Desk study report on bulk density

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0. SUMMARY

The present report provides an evaluation of the possibility of harmonizing standards for bulk density in sludge, soil and biowaste.

The comparison of standards for the determination of bulk density demonstrates that none of the existing standards is suitable as a direct basis for a harmonized and horizontal standard covering all concerned matrices; sludge, soil, and biowaste. However, it is considered feasible to produce a horizontal standard for determination of ex-situ bulk density covering sludge, soil and biowaste by modifying the standard EN 12580 - Soil improvers and growing media – Determination of a quantity. Another standard on determination of bulk density in soil improvers and growing media was considered as basis for a horizontal standard: Annex A in EN 13040:1999 – Determination of laboratory compacted bulk density. However this standards covers compacted bulk density and may not be suitable for sticky material, such as sludge - without modifications.

For soil replaced from one site to the other (e.g. traded soil or contaminated soil) the laboratory bulk density (ex-situ bulk density method) is relevant. However, it is emphasised that the accuracy and representativeness of the results may be compromised compared to the use of in-situ core methods for soils, which are particularly suitable for the examination of in-situ soil properties (in respect to plant growth, storage capacity etc.) A horizontal method for laboratory bulk density will not replace the in-situ methods for bulk density in soil.

1. INTRODUCTION

The objective of the project "Horizontal" is to develop horizontal and harmonised European standards in the fields of sludge, soil and biowaste to facilitate regulation of these major streams in the multiple decisions related to different uses and disposal governed by EU Directives.

The revision of the Sewage Sludge Directive 86/278/EEC, the upcoming Directive on the biological treatment of biodegradable waste and the Soil Monitoring Directive call for standards on sampling, on hygienic and biological parameters, on methods for inorganic and organic contaminants and for mechanical properties of these materials. This project considers development and implementation of horizontal standards to be used for sludge, soil and bio-waste.

The work for developing horizontal and harmonised European standards is split up in coherent Work Packages (WPs), each of which addresses a main aspect of all relevant standards required or likely to be required in EU regulations regarding sludge, biowaste and soil. The report in hand deals with a desk study under WP 6: Inorganic Parameters: Assessment of the feasibility of draft horizontal standards for:

- bulk density in sludge, soil, biowaste and neighbouring fields

Existing standards and/or draft standards are assessed and key points where possible differences exist are identified and the differences evaluated in order to prepare a draft horizontal and harmonized standard.

Ongoing activities in the active CEN/TC workgroups are included in the reports to the extent it has been possible:

CEN/TC 308, Sludge - The secretary of TC 308 and a member of working group, Ms.Bente Nielsen, Krüger Denmark, has been consulted.

CEN/TC 223, Soil improvers and growing media - Members of TC 223, Mr.Morten F.Carlsbæk has been consulted. In addition valuable information was provided by members TC 223/WG 3 Howard Burnett and Tim Evans upon the first consultation period.

The present report is a revised version revised of the desk study report given for consultation early 2005. The revisions are based on the comments provided during the first consultation period. As a consequence of the received comments a revised draft standard has been prepared as well.

2. EXISTING STANDARDS OR DRAFT STANDARDS

The weight of matrices such as soil, sludge and biowaste is referred to as e.g. soil bulk density. Density is the mass of material contained within a given volume. The weight of water is the reference for density measurements: 1 gram of water = 1 cubic centimetre, and 1 cc water = 1 mL.

Bulk density (BD) is the density of a medium as it occurs, e.g. bulk density of a soil is the density of soil as it occurs in the ground. The bulk density takes into account the total volume of the solid media (the space occupied by the solid particles plus the space occupied by the air of the pores or pore space. The weight of the media can be given on a dry weight basis or on wet weight basis. The volume of the particles, excluding the air space, determines the particle density. The present project concerns the possibility to establish a horizontal standard on bulk density. Particle density is not considered.

The available standards for determination of bulk density relevant for biowaste, soil and related matrices are listed in Table 1 together with an outline of the sample material and principles. No existing standards or draft standards have been identified for sludge.

