogen carbonate solution.		
	ISO DIS 15178	Determination of total sulfur by dry combustion.	308.034	Characterisation of sludges - Determination of total phosphorus
	ISO 11048	Determination of water-soluble and acid-soluble sulfate.		
	ISO DIS 14870 ISO N 000	Extraction of trace elements by buffered DTPA solution. Leaching.		

Sample Preparation

	ISO TC 190		CEN TC 308	
	Ref.	Title	Ref.	Title
Sample preparation	ISO 11464	Pretreatment of samples for physico-chemical analysis	EN 13346	Characterization of sludges: Aqua regia extraction methods - Determination of trace elements and phosphorus, see annex A (informative)

Sample Digestion/Extraction/Determination

ISO 11466	Extraction of trace elements by aqua regia	EN 13346	Characterization of sludges :Aqua regia extraction methods - Determination of trace elements and phosphorus
ISO 11047	Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc: Flame and electrothermal atomic absorption spectrometric methods		
ISO CD 16772:	Determination of mercury in aqua regia soil extracts.		
ISO N 373	Determination of arsenic, antimony and selenium in aqua regia soil extracts by atomic absorption spectrometry.		

Comparison

Aqua regia extraction		
ISO 11466	EN 13466	EN 13657
cadmium, chromium, cobalt, copper, lead, manganese, nickel, zinc, arsenic, selenium, antimony and mercury 3g sample in 21 ml HCl +7 ml HNO ₃	cadmium, chromium, copper, lead, nickel, zinc, arsenic, selenium, mercury and phosphorus 3g sample in 21 ml HCl +7 ml HNO ₃ method A 0.5 g in 6 ml HCl + 2 ml HNO ₃ method B 0.3 to 1 g in 6 ml HCl + 2 ml HNO ₃ method C –closed system microwave 0.5 to 1g in 18 ml HCl + 6 ml HNO ₃ method C –open system microwave	31 elements including those of ISO 11466 and CEN 13466
Washing solution: HNO ₃ 0.5 mol Final volume: 100 ml Note on C content	Washing solution: H ₂ O Final volume: 25 to 100 ml	

Determination on aqua regia extract	
ISO 11466 – ISO CD 16772 – ISO N 373	EN 13466
cadmium, chromium, cobalt, copper, lead, manganese, nickel, zinc, arsenic, selenium, antimony and mercury FAAS - ETAAS - ZETAAS - HGAAS - CVAAS - AFS Recommended procedure Normative references: soil quality Preparation of the sample, dry matter, ...	cadmium, chromium, copper, lead, nickel, zinc, arsenic, selenium, mercury and phosphorus FAAS - ETAAS - ZETAAS - HGAAS - ICP/OES - ICP/MS General recommendations Normative references: Water quality Determination of Cr, Hg, Cd, As, ...

Sample Digestion/Extraction/Determination

ISO DIS 15178	Determination of total sulfur by dry combustion.		
ISO DIS 14869-1	Determination of total trace element content: Part 1: Digestion with hydrofluoric and perchloric acids.	308.034	Characterisation of sludges - Determination of total phosphorus
ISO DIS 14869-2:	ISO DIS 14869-2 Determination of total trace element content: Part 2: Dissolution by alkaline fusion.		
ISO 11261	Determination of total nitrogen: Modified Kjeldahl method.	EN 13342	Characterisation of sludges - Determination of Kjeldhal nitrogen
ISO 13878	Determination of total nitrogen content by dry combustion ("elemental analysis").		

Comparison

Soil	Sludge
<p>ISO 11261 Determination of total nitrogen: Modified Kjeldahl method. Reagent: 4 ml of sulphuric acid + salicylic acid + sodium thiosulphate Catalytic reagent: 1.1 g of cupric sulphate + potassium sulphate + titanium dioxide 0.5 to 1 g of sample Thermal program under visual control Addition of 20 ml H₂O Addition of 20 ml NaOH 10 mol/l Boric acid 20 g/l Steam distillation: 40 ml Titrimetric method</p> <p>ISO 13878 Determination of total nitrogen content by dry combustion ("elemental analysis").</p>	<p>EN 13342 Characterisation of sludges - Determination of Kjeldhal nitrogen Reagent: 10 ml of sulphuric acid Catalytic reagent: 5 g of sodium sulphate + selenium 0.25 to 0.5 g of sample Thermal program under visual control Addition of 40 ml H₂O Addition of 30 ml NaOH 12.5 mol/l Boric acid 40 g/l Steam distillation: 120 ml + 30 ml Titrimetric method ???</p>

