

March 2006

HORIZONTAL 33-1

Project 'Horizontal'

Desk Study DS 33-1:

Horizontal standards for soil, sludge and treated bio-waste -

Report of the Desk-Study "Pre-treatment for physico-chemical and inorganic parameters

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ACKNOWLEDGEMENT

This work has been carried out with financial support from the following EU Member States: UK, Germany, France, Italy, Nordic Countries, Netherlands, Denmark, Austria, EU DG XI and JRC, Ispra.

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Summary

The scope of this desk study DS-33.1 was the development of a horizontal draft standard on the pre-treatment of soil (including sediments), sludge, bio-waste and (soil-related) waste samples, concerning the current practices on the pre-treatment of samples prior to chemical analysis of inorganic constituents and physico-chemical measurements.

Sample pre-treatment is an important link between the sampling procedure and the chemical analysis and physico-chemical measurements respectively. But there exists no clear border-line between the end of sampling and the beginning of the pre-treatment and the beginning of the testing.

Because of these uncertainties sample pre-treatment in this desk study is defined as operations and treatment steps done on a field sample/or laboratory sample with the necessary steps applied for the production of a homogeneous test sample used for the subsequent analytical measurements.

Although in standards the term pre-treatment is used for both sample handling and manipulation in the field as well in the laboratory a linkage between both steps is missing. Therefore in accordance with the definition given above it is proposed to combine the sample pre-treatment steps which can be carried out in the field soon after sampling with pre-treatment steps that are restricted to the laboratory in one standard.

The comparison of standards on sample pre-treatment demonstrates that there is a limited possibility of preparing a harmonised and horizontal standard for sediment, sludge, soil and waste. The horizontal standard may integrate ISO/FDIS 11464 and document CEN/TC 308/WG 1/TG 4/N0058 completely and consider several clauses of ISO/FDIS 10381-8. However numerous standards and draft standards are dealing with sample pre-treatment more or less but refer to ISO/FDIS 11464 if pre-treatment requirements have to be specified – only some of them give further instructions. Such requirements are respected as far as possible.

In consideration of document CEN/TC 308/WG 1/TG 4/N0058 the treatment of sludge can be integrated in a horizontal sludge because it is treated like a soil. Liquid sludge is integrated as well even though its treatment differs from that of solid sludge.

Some standards in the field of interest are not suitable for a horizontal standardisation because of methodological differences from the standards mentioned above and missing agreement throughout Europe. For example draft standard NEN 5709 permits the crushing of the whole soil sample while the general accepted ISO/FDIS 11464 requires the removing of extraneous material and EN 12457-1 to -4 as well as EN 13040 (for soil improvers) require sieving steps that could not be integrated.

1 Introduction

The objective of the project HORIZONTAL is the development of across-matrix, horizontal approaches (standards) for the characterisation of soils, sludges and bio-wastes. Up to now considerable attention was given to the issues of sampling and the measurement processes themselves, while the intermediate step of sample pre-treatment was neglected. In order to close that gap a strategy towards the development of a horizontal sample pre-treatment standard was proposed by the Joint Research Centre – Institute for Environment and Sustainability (JRC-IES).

The work items of Project HORIZONTAL are subdivided into three parts. The first part is the performance of the desk studies with additional elaborations of proposals of horizontal approaches on the basis of existing standards dealing with the issue. The next step will be the experimental examination of the proposed approach by a series of tests on soils, sludges and biowastes. The third part will be the actual validation – proofing that a given standard is “fit for purpose”.

The scope of this desk study is the development of a horizontal draft standard on the pre-treatment of soil (including sediments), sludge, bio-waste and (soil-related) waste samples, concerning the current practices on the pre-treatment of samples prior to chemical analysis of inorganic constituents and physico-chemical measurements. Reference documents to be mentioned as a starting point are for instance ISO/FDIS 11464, ISO 14507 and ISO 5667-15. Beside bio-waste other waste materials are not included at the moment, since no interest has been expressed so far. Hygienic and microbiological parameters are not included in this study either.

This desk study is dealing with aspects on sample pre-treatment as follows:

- ∅ A definition of sample pre-treatment in the context of this desk study is given.
- ∅ Available vertical international (ISO), European (CEN), and national standards (EU) as well as of CEN committee drafts dealing with pre-treatment and testing of the matrices mentioned above are assessed critical.
- ∅ The different methods and the potentials for harmonisation are evaluated.
- ∅ Critical points are described and recommendations for a harmonized draft standard are given.
- ∅ A draft standard is generated.