Table 1 Bulk density

Number	Title	Sample material/scope	Principle
EN 13040: 2000, Annex A	**Soil improvers and growing media** – Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density.	Soil improvers and growing media.	Filling of a one-litre test cylinder with sample "as received", static compaction and weighing of the contents of the cylinder.
EN 13041 2000	**Soil improvers and growing media** – Determination of physical properties – Dry bulk density, air volume, water volume, shrinkage value and total space.	Soil improvers and growing media. Not applicable to liming materials or sewage sludge.	Saturation with water, equilibration at minus 50 cm water pressure. Repeated wetting and equilibration at minus 10 cm water pressure. Weighing of the content of the cylinder. Drying at 103 ºC and reweighing and measure of volume.
EN 12580: 1999	**Soil improvers and growing media** – Determination of quantity (Includes determination of bulk density, volume and mass)	Soil improvers and growing media.	Filling of a 20 little measuring cylinder and weighing of the contents of the cylinder.
Draft prEN 15238	**Soil improvers and growing media** – Determination of quantity for materials with particle size greater than 60 mm (Includes determination of bulk density and volume)	Soil improvers and growing media.	For each batch of material the quantity of material is determined and declared either volume or by weight
ISO 11272: 1998	**Soil quality –** Determination of dry bulk density.	Soil.	Drying and weighing of a soil sample. Volume of soil sample is either known by 1. the core method 2. excavation method 3. clod method
[1)]ASTM D2937-04	Standard test method for density of **soil** in place by the drive-cylinder method	Soil. Not applicable to soil that can compress upon sampling, very hard soil, soil of low plasticity or soil with high content of gravel coarser than 4.75 mm.	In-place density is determined by the drive-cylinder method, which collects an undisturbed soil sample. The density is expressed by mass per unit volume. Modifications of this method can be used to determine bulk density and dry bulk density
EN 1236: 1995	**Fertilizers** – Determination of bulk density (loose).	Solid fertilizers except powder. Free-flowing fertilizers included. Materials containing more than 20 % by mass of particles exceeding 5 mm (diameter) excluded.	Pouring of the fertilizer from a specified funnel into a specified measuring cylinder of known volume and weighing of the contents of the cylinder.
EN 1237: 1995	**Fertilizers** – Determination of bulk density (tapped).	Solid fertilizers except powder. Free-flowing fertilizers included. Materials containing more than 20 % by mass of particles exceeding 5 mm (diameter) excluded.	Pouring of excess fertilizer from a specified funnel into a specified measuring cylinder of known volume. Tapping of the cylinder, removal of excess and weighing of the contents of the cylinder.
EN ISO 7837:2000	**Fertilizers** – Determination of bulk density (loose) of fine-grained fertilizers.	Solid fine-grained fertilizers. Applicable to fertilizers, which contain a large proportion of particles of diameter less than 0,5 mm.	Pouring of the fertilizer from a specified filling device into a specified measuring cylinder of known volume and weighing of the contents of the cylinder.

[1)] ASTM – American Society for Testing and Materials

3. EVALUATION OF THE POTENTIAL FOR HARMONIZED AND HORIZONTAL STANDARDIZATION

A comparison of selected existing international standards for the determination of bulk density is given in Table 2. Of the standards listed in Table 1 the following standards are used as the basis for consideration of the possibility for establishment of a horizontal and harmonized standard:

1) EN 12580:1999. Soil improvers and growing media
2) EN 13040:1999, Annex A. Soil improvers and growing media
3) ISO 11272:1998. Soil

EN 13041 is not applicable for sewage sludge and (liming materials) and therefore not considered feasible for harmonization in this respect. The method determines bulk density from a water-saturated sample. The principle of the method is given in Table 2. PrEN 15238 is describing the determination of quantity for materials with particle size greater than 60 mm. Although the applicability and use as a horizontal standard on bulk density is not fully enlightened, the use of prEN 15238 as the basis is not considered adequate.

Table 1 contains three methods described for determination of bulk density for fertilizers. Similarities to the standards on soil and soil improvers can be identified, however the standards are developed specifically for the more homogeneous product. Potentially, methods developed for fertilizers could practically be used for certain types of dried sludge. However, this issue is not covered here, as the methods would not be applicable in general for soil, sludge and biowaste.