Sample Digestion/Extraction/Determination

ISO 14255	Determination of nitrate nitrogen, ammonium nitrogen and total soluble nitrogen in air-dry soils using calcium chloride solution as extractant.		
ISO DIS 14256-1	Determination of nitrate, nitrite and ammonium in field moist soils by extraction with potassium chloride solution: Part 1: Manual method	308.012	Characterisation of sludges - Determination of ammoniac nitrogen
		308.013	Characterisation of sludges - Determination of nitrate nitrogen
ISO CD 14256-2	Determination of nitrate, nitrite and ammonium in field moist soils by extraction with potassium chloride solution: Part 2: Automated method.		
ISO 11263	Determination of phosphorus: Spectrometric determination of phosphorus soluble in sodium hydrogen carbonate solution.		
ISO 11048	Determination of water-soluble and acid-soluble sulfate.		
ISO DIS 14870	Extraction of trace elements by buffered DTPA solution.		
ISO N 000	Leaching.		

Conclusion and Observation

1. Lack of standard on “pre-treatment of sludge sample for physico-chemical analysis”
Proposal: working group (ISO/TC 190, CEN/TC 223, 292 and 308) to elaborate one standard on the topic including soils, wastes and sludges on the basis of ISO 11464 and works of CEN /TC 223 and 292.
2. Aqua regia extraction :
 - 2.1 no microwave method for soils.
Proposal: interlaboratory assays to validate the use of microwave method on the basis of CRM.
 - 2.2 Scandinavian countries work with an alternative method (HNO_3)
Proposal: alternative method is acceptable if it gives the same results with CRM of soils and sludges (data given on aqua regia basis).
 - 2.3 There are two different washing procedures for the filter
Proposal: One laboratory tests the two procedures working with CRM of soils and sludge, in order to give data to make a choice between the two of them.
 - 2.4 Lack of note on C content for sludges as it is given for soil standard.
 - 2.5 Determination techniques:
 - Add ICP-OES and ICP-MS in soil standard
 - Add CVAAS and AFS in sludge standard
3. Total sulphur determination by dry combustion is suitable for both soil and sludge.
4. There are similar works on trace elements total content: part 1 (HF) and part 2 (Alkaline fusion) in ISO/TC 190 and CEN/TC 292.
Proposal: working group to elaborate one standard for soil, sludge and wastes.
5. Total nitrogen determination by dry combustion (ISO 13878)
Proposal: One laboratory tests this method with sludge. If it is suitable, one standard for soil and sludge.
6. Strong need of a working group (ISO/TC 190, CEN/TC 223, 292 and 308) to elaborate one Kjeldahl method for soil, wastes and sludge.
7. $\text{NH}_4 - \text{N}$ extractable: need of an agreement on the concentration of the extracting solution (ISO/TC 190, CEN/TC 223 and 308).
8. Total phosphorus determination: dry combustion (ISO/TC 190) or wet digestion (CEN/TC 308)? Proposal: working group to elaborate one standard for soil, sludge and wastes (ISO/TC 190, CEN/TC 292 and 308).
9. Extractable phosphorus and sulphur, trace elements extraction with DTPA: sludges are not concerned.
10. Leaching test: there is an important need for sludge and soil. There are works of CEN/TC 292. Proposal: working group to elaborate one standard for soil, sludge and wastes (ISO/TC 190, CEN /TC 292 and 308).

