

Methods Book

**for the Analysis
of Compost**

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Federal Compost Quality
Assurance Organisation (FCQAO)
Bundesgütegemeinschaft Kompost e.V. (BGK)**

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Preface

Essential prerequisites for a workable quality assurance system have been created with the development of a binding methods book for the investigation of composts within the scope of the outside monitoring of the compost quality by the Federal Compost Quality Assurance Organization FCQAO.

Through the broad distribution that the methods book has had in the meantime and the realization of the 1993 interlaboratory test, a series of competent replies with fruitful criticism of individual methods, regarding feasibility and practicality, have come in at the Federal Quality Association.. Modification and improvement suggestions were discussed by the Federal Quality Association and are reflected in this third edition of the methods book. For the determination of the conductivity, for example, the simpler extraction with distilled water was fallen back on again. Uncertainties that were still existing, such as the calculation of the C/N relationship or the correct designation of the individually determined nitrogen fractions, for instance, were able to also be clarified through additions or explanations. Further supplemental methods for the analysis of composts were proposed to the Federal Compost Quality Assurance Organization and are under discussion at present. These methods will become established in Chapter III in the course of the updated continuation of the method book.

The prerequisites for the qualified analysis of compost have been improved further with the third edition of the method book. Sound and competent quality assurance of composts and compost products can consequently be guaranteed now and in the future.

The 4th issue of the Methods Book contains for the first time the statistical quotations “**frequent range of values**” which shows the 10 - 90 % percentile range and the “**repeatability limit**” which gives a critical difference amount of repeatability limit which is allowed for 2 individual measuring results with a given probability of 95 %. Basis are the repeatability standard deviations of the ring tests carried out by the German Compost Quality Assurance Organization in the years 1993 and 1995. The calculation of the repeatability standard deviations is carried out according to German Standard DIN 55 350 T 13. The repeatability standard deviation as variation parameter is the measure for the repeatability precision and characterizes the efficiency of exact analyzing methods. The repeatability limit result from the multiplication of the repeatability standard deviation with the factor $1.96 \times \sqrt{2}$.

Cologne, February, 2003

Dr. Bertram Kehres, Bundesgütegemeinschaft Kompost e.V. (Federal Compost Quality Assurance Organisation FCQAO)

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Carrying out the taking of sample

1 Preliminary remark

In order to be able to get representative analytical findings, the taking of the sample may only be burdened with incidental, i.e. unavoidable, errors. According to Garvert, the sample taking should fulfill three conditions to the extent possible.

"It should

- a. supply a representative sample
- b. be able to be carried out with low effort and
- c. not require a major technical expenditure."

Compost samples that are examined by the Federal Compost Quality Assurance Organisation within the scope of the outside monitoring of the compost quality may in principle only be taken from goods ready for sale !

If the goods ready for sale have already been classified, the classification is to be indicated. If they have not been classified, they are to be classified at the taking of the sample. Sieves with a mesh width of at least 10 mm, 40 mm at the most, though, have to be used. The mesh width is to be indicated (see Chap. II, Method 2).

2 Materials

No devices or materials may be used for the sample taking, processing and packing that could pass on one of the materials to be analyzed to the samples.

3 Carrying out the taking of the sample

3.1 Taking a sample from a profile section

- the profile section is to be dug out with a wheel loader
- the entire profile section height is to be sampled equally
- the minimum amount of the partial sample per section wall should be 30 liters

3.2 Taking a sample from a dormant deposit

- bore in the respective pit section to the pit floor (screw borer)
- cross section: at least 5 borings of the same type

3.3 Taking a sample from agitated compost

- see Table 1.1 for the number of individual samples
- the minimum amount of the partial sample should be 30 liters

4 Remarks

Definitions:

- An **individual sample** is an amount of substance taken out in one work step.
- A **collective sample** consists of several individual samples that were thoroughly homogenized.

4.1 Individual samples

The minimum number of required individual samples depends on the mean grain size of the compost or waste and on the amount to be evaluated. Table 1.1 gives a basis for the establishment of the minimum number of individual samples.