3.1 Principles used in the existing standards

The various steps in determination of bulk density in given standards will to a high degree determine the possibility to produce a horizontal standard for bulk density determination covering soil, sludge and biowaste.

The standard EN 12580:1999 (soil improvers and growing media) describes the principle of filling of a 20-litre measuring cylinder with sample and weighing of the contents of the cylinder. Compacted material or dried material shall be loosened or moistened according to the use of the material as specified by the manufacturer before measurements are performed. The test cylinder, as used for determination of bulk density of soil improvers and growing media, contains a collar (to be removed before weighing) and above this a fall controller, which is a sieve with max (60 ± 2) mm mesh size. The use of the fall controller serves to regulate the velocity of particles entering the cylinder and stops larger particles.

The standard EN 13040, Annex A (soil improvers and growing media), which is a normative part of the standard, describes the principle of filling a one-litre test cylinder with sample "as received", static compaction and weighing of the contents of the cylinder. It is described in a note in the standard that the method is not applicable to liming materials or sewage sludge and that it is not suitable for materials like rockwool and foam slabs. Communication with members of CEN/TC 223 reveals that excluding sewage sludge and liming material from the standard was a mistake. The standard is due for a revision in this respect. The test cylinder, as used for determination of bulk density of soil improvers and growing media, contains a sieve with 20 mm square apertures, which is used to separate coarse material prior to the determination of bulk density. Sieving of samples is not feasible for wet and adhesive/sticky samples (e.g. sludge) but would be possible for many soil and sediment sample types. As the primary purpose of the sieve is to control the fall velocity it could be considered filling the cylinder gently without the use of the sieve, however this option is not mentioned.

The standard ISO 11272 (soil) describes three principles for the determination of dry bulk density; the core method, the excavation method and the clod method, of which only the first

principle is considered for other matrices. The core method describes sampling of soil (in-situ) in a metal tool of known volume. The dry bulk density is calculated from the mass of the dried material sampled and the volume of the tool. This sampling method is only relevant for soils free from, or almost free from, stones and practical problems are foreseen also for coarse materials of compost/biowaste and sludge. Thus the method is not considered generally applicable for sludge and biowaste, due to the in-homogeneity of the sample and coarse material.

For the excavation method (or sand replacement method) a soil sample is removed/sampled in-situ and the volume of the soil sample is determined by filling the excavated hole with a known volume of fine sand. The clod method is a method in which aggregates (clods and coarse peds) are removed from the soil and "frozen" exactly the way they appear in the soil by coating them with a water-repellent substance, followed by the determination of the volume by immersion in water. This overcomes the problem that structural aggregates may be crushed and compacted once removed from the soil and placed in a cylinder.

3.2 Objectives of the determination of bulk density

As outlined above a direct harmonisation of the existing European and International standards for the determination of bulk density in soil, sludge and biowaste does not seem relevant without modifications of the existing standards. This is further emphasized by the fact that the objectives for the determination of bulk density differs in the above-mentioned standards for soil improvers and growing media (EN 13040, Annex A and EN 12580) and soil (ISO 11272) For soil the variation in bulk density is attributable to the relative proportion and the specific gravity of solid organic and inorganic particles and to the porosity of the soil. Mineral soils typically have densities between 1 and 2. The principles as described in ISO 11272 covers methods suitable for the determination of bulk density in different soils. In-situ bulk density determination of soil often will have the purpose to determine the soil properties related to plant growth and storage capacity for e.g. water.

For soil improvers and growing media the bulk density is used for physical characterisation of the material in question and for calculation of the composition of the material on a volume basis. This forms the basis on which these materials are traded.

The present report includes only ex-situ measurement of bulk density of solid materials and intends to enlighten the relevance of a horizontal standard covering soil, sludge and biowaste. The successful elaboration of such a standard would enable a comparison of bulk density of different products, whether originating from soil, sludge or biowaste.