Results of the Working Group C2 Sample Preparation, Extraction and Analytical Methods for Organic Compounds

Chairman: Prof. Dr. R. Leschber
CEN/TC 308 and DIN

Participants from:

10 experts present from 6 European countries and from the EU-Commission

Conclusions and observations

1. The Group discussed the ISO Standard 5667, Part 15: “Water Quality Sampling: Guidance on Preservation and Handling of Sludge and Sediment Samples”. It was resolved that this International Standard fulfils present analytical needs. Once the revised Directive 86/278(EEC will cover organic compounds and hygienic requirements, then revision of ISO 5667, Part 15 may be required which has not yet been started due to other reasons.
The standard contains in a table of data for storage conditions (temperature range, times) which refer to different parameters and organic substances as well.
2. With regard to that, the Group raised the issue of resolution of disputes: how should reference storage be made? Do we need stability tests of reference storage methods? What about the stability of substances in reference materials which have to be stored over longer times?
3. The Group discussed then ISO FDIS 14507 “Pretreatment of Samples for Organic Analysis in Soils” and recommended this as a useful document for the analytical procedures for organic determinants in soil.
4. The Group did not see reasons for developing further methods for soils specifically as existing at present; the development or revision of ISO and CEN methods for sludges should also consider inclusion of soils and solid wastes.
5. The Group also considered the possibility of analysing for further organic contaminants, which are under discussion in national scientific and official expert groups, like endocrine disrupters, brominated flame retardants etc.
It was resolved, that it is up to national or local authorities to extend research activities in cases of point or non-point source pollution.
6. The Working Group considered the list of organic contaminants in Annex IV of the 3rd Draft of the EU working document.

AOX, PCB and dioxins are part of German standards and DIN methods exist for these substances.

In Denmark, national standard methods are available for LAS, DEHP, NPE and PAH9. Germany also has methods for PAH₆ and PAH1₆ (EPA list of PAH).

France has a standard method for PAH₃ and PCB.

In Holland, there are validated methods for AOX, PAH and PCB.

Sweden and Austria: no standards.

At present CEN/TC 308 is developing standard methods for AOX and PCB. It is suggested that these methods are the first step to European Standards in this field and that this programme could be extended and improved if it seems necessary from the view of the Commission.

But there is no doubt that these improvements are closely connected with financial and man power support for especially practical method testing and interlaboratory comparisons.

Finally an important aspect was discussed:

7. The Group recognised that the table of organic contaminants and the suggested limit values are related to the given national method of analysis used. It is important that the Commission is aware that the development of new analytical methods may significantly improve recovery of a contaminant or groups of contaminants, which might in future, result in limit values being revised.

Summary and Results of the Workshop

The Overall Conclusions of the Workshop

1. ISO-Soil Standards for sampling need to be re-drafted.
2. Basic requirements for sampling should be given in the annex to the sludge directive.
3. The sampling guidance for sludge (ISO 5667-13) requires redrafting.
4. The standards for pH, organic matter and dry matter are acceptable.
5. The Workshop agreed that the determination of pH in soil should be carried out in 0.01M calcium chloride solution.
6. Neither the ISO nor the CEN Standards for the determination of nutrients fulfil the requirements of the draft sewage sludge directive.
7. Additional research and basic standardisation work is necessary for the determination of pathogens and micro-organisms.
8. ISO Standard 5667, part 15: 'Water Quality Sampling: Guidance on Preservation and Handling of Sludge and Sediment Samples' fulfils present analytical needs.
9. ISO FDIS 14507: 'Pre-treatment of Samples for Organic Analysis in Soils' fulfils present analytical needs for the determination of organic pollutants in soils.
10. For additional requirements for pre-treatment (e.g. pathogens, physico-chemical analysis), harmonisation between Technical Committees: ISO/TC 190, CEN/TC 223, 292 and 308 is considered necessary.
11. The present ISO-method for the extraction of heavy metals from soil material does not take into account present laboratory practice (micro-wave digestion).
12. The Workshop mainly agreed with 'aqua regia' for digestion.
13. Analytical determination techniques have to be equivalent (AAS/ICP).
14. For the determination of nutrients (e.g. secondary nutrients, nitrogen) a horizontal approach is required.
15. There is a need for pathway-specific methods (leaching/weak extractant) for different materials.
16. The national methods of analysis should be used for organic contaminants indicated in the table of the draft directive, in relation to the proposed limit values.

The workshop demonstrated that a horizontal approach in standardisation work is urgently required.