Table 1.1: Minimum number of individual samples in dependence on the grain and condition of the compost batch

Grain size of the compost or of the waste	Minimum number of individual samples				
	from agitated substances dependent on the volume flow		from dormant composts or wastes in vehicles	from dormant composts or wastes in deposits	
	up to 50 t	> 50 t		50 - 150 t	>150 t
> 20 mm	10	1 per 5 t	10 per vehicle	1 per 5 t	15
< 20 mm	5	1 per 10		1 per 10 t	10

The minimum volume of an individual sample should be 5 liters for a grain size of > 20 mm and 3 liters for < 20 mm.

The minimum amount of an individual sample can also be calculated according to the following formula:

$$G \text{ [kg]} = 0.06 \times d \text{ [mm]}$$

The coarser and the less uniform the material, the larger the individual sample and the collective sample have to be selected.

4.2 Collective samples

The collective sample gotten from the individual samples is well mixed on a plastic foil (flat spreading and a joining of sectors lying across from one another). The reduction of the collective sample takes place by throwing out a quarter to a third of the material. Following this, mixing takes place again and a portion of the sample is thrown out anew.

This procedure is repeated until the desired amount of sample is left (see Garvert).

In the case of a collective sample of more than 100 l, the mixing method described above can only be carried out with substantial difficulties under certain circumstances. The case can also arise with very damp material that the sample can no longer be sufficiently homogenized. The homogenization of the sample is to be carried out through repeated heaping in a cone and pulling the material apart. The mixing has to take place on a secure and clean foundation (concrete floor, asphalt) or on a solid foil.

4.3 Transport:

- the samples are transported into the laboratory in well-sealed PE containers
- the sample amount should be at least 20 liters
- the sample has to be in the laboratory after 24 h, if possible, and be cooled if necessary
- deviations from the described methods have to be carefully documented

5 Literature

Anonymous; LAGA Guidelines; Garbage Handbook Volume 2

Garvert, U.; The technique of taking samples and annual variations of important parameters of garbage-slurry composts; Dissertation 1977.

Meyer-Spasche, H.; General rules for taking a compost sample; Working regulation of the Institute for Soil Ecology and Environmental Assessment; Bohlsen 1992.

Sample preparation in the laboratory

1 Preliminary remark

The sample taker delivers fresh compost samples to the laboratory. They are designated as the original sample in the following. The original samples are prepared and treated further in the laboratory.

The preparation and homogenization steps can be taken from the procedure diagram on page 8.

2 Materials

- Sieve with a mesh width of 10 mm, 2 mm and 0.25 mm
- Suitable grinders
- Scales (0.1 g reading)
- Drying chamber (adjustable, 40°C and 105°C)

3 Procedure steps for the sample preparation

3.1 Analyses in the fresh substance

Screening the fresh original sample

For all analyses that are carried out in the fresh substance (see Chap. II, Methods 3, 4, 5, 6, 7, 8, 9, and Chap. III, Methods 1, 2, 3, 4), a portion of the original sample has to be screened down to < 10 mm (approx. 20 l sieve through fraction).

The amounts from the sieve overflow and sieve through fraction are to be determined with a gravimetric method during this and are to be stated in connection with the total mass.

$$\text{STF}_r = [\text{STF} / (\text{STF} + \text{SOF})] \cong 100 [\%]$$

$$\text{SOF}_r = [\text{SOF} / (\text{STF} + \text{SOF})] \cong 100 [\%]$$

STF_r : Sieve through fraction in [%]

SOF_r : Sieve overflow in [%]

STF : Sieve through fraction in [g]

SOF : Sieve overflow in [g]

Adjusting to an optimum water content using a fist test

For the determination of the degree of rotting on the basis of self-heating or exchange activity of the sample material (see Chap. II, Method 4 and Chap. III, Method 1), a portion of the fresh sieved substance (approx. 5 liters) has to be adjusted to an optimum and standardized water content. The adjustment to the optimum water content takes place using the so-called 'fist test'; the execution is described in Chap. II, Method 4.

3.2 Analyses in the dry substance

Drying the unscreened original sample at 105°C

For the determination of the water content and of the parameters with regard to the dry substance, approx. 5 liters of the unscreened original sample are dried at 105°C up to a constant weight.

A portion of the dried material is kept as a reserve sample (approx. 1 liter).

For the determination of the foreign matter and stone content, 1-3 liters of material are necessary, depending on the grain of the original sample (see Chap. II, Method 10).