Table 2 Comparison of standards for the determination of bulk density. Only information deemed most relevant for the comparison is listed below. Further details are found in the standards

Standard	**EN 12580:1999** **Soil improvers and growing media**	**EN 13040:1999, Annex A** **Soil improvers and growing media**	**EN 13041:2000** **Soil improvers and growing media**	**ISO 11272:1998** **Soil**
Scope/sample materials	Determination of quantity of **soil improvers and growing media** in bulk and in packages. Restricted to materials that do not contain more than 10 % by volume of particles greater than 60 mm in any dimension. **Not applicable to blocks sold as such**.	Determination of laboratory compacted bulk density in **soil improvers and growing media**. Restricted to materials that do not contain more than 10 % by weight greater than 20 mm in any dimension. **(Not applicable to liming material and sewage sludge** – see note in text)	Determination of dry bulk density in **soil improvers and growing media.** (Other parameters are included in the standard as well). **Not applicable to liming material and sewage sludge**.	Determination of dry bulk density in **soil.** 1. Core method: Stoneless and slightly stony soils 2. Soils containing gravel and/or stones 3. Clod method: Clods and coarse peds
Terms and definitions	**Bulk density** Density of material as received or reconstituted for use in accordance with the manufacturer's instructions, determined by the method specified in this standard **Volume** Out-turn volume determined by the method specified in this standard.	**Defines** "test sample" and "test portion". **Laboratory compacted bulk density:** density, expressed in grams per litre of the material as determined in the laboratory using a 1 L cylinder; the sample being compacted under defined conditions	**Dry bulk density** Ratio of the dry mass and volume of the sample in grams per litre **Shrinkage volume** Loss in volume of the sample after drying a moist sample **Water volume** That part of the volume of a sample filled by water measured under the conditions specified in the standard. **Particle density** The ratio of the total mass of oven-dry solid particles (minerals, organic matter) to the volume of these particles. The volume of the internal pores of the particles and the pore spaces between particles are excluded.	**Dry bulk density** Ratio of the oven-dry mass of the solids to the volume of the soil (Including volume of soil and of the pore space)
Principle	The material is weighed, its bulk density is determined from the weight and the volume of the cylinder.	A.2 Filling of a one-litre test cylinder with sample "as received", static compaction and weighing of the contents of the cylinder.	The sample is saturated in water and then equilibrated on a sand box at minus 50 cm water pressure head. Transferred into double ring sample cylinders, re-wetted and equilibrated at minus 10 cm water pressure head. After equilibrium the physical properties are calculated	Core method: Known volume of sample is taken and dried in oven, weighed and the dry bulk density is calculated. Excavation methods: A quantity of soil is excavated, dried and

Standard	**EN 12580:1999** **Soil improvers and growing media**	**EN 13040:1999, Annex A** **Soil improvers and growing media**	**EN 13041:2000** **Soil improvers and growing media**	**ISO 11272:1998** **Soil**
			from the wet and dry weights of the sample in the lower ring.	weighed and the volume of the excavation is determined by filling with sand of known volume. Clod method: Coating with water-repellent substance and weighing in air and immersed in water.
Use of processed material	Not applicable to blocks sold as such.	A.6.1. Restrictions in the use of the processed material for the determination of any physical property.	-	-
Sample storage and preparation	A sample size of not less than 30 litres shall be used for the determination of BD	A.6.2. If not for immediate use the sample should be re-placed into a moisture-proof bag or container and the container re-sealed and stored below 5 ºC but not frozen. A.4.1 Homogenisation of the sample. Sieving of the material through sieve, with 20 mm square apertures. If less than 10 % is retained, this material shall be physically reduced to permit the entire sample to pass the sieve.	Reference to Clause 8.4 EN 13040:1999: Approx 10 L of sample is passed through 25 mm sieve. Any sample not passing shall be physically reduced to less than 25 mm (< 80 mm for flexible fibres).	
General Hazards	-	-	-	-
Apparatus	**Measuring cylinder:** Rigid, (20 ± 0,4) L with a height to diameter ratio between 0,9:1 or 1:1. The volume, V_1, shall be known to the nearest 10 ml at 20 °C. **Collar:** Rigid, of the same diameter as	**Test cylinder**: A rigid test cylinder having a capacity of 1000 ml, diameter of 100 mm and height of 127 mm **Removable collar** **Plunger,** with a mass of 650 g	**Double rings** **Plastic tubes** **Water bath** **Sand suction table**	***Core method:*** **Core sample holder** **Oven** **Desiccator** **Laboratory balance** ***Excavation method:*** **Earth digging equipment** **Sampling equipment**