2 Sample pre-treatment – a definition

Standards dealing exclusively with sample pre-treatment in the field of interest of this desk study are scarce. Basically standardized methods on soil and water pre-treatment and a few in the field of sludge treatment are available. Nevertheless, the borderline between the end of sampling procedure and the start of the pre-treatment as well as the end of the pre-treatment and the beginning of testing standard is fuzzy.

Because of this uncertainty the development of a horizontal standard on sample pre-treatment requires a definition on what should be understood by pre-treatment, that is, which steps should be implied. In the context of this desk study and the generated draft European Standard

sample pre-treatment is defined as operations and treatment steps done on a field sample and/or laboratory sample with the necessary steps applied for the production of a homogeneous test sample used for the subsequent analytical measurements.

The definition excludes explicitly the issue of extraction and digestion of analytes. Hygienic and microbiological parameters are not included in this work as well.

Most sample pre-treatment steps as defined above have to be performed in the laboratory. But some treatment steps are not restricted to the laboratory since first pre-treatments like sample division may be required and realised in the field. Sub-sampling by sample division may be achieved in the field for example if samples are too large to take to the laboratory or if the amount of material sampled is larger than the amount of material necessary for the laboratory sample and subsequent analysis.

In particular, sample pre-treatment implies the issue of storage, homogenisation, and sub-sampling (sample division) and includes operations such as stabilization (drying), particle size reduction (milling, grinding) and fractionation (sieving). It has to be pointed out that further analytical steps such as the efficiency and characteristics of digestion and extraction are strongly influenced by the pre-treatment applied.

3 Methodology

For critical assessment of available working results general information were compiled step by step as follows:

- Ø Check of the database (DIN and Perinorm).
- Ø Receiving standards, draft standards and adequate working documents by DIN.
- Ø Existing standards, draft standards and further documents were assessed and key points of supposed differences were identified and the differences were evaluated in order to prepare a draft horizontal standard.
- Ø Ongoing activities in the CEN/TC working groups were assessed:
CEN/TC 292/WG 1, WG 5 Waste sampling and analysis of waste
CEN/TC 308/WG 1 Sludge (Prof. Dr. Leschber)
CEN/TC 345 Soil (Prof. Dr. Dr. Terytze)
CEN/BT/TF 151 (A. Paetz)

4 Existing standards or draft standards

Quite a number of standards and draft standards are dealing with sample pre-treatment more or less. The available standards, draft standards and documents on sample pre-treatment exclusively or partly are listed in Annex A, table 1.

Only a few standards are dealing with the pre-treatment of soil, sludge or biowaste samples exclusively.

Especially for the matrix soil two ISO standards, namely ISO/FDIS 11464 and ISO 14507 were available. In addition an actual standard by the Netherlands Standardization Institute of Normalization NEN (draft NEN 5709) is on the pre-treatment of soil samples only.

Additional methods for soil pre-treatment are given in ISO 10381-1 and ISO/FDIS 10381-8, which are determining requirements on sampling as well as on storage of sampled material mainly.

For sludge, an ongoing CEN/TC draft version from August 2003 (CEN/TC 308/WG 1/TG 4 N0058) on pre-treatment and additional a standard on sampling of sludges (ISO 5667-13) and one on preservation and handling of sludge and sediment samples (ISO 5667-15) were reviewed.

In the field of soil improvers and growing media two European standards on sampling (EN 12579) and sample preparation (EN 13040) were examined.

A comparison of the documents ISO/FDIS 11464, draft NEN 5709, CEN/TC 308/WG 1/TG 4 N0058, ISO 5667-13 and ISO 5667-15 is given in Annex A, table 2a and 2b.

A lot of standards refer either to ISO/DIS 11464 or to ISO/DIS 14507 if pre-treatment requirements have to be specified. However, only some of them give further instructions. Such "hidden" requirements are considered and included in the horizontal draft standard as far as possible to avoid sample pre-treatments that could be unsuitable for some analysis. An outline of the sample material and the principles within the standard's scope on sample is listed in table Annex A, table 3.

5 Evaluation of the suitability for horizontal standardization

5.1 Relevant standards

It should be regarded that every type of pre-treatment will have an influence on several material properties and the results of subsequent investigations.