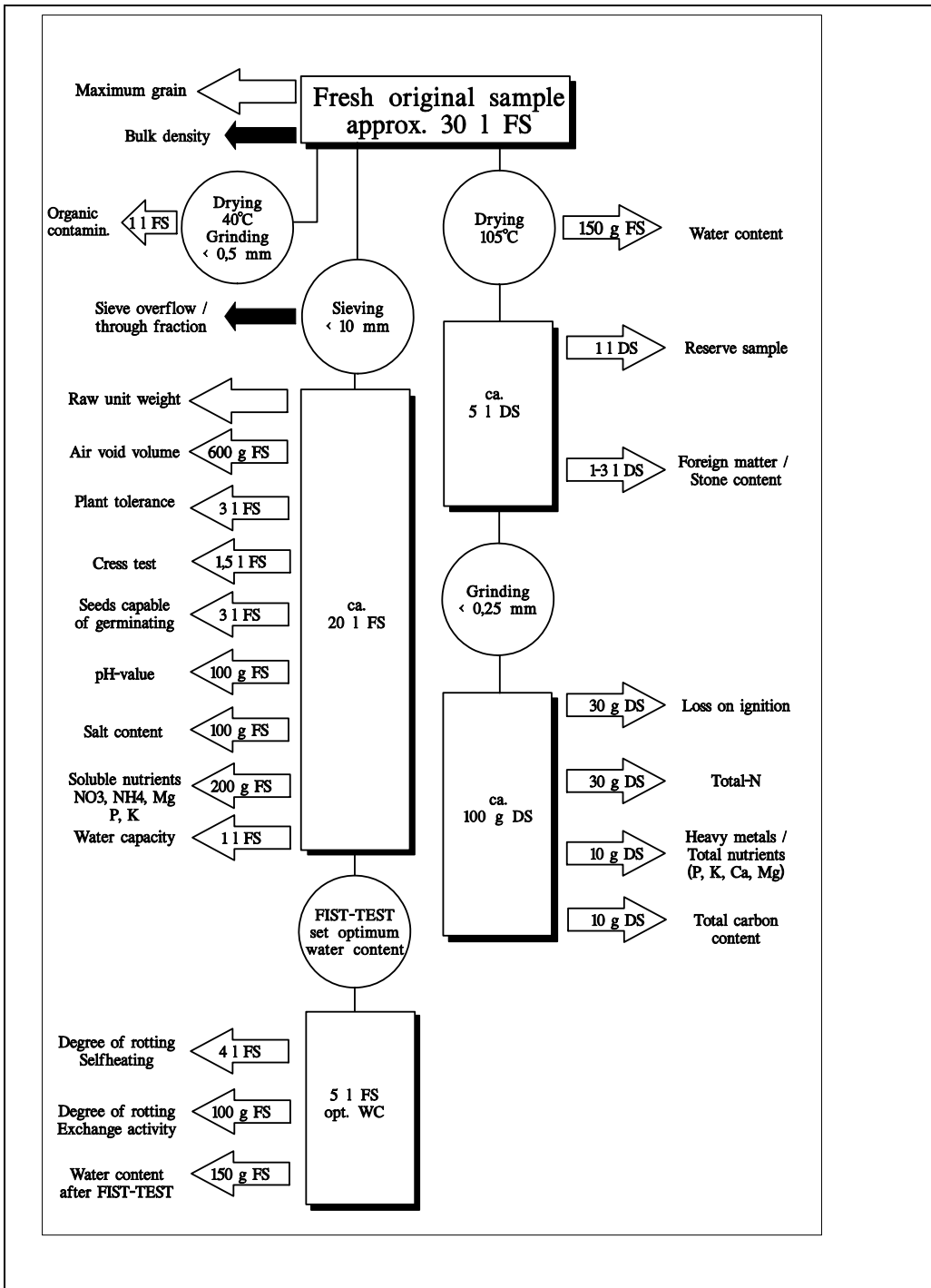
Grinding the dried sample material

At least 100 g of the dried sample are ground with suitable grinders to < 0.25 mm for further analyses (see Chap. II, Methods 11, 12, 13.1, 13.2 and Chap. III, Method 6).