Standard	EN 12580:1999 Soil improvers and growing media	EN 13040:1999, Annex A Soil improvers and growing media	EN 13041:2000 Soil improvers and growing media	ISO 11272:1998 Soil
	the measuring cylinder and vith a height of (75 ± 2) mm. **Fall controller**: Of either (20 ± 0,6) mm, (40 ± 1,3) mm, or (60 ± 2) mm mesh size as required, held not more than 50 mm above the collar. **Weighing machine:** At capacity of > 40 kg max. scale interval of 200 g. **Straight-edge:** Rigid, of rectangular cross section, or a knife, and at least 200 mm longer than the diameter of the collar.	**60 °funnel** **Sieve, 20 mm square apertures** **Analytical balance,** scale interval of 1 g and capacity of 5000 g **Straight edge** **Tripod** **Scoop**	**Ventilated drying oven** **Analytical balance** **Shallow vessel, spoon or scoop**	**Equipment for collecting and cleaning** **Plastic film** **Equipment for spreading of sand** **Dry graded sand** **Balance** **Oven** **Vacuum desiccator** **Sieve, 2 mm apertures**
Interferences	-	-	-	-
Procedure	Describes the determination of bulk density, volume and mass of material. Bulk density: Empty cylinder is weighed and put in horizontal position. Any material in the final sample, which has been compressed or dried, shall be loosened or moistened for usage according to the manufacturer's instructions. Material is passed thorough the sieve (fall-controller) and the cylinder and collar is filled. Collar is removed and the material is levelled off. The mass of the filled	Empty cylinder is weighed and put in position. Material is passed thorough the sieve and the cylinder is filled. The plunger is placed on the material in 180 s and removed. Material of compaction more than 50 mm is reanalysed without compaction. Special procedure if coarse, fibrous, woody or otherwise material is present. The analysis is carried out in triplicate.	1. Equilibrium at minus 50 cm pressure head 2. Filling tubes 3. Suction 4. Separation of rings, drying of the ring + sample, measuring the dry volume and dry weight	**Core method:** Core of known volume is pressed into the soil and filled. Core with content is dried and weighed**.** **Excavation method:** A hole is dug in the soil and the soil is removed and stored for laboratory weighing. The whole is covered by plastic and filled with known volume of sand. The soil is separated in sand (<2mm) and stone and gravel and dried and weighed separately. **Clod method:** Clods and peds are separated from other soil constituents. Weighed and coated in oil. Coated clods and peds are

Standard	EN 12580:1999 Soil improvers and growing media	EN 13040:1999, Annex A Soil improvers and growing media	EN 13041:2000 Soil improvers and growing media	ISO 11272:1998 Soil
	cylinder is determined.			weighed in air and immersed in water. Density of water is calculated for temperature table. Correction of water is carried out after drying and weighing.
Calculation	Bulk density is calculated in grams per litre	The (wet) bulk density is calculated as the arithmetic mean of all the results	Dry bulk density is calculated in kilograms dry matter per cubic metre	The dry matter is expressed in percentages by mass or grams per kilogram.
Quality control	-	Weighing of the empty cylinder and of the cylinder filled with water. The difference should equal 1000 g ± 30 g. Difference to repeated test should not exceed ± 5 g.	The volume of the lower sample ring is controlled by the manufacturing requirement.	-
Precision	Reference is made to an interlaboratory study.	Repeatability and reproducibility of the compacted material: Reference is made to an interlaboratory study.	Reference is made to an interlaboratory study, however applicability is dependent on nature of material	-

4. CRITICAL POINTS AND RECOMMENDATIONS

The comparison of standards for the determination of bulk density demonstrates that the existing standards for the determination of bulk density are different and that the use of data from bulk study determination has different purposes for the different matrices. It further demonstrates that none of the identified European and International standards can be used directly as basis for a harmonized standard covering soil, sludge and biowaste:

Standards for the determination of bulk density exist for soil (ISO 11272) and for soil improvers and growing media - 2 standards (EN 13040, Annex A and EN 12580:1999). A third draft European standard for soil improvers and growing media (prEN 15238) is not considered in the present report, as it relates to particles greater than 60 mm.
The ISO standard for determination of dry bulk density for soil (ISO 11272) is not considered feasible for biowaste, e.g. compost.