Thus pre-treatment steps that can be performed horizontal on each matrix and prior the analysis of several parameters have to be evaluated as carefully as any recommendation on the separation of sample pre-treatment steps.

For the purpose of this desk study the most relevant documents on sample pre-treatment that have been assessed are

- ∅ for soil: ISO/FDIS 11464, ISO/FDIS 10381-8, draft NEN 5709;
- ∅ for soil, sediments and waste: ISO/FDIS 11464, ISO/FDIS 10381-8, ISO 14507;
- ∅ for sludge: ISO 5667-13, ISO 5667-15, document CEN/TC 308/WG 1/TG 4 N0058;
- ∅ for biowaste: EN 12579, EN 13040.

However, as mentioned above two documents on pre-treatment are cited by analytical standards mainly: ISO/FDIS 11464 in the field of inorganic contaminants and ISO 14507 if organic contaminants are measured. For the purpose of this desk study on pre-treatment of samples prior inorganic analysis the international standard ISO 14507 is not as important as other documents listed in Annex A, table 1 since it's scope is limited to sample preparation prior organic contaminant analysis.

Requirements determined in several other standards are restricted on narrow methodological aspects, e. g. how drying, crushing or milling have to be performed (see Annex A, table 1 and 3).

The Dutch draft standard NEN 5709 comprises three pre-treatment methods for soil samples. The first one is comparable to ISO 14507 and only suitable prior to the analysis of organic contaminants. Due to chemical drying with sodium sulfate prior to grinding it is unsuitable for the analysis of inorganic contaminants. The second method is comparable to ISO/FDIS 11464 and can be performed when non-volatile inorganic contaminants are analysed. The soil sample is dried in the air, in the desiccation stove at 40 °C or 75 °C or by freeze-drying prior to grinding. The third method is suitable for inorganic and organic contaminants which are semi- or non-volatile. It is the fresh sample pre-treated, but cooled by nitrogen or solid CO₂ (means: no drying step prior to grinding).

The standard ISO/FDIS 10381 comprises eight parts. Part 8 is a general guidance on the sampling of stockpiles. Besides other methods it defines the sample pre-treatment directly after sampling (when necessary) and the packing, preservation, storing, transport and delivery of the sample.

ISO 5667 consists of 19 parts, prepared by the Technical Committee ISO/TC 147, water quality. Guidance on sampling, storage, preservation and handling of sludge are provided by ISO 5667-13 and ISO 5667-15. ISO 5667-13 is dealing with sampling and sub-sampling and in ISO 5667-15 preservation techniques and sample containers suitable for different requirements are described.

The document CEN/TC 308/WG 1/TG 4 N0058 is a working draft on pre-treatment of sludge for subsequent analysis from August 2003. It refers to three standards already mentioned above: ISO 5667-15, ISO/FDIS 11464 and ISO 14507. The draft document of CEN/TC 308 specifies the pre-treatment required for sludge samples that are to be subjected to physico-chemical analysis and describes the following types of pre-treatment of samples: storage, drying and sieving. Because high water contents may cause difficulties in the analysis of sludges it is distinguished between liquid and more or less solid sludge samples.

The desk study “pre-treatment for organic parameters” has been checked carefully. It was decided that this study is not finished yet for several reasons: a draft pre-treatment standard is missing – therefore a comparison of treatment steps is not possible –, the flow sheet seems to be incomplete and the methodological basis is ISO/FDIS 11464 more than ISO 14507. Furthermore the crushing under cryogenic conditions is not applicable to the treatment for inorganic and physico-chemical parameters.

EN 13040 defines the preparation of soil improvers and growing media samples for chemical and physical tests, the determination of dry matter content, moisture content and laboratory compacted bulk density. The sample pre-treatment is an integrated part of the standard. EN 12579 is suitable for the sampling of soil improvers and growing media only.

5.2 Comparison of pre-treatment requirements

In the following the main similarities and differences of ISO/FDIS 11464, draft NEN 5709, ISO 5667-13, ISO 5667-15 and document CEN/TC 308/WG 1/TG 4 N0058 are evaluated by comparison of the requirements (see Annex A, table 2a). An abstract of ISO/FDIS 10381-8 is given in Annex A, table 2b.