No contamination of the sample with the heavy metals to be determined may take place during the preparation of the heavy metal analysis (see Chap. II, Method 12). Planetary ball grinders (fused corundum, zirconium oxide) or flywheel type belt grinders (tungsten carbide) suggest themselves as suitable tools.

Drying the unscreened original sample at 40°C

A portion of the fresh material (approx. 1 liter) is dried at 40°C for the determination of the organic contaminants (see Chap. III, Method 5) and subsequently ground to < 0.5 mm.



PROCEDURE DIAGRAM

Determination of the water content

1 Preliminary remark

A portion of the fresh original sample has to be dried for the execution of various analyses (foreign matter content, loss on ignition, heavy metal and total nutrient contents). The water content is simultaneously determined during this. A portion of the dried material (approx. 1 l DS) is to be kept as a reserve sample (RS).

2 Sample preparation

A portion (approx. 3 - 5 l FS) of the fresh, unscreened original sample is used for the drying of sample material and for the determination of the water content.

3 Materials

- Scales (0.1 g reading)
- Drying chamber for 105°C
- Suitable vessels (large glass, porcelain or aluminum basins) or suitable plates

4 Carrying out the testing

A representative sample is weighed in for the determination of water content, spread out in a thin layer on plates of the drying chamber or loaded into suitable vessels and dried at 105°C up to a constant weight (24 hours as a rule). The sample is weighed back in directly after the removal from the drying chamber with suitable scales.

5 Calculation of the results

The water content is related to the fresh mass. The result is to be stated in [% DS], accurate to one decimal place.

$$\text{WG} = [(M_{\text{moist}} - M_{\text{dry}}) / (M_{\text{moist}} - M_{\text{tara}})] \cong 100 [\%]$$

$$\text{DS} = [(M_{\text{dry}} - M_{\text{tara}}) / (M_{\text{moist}} - M_{\text{tara}})] \cong 100 [\%]$$

WG : Water content in [% DS]

DS : Dry substance in [% DS]

M_{tara} : Mass of the empty basin (plate) in [g]

M_{moist} : Mass of the moist sample + M_{tara} in [g]

M_{dry} : Mass of the dried sample + M_{tara} in [g]

6 Remarks

The total amount of the sample to be dried depends on the grain of the original sample, because different amounts of dry sample mass are required for the determination of the foreign matter and stone content (Chap. II, Method 10), depending on the grain.

The total sample to be dried does not necessarily have to be used for the purely analytical determination of the water content. The drying of a small representative amount of sample (at least 150 g DS) appears to be sufficient, especially with fine-grained material (0 - 10 mm).

In order to be able to relate parameters, that are determined in the < 10 mm sieved fresh substance (soluble nutrients, salt content, raw unit weight), to the dry substance, the water content of a correspondingly prepared sample (sieving < 10 mm) has to additionally be determined.

7 Literature

VDLUFA, Methods Book, Volume I (1991): The investigation of soils

DIN 38 414 (S2), German uniform proc. for water, waste water and slurry investigation;
slurry and sediments: Determination of the water content and of the dry residue

Determination of the maximum grain size

1 Preliminary remark

In the case of a known pre-screening (classification), the statement of the sieve mesh width suffices. Otherwise, the maximum grain of the fresh original sample is to be determined after sieve analysis.

2 Sample preparation

Fresh original sample, dry, if appropriate to the sieve capability.

3 Materials

- Suitable sieve (gradation of the sieve mesh widths: 5 mm)

4 Carrying out the testing

Sieve analysis up to finding the smallest sieve mesh width, for which no material remains any longer as sieve overflow.

5 Calculation of the results

Statement of the sieve mesh width in [mm].

Determination of the raw unit weight

1 Preliminary remark

Different parameters of composts are related to the volume of the fresh substance (salt content, soluble nutrients). The determination of the raw unit weight of the fresh compost substance is necessary for the conversion of the value related to the fresh mass at first. For the determination of the raw unit weight, the material has to be prepared in the same way as for the determination of the above-mentioned parameters. The material that is used for the determination of the raw unit weight can still be used for further investigations (e.g.: plant tolerance, seeds capable of germinating, self-heating tests, etc.).

2 Sample preparation

Sieving the fresh original sample to < 10 mm.

3 Materials

- Scales (1 g reading)
- 1 liter measurement cylinder made of transparent plastic, graduations in increments of 10 ml
- Wide