Annexes

Workshop on Harmonization of sampling and analysis methods for heavy metals, organic pollutants and pathogens in soil and sludge

Stresa - Lago Maggiore, Italy
8-9 February 2001

PROGRAMME

Thursday 8 February 2001

Session I: Introduction

Chair: Mr. Langenkamp/Marmo

- 9.00-9.15 General introduction
Mr. G.M. Martin (JRC Ispra)
- 9.15-9.45 Harmonisation in view of the revision of the Sewage Sludge Directive 86/278/EEC
Mr. L. Marmo (DG ENV)
- 9.45-10.15 Issues and problems related to sewage sludge application in agriculture and forest land
Mr. A. De Angelis (DG AGRI)
- 10.15-10.45 *Coffee break*
- 10.45-11.15 EU policy on contaminants in foodstuff
Mr. M. Slayne (DG SANCO)
- 11.15-11.45 Co-normative research in support to standardisation in the work programme of the Key Action on Water
Mr. J.F. Junger (DG RTD), Mr. M. Gadeberg (DG RTD)
- 11.45-12.15 Present situation of heavy metal background values in European soils
Mr. H. Langenkamp (JRC), Mr. J. Utermann (BGR)
- 12.15-14.00 *Lunch*

Session II: Working groups on current ISO, CEN and national standards

Working Groups:

- 14.00-18.00 A1) Sampling methods for soils
Mr. S. Nortcliff (UK) (Expert ISO/TC 190)
- 14.00-18.00 A2) Sampling methods for sludge
Mr. T. White (UK) (Expert in ISO/TC 147)
- 15.30 16.00 *Coffee break*

- 14.00-18.00 B1) Analytical methods for pH, dry matter, organic matter and nutrients
Mr. P. Jennische (S) (Expert ISO/TC 190)
- 14.00-18.00 B2) Analytical methods for the measurement of pathogens and
micro-organisms
Mrs. M.R. de Roubin (F) (Expert CEN/TC 308)
- 14.00-18.00 C1) Sample preparation, extraction and analytical methods for heavy
metals and nutrients
Mr. A. Gomez (F) (Expert CEN/TC 308)
- 14.00-18.00 C2) Sample preparation, extraction and analytical methods for organic
compounds
Mr. R. Leschber (D) (Expert CEN/TC 308)
- 20.00 Workshop dinner

Friday 9 February 2001

Session III: Presentation of the results of the working groups

Chair: Mr. G. Bidoglio

- 9.00-9.20 A1) Sampling methods for soils
Mr. S. Nortcliff (UK)
- 9.20-9.40 A2) Sampling methods for sludge
Mr. T. White (UK)
- 9.40-10.00 B1) Analytical methods for pH, dry matter, organic matter and
nutrients
Mr. P. Jennische (S)
- 10.00-10.30 *Coffee break*
- 10.30-10.50 B2) Analytical methods for the measurement of pathogens and
micro-organisms
Mrs. M.R. de Roubin (F)
- 10.50-11.10 C1) Sample preparation, extraction and analytical methods for heavy
metals and nutrients
Mr. A. Gomez (F)
- 11.10-11.30 C2) Sample preparation, extraction and analytical methods for organic
compounds
Mr. R. Leschber (D)
- 12.00-14.00 *Lunch*

Session IV: Conclusions

Chair: Mr. H. Langenkamp

- 14.00-16.00 Final discussion and elaboration of the recommendations of the
workshop with presentation of the proposed standards
- 16.00 *End of the workshop*

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European Commission

**EUR 19809 – Workshop on Harmonization of Sampling and Analysis Methods
for Heavy Metals, Organic Pollutants and Pathogens in Soil and
Sludge**

H. Langenkamp, L. Marmo

2001 – 76 pp. – 16.2 x 22.9 cm

Environment and quality of life series

In February 2001, DG Environment and the JRC organised a technical workshop on “Harmonisation of analytical methods of sampling and analysis methods for heavy metals, organic pollutants and pathogens in soil and sludge”. The workshop was attended by 48 experts from 14 member states from the EU. The reason for this workshop was the revision of the Sewage Sludge Directive 86/278/EEC. In the working document for the revision of the directive it is proposed that the investigations should follow internationally accepted standards, which should be by CEN or ISO or national standards.

This booklet contains the programme of the workshop, the presentations made over the two days and the results of the discussions in the different working groups.

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