EN 13040 is not included in Annex A, table 2a because it differs from these documents in main points:

- The laboratory sample is sieved by several sieving steps.
- Test samples are the parts which pass a 40 mm, 25 mm and 20 mm sieve respectively. This differs from e. g. soil pre-treatment.

- Samples are dried at (75 ± 5) °C (differs from e. g. soil pre-treatment).

Sample division

In ISO/FDIS 10381-8 methods for sub-sampling by sample division in the field are described. That could be required for example if samples are too large to take to the laboratory or if the amount of material sampled is larger than the amount of material necessary for the laboratory sample and subsequent analysis. The methods of sample division (sub-sampling) described in the informative Annex J are:

- Long pile and alternate shovel method
- Coning and quartering
- Riffing
- Application of Tyler divider

In ISO/FDIS 10381-8 it is also pointed out that “when possible, the sample pre-treatment will take place in the laboratory, as sample integrity can be best guaranteed under laboratory conditions.” The methods described in the informative Annex J are also suitable if the sample division has to be performed in the laboratory – ISO/FDIS 11464 refers to ISO/FDIS 10381-8.

Sample division (sub-sampling) of the field sample – to produce an adequate laboratory sample – or of the sample received by the laboratory if it is too large for further treatment is not included in NEN 5709 explicitly. But the laboratory sample is to be divided in order to get sub-samples for subsequent treatment for organic and inorganic analysis.

Coning and quartering is also submitted if solid sludge has to be divided (ISO 5667-13, CEN/TC 308/WG 1/TG 4 N0058). Prior sample division liquid sludge samples can be centrifuged or filtrated to obtain a sample with a lower water content.

Water

According to draft standard NEN 5709 water should be siphoned off before further treatment steps if a soil sample is too wet. The elimination of water from sludge samples by centrifugation is also submitted in document CEN/TC 308/WG 1/TG 4 N0058. However none of the standards specifies whether the water is analysed – e. g. in the case of water soluble inorganic contaminants – or not. Neither ISO/FDIS 11464 nor ISO/FDIS 10381-8 contain any instructions on the treatment of wet samples.

Drying (air, oven and freeze drying)

ISO/FDIS 11464 specifies that it is essential that the drying temperature does not exceed 40 °C – direct sunlight shall be avoided if the sample is dried in the air. 40 °C is the highest permissible temperature in other standards, too. Only liquid sludge samples could be dried at max. 105 °C. But it has to be remembered that some parameters can be lost at high temperature.

To avoid the loss of contaminants the drying temperature shall not exceed 40 °C. For example Hg is volatile at approximately 60 °C. If metal-organic compounds have to be detected every drying could be a problem and should be avoided.

Therefore draft NEN 5709 is not uniform with European and International standards because it permits drying at 40 °C or 75 °C. With the integrated method the field wet sample is pre-treated without a drying step. But the sample is cooled by N₂ or CO₂.

Storage temperature

Proposals on the storage temperatures of samples deviate slightly in the standards. Samples are stored in a dark place and refrigerated at 2 °C to 5 °C (draft NEN 5709, ISO 5667-15). According ISO/FDIS 10381-8 and ISO 5667-13 the storage temperature should be (4 ± 2) °C and ISO 5667-3 requires 1 °C to 5 °C. ISO/FDIS 11464 specifies no storage temperature.

Particle size

Particle size reduction is restricted to being a laboratory operation (ISO/FDIS 10381-8).

According ISO/FDIS 11464 the dried sample is sieved through a 2 mm sieve. Any large dried particles remaining on the 2 mm sieve should be crushed (using suitable apparatus) to less than 2 mm. Oversized material is removed, recorded and kept for possible further measurements.

Draft NEN 5709 requires the reduction of the whole sample material except non-crushable material (metal etc.). The wet field soil particles are made smaller to $D_{95} < 2$ mm.

Crushing and extraneous material

The most important difference between ISO/FDIS 11464 and draft NEN 5709 are the requirements on the treatment of non-crushable and extraneous materials (see Annex A, table 2a). When pre-treatment is performed in accordance with draft NEN 5709 only parts that cannot be ground (metal etc.) are removed. That is, the sample is crushed as completely as possible.

In contrast to this method ISO/FDIS 11464 requires the elimination of extraneous (means untypical e. g. stones, glass, plastic) material even if it could be ground. This resembles ISO 14507 where “parts that are not representative for the soil e. g. all visible metal parts” are removed.