One method for soil improvers and growing media is described for compacted bulk density (EN 13040, Annex A) and one method for soil improvers and growing media is described for bulk density without compaction (EN 12580). Results from the use of these two standards are not expected to be comparable. EN 13040 is restricted to material with less than 10 % of particles greater than 20 mm in any dimension and EN 12580 is restricted to material with less than 10 % of particles greater than 60 mm in size.

It would in principle be possible to use one of the EN standards developed for soil improvers and growing media (EN 13040, Annex A or EN 12580) also for soil samples, with possible modifications. However, due to an expected compaction and disturbance of the soil particles by using EN 13040, comparability with the in-situ methods for soil is not expected. Results for the use of EN 13040 for determination of bulk density in soil samples is expected to be less accurate compared to e.g. in-situ measurements of bulk density in soil.

No standard or drafts standard for the determination of bulk density in sludge have been identified in the present study. Elaboration of a horizontal standard covering also sludge is a possibility. The compression of dried sludge should be avoided, thus leaving the principles in EN 12580 a better alternative than the principles in EN 13040. It should be considered, however, to modify the procedure as described in EN 12580 for wet and sticky materials, where the use of sieves as fall control may be less appropriate. The use of EN 12580 demands a larger test cylinder to be used than the test cylinder in EN 13040 (20 L cylinder contra 1 L cylinder) and allows more coarse particles to be included (fall-controller max 60 mm contra max 20 mm). The applicability of EN 12580 in terms of matrices is thus broader.

EN 12580:1999 has been validated for different types of growing media (biowaste, peat and clay) and soil improvers (biowaste, bark and peat), however there is no data on sludge and soil types apart from clay. Thus there is a need for an interlaboratory comparison in this respect. The interlaboratory study has revealed a reproducibility relative standard deviation of 4 – 13 % in materials comprising peat, bark, clay, biowaste and wood fibres.

The broad range of materials that have been tested in the validation studies in TC 223 may give an indication of the expected intermediate standard deviations expected for other materials (sludge and soil). An interlaboratory validation of sludge and soil, which will determine the interlaboratory standard deviation, is needed prior to standardization. However, the gained information by production of intermediate standard deviations (“within one laboratory reproducibility) within an evaluation test is deemed too low compared to the investments.

Suitable standards already exist in the area of growing media and soil improvers. The value of a horizontal standard for soil, where suitable in-situ methods exist, is questioned. Interests have so far not been identified for the area covering sludge. A horizontal standard can be produced covering soil, sludge and biowaste, however the added value on standardization of a horizontal standard on ex-situ bulk density without the expressed needs may be questioned.

A horizontal standard for the determination of bulk density, based on the principles mentioned in the normative EN 12580 is proposed. A draft standard has been produced for this purpose.

5. DRAFT STANDARD

It is proposed that the EN 12580 be used as the basis for a horizontal standard for the determination of bulk density in sludge, soil and biowaste. The following major technical and editorial changes are proposed:

1) The draft standard is edited according to the general rules for drafting standards and the specific requirements as given for editing horizontal standards.
2) A.1.1: …..soil improvers or growing media …… is changed to sediment, soil, sludge and soil improvers and growing media.
3) A line is added in chapter A1. *When used for wet and sticky material, such as sludge, the use of the fall control device is omitted.*
4) Reference to methods and recommendation for the determination of dry matter in enclosed.
5) Determination and calculation of the gross volume and mass is omitted.

A draft horizontal standard for determination of bulk density in sediment, soil, sludge, soil improvers and growing media and following the above described principles is elaborated

REFERENCES

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