In accordance with CEN/TC 308/WG 1/TG 4 N0058 solid sludge should be treated as determined in ISO/FDIS 11464.

Document CEN/TC 308/WG 1/TG 4 N0058 requires also the examination and description of the sample and the elimination of extraneous matter (e. g. glass).

In any case the nature and mass of the removed material must be documented. The material has to be kept for separate analysis, if necessary.

Milling

Milling of soil, sludge and sediment particles < 2 mm to < 150 μ m is required for example prior to *aqua regia* digestion according e. g. ISO 11466 or ISO 16772. Milling to a particle size < 250 μ m is required if the total element content according ISO 14869-1, ISO 14869-2 has to be determined. Milling is also recommended if a test sample of less than 2 g is to be taken for the analysis. The sample is milled to a particle size < 250 μ m.

NEN 5709 requires “Make smaller up to $D_{95} < 500$ μ m after drying and < 125 μ m (optional) respectively smaller to $D_{95} < 500$ μ m, if also moderate volatile substances are being determined” (in the integrated method)

6 Critical points and recommendations

The objective of this desk study was the development of a draft horizontal standard on sample pre-treatment suitable for soil, sludge and waste material. For this purpose existing international, European and national (EU) standards, draft standards and related working documents were reviewed and the suitability of the methods for harmonization was assessed. The work package proposal by JRC implied the standards ISO/FDIS 11464, ISO 14507 and ISO 5667-15, draft standard NEN 5709 and document CEN/TC 308/WG 1/TG 4 N0058. These documents and many others were reviewed. The examination had resulted in only a few standards which have to be considered mainly.

Essential requirements are given in ISO/FDIS 11464 and ISO/FDIS 10381-8. For that the requirements are suitable for soil investigations mainly but they are also applicable for soil material, sediments and sludge (see Annex A, table 1 and table 3). Especially for the purpose of sludge analysis ISO 5667-15 and document CEN/TC 308/WG 1/TG 4 N0058 are integrated. All together the resulting draft standard is a mix of these documents mainly. ISO/FDIS 11464 has been taken over completely the other documents were taken into consideration partly. They have to be revised if the proposed draft standard is accepted as it is. Further requirements but to a lesser extent were found in several standards on sample analysis (see Annex A, table 3).

Some pre-treatment methods differ considerably from the main pre-treatment methods suitable for horizontal standardisation and have not been taken into account in the draft standard. For example the series EN 12457 requires “that the tests shall be made on material with a grain size of at least 95 % (mass) less than 10 mm (respectively 4 mm). If oversized material exceeds 5 % (mass) the entire oversized fraction shall be crushed with a crushing equipment. On no account shall the material be finely ground. Non-crushable material (e. g. metallic parts such as nuts, bolts, scrap) in the sample shall be separated”. These requirements differ from e. g ISO/FDIS 11464 and consequently from the horizontal draft standard proposal because the crushing of oversized material is allowed at some circumstances (if oversized material exceeds 5 %).

For the purpose of a draft standard on sample pre-treatment prior inorganic analysis recommendations and critical points are given in the following.

1. The standards examined within the framework of this desk study do not link the pre-treatment steps possible in the field (sample division) with pre-treatments restricted to the laboratory. However a linkage is necessary. For example it has to communicate by the laboratory or the client if pre-treatment in the field is permitted. The requirements have to be included into the sampling plan.
2. For this reason according the definition given in the desk study the draft standard combines pre-treatments suitable for the field with the laboratory methods. This is because sample divisions that could be performed in the field are also applicable for sub-sampling in the laboratory.

On the other side one must take into consideration, that if the staff who executes the sampling and the laboratory analysts are not the same both will need two standards.

Therefore for practical reasons it is proposed to summarize the horizontal standard on sampling and on pre-treatment in only one standard.

3. Draft standard NEN 5709 has been examined carefully and regarded in this desk study. Unfortunately it could not be considered in the horizontal draft standard for several reasons.

Essential methodological differences between draft standard NEN 5709 and ISO/FDIS 11464 have been determined. That is, according draft standard NEN 5709 the sample is treated as completely as possible – only non-grindable material is removed. In contrast ISO/FDIS 11464 requires the elimination of extraneous and oversized material (stones, glass, rubbish etc.) even if it could be ground. The question which part of the sample has to be taken can not be solved by this desk study as long as dissenting opinions do exist.

Draft standard NEN 5709 is a national standard that is obviously not accepted completely throughout Europe. Therefore a general agreement is required before the methods described in draft standard NEN 5709 could become an integral part of a horizontal European pre-treatment standard. It has to be considered, that European directions refer to European but not to national standards.

4. The definition of extraneous material in the draft standard on pre-treatment is similar to that given in ISO/FDIS 11464. That is: everything not typical for the investigated matrix has to be removed. According ISO/FDIS 11464 extraneous matters in soils are e. g. stones, fragments of glass and rubbish. In this context in a soil sample roots are extraneous matter as well. For other matrices the sampling plan respectively the client shall define which extraneous matters have to be removed from the sample and if they should be stored for additional measurements.
5. It should be considered that if extraneous matter and the skeleton are not removed from a heterogeneous sample this material can “dilute” the fraction of interest. This is the grain size ≤ 2 mm when heavy metals are analyzed for example.
6. It is proposed to mill the material generally to a grain size ≤ 150 μm if the mass required is smaller than 2 g or if the sample is needed for extractions.
7. The integrated pre-treatment method according NEN 5709 has not been regarded because of lacking agreement on the method: cooling of the whole field wet sample without drying with N_2 or CO_2 and grinding of the whole sample.
8. ISO 14507 has been regarded but is not considered because sample pre-treatment prior inorganic analysis is described only.
9. The existing separation into two pre-treatment methods – one prior organic another prior inorganic analysis – should be taken as it is because of lacking agreement on an integrated method.
10. Sludge: In accordance with the proposal CEN/TC 308/WG 1/TG 4 N0058 in the draft standard on pre-treatment the method is parted depending on the water content. More or less solid sludge should be treated like soil as described in ISO/FDIS 11464, liquid sludge should be treated like a sludge (is described separately in the desk study).

In document N0058 it is proposed to centrifuge or filtrate sludges with high water content. However whether the filter is specified nor a percentage for “high water content” is given.

11. The pre-treatment of biowaste is not integrated because there exist no specific method.
12. For some contaminants the field wet sample has to be analysed (e. g. cyanide). This requires the integration of a homogenizing and sub-sampling step before the drying step in

the laboratory. Either the field sample or, better, the laboratory sample is dried. Sub-sampling prior drying is also necessary if a sample is dried by 105 °C (e. g. according EN 1233).

13. Sample sizes are taken into consideration in the draft standard as far as possible. But it is not possible to give examples for every situation, because the sample size depends for example on the matrix, the number of parameters and the number of duplicate analysis. For instance the size of a sludge sample depends on the water content.
14. To avoid the loss of contaminants the drying temperature shall not exceed 40 °C. For example Hg is volatile at approximately 60 °C. Draft NEN 5709 is not uniform with European and International standards because it permits drying at 40 °C or 75 °C.
15. A drying temperature of 40 °C in an oven is preferable to air drying at room temperature because of the increased speed of the drying (ISO/FDIS 11464). Anyway, the term "room temperature" is very imprecisely and can differ considerably between laboratories.
16. Some standards advise the analyst to reject water from wet or liquid samples but remain unclear in statements on how the water should be treated (e. g. NEN 5709, CEN/TC 308/WG 1/TG 4 N0058). To close the gap it is suggested to store the water if a balance of the total contaminant content inclusive of water soluble contaminants is required.
17. Regulations on the sub-sampling of replicates are somewhat indifferent. In ISO/FDIS 11464 regulations on replicate analysis are given in a very broadly way: "When replicate analysis are required, it shall be clarified in the overall investigation plan at which stage of sub-sampling replicates must be separated. The most representative stage would be a very early one." This formulation is somewhat inexact and has to be more precise in the draft standard.
18. EN 13040 should not be integrated in the horizontal standard because of several reasons:

The separation of the pre-treatment method from the chemical and physical test procedures will result in a confusing "rest standard".

The laboratory sample is sieved by several sieving steps different from ISO/FDIS 11464 and samples are dried at (75 ± 5) °C (differs from e. g. soil pre-treatment).
19. EN 12579 is suitable for the sampling of soil improvers and growing media only. This document is not relevant for the desk study on pre-treatment.

Annex 1 Tables

Annex 2 Draft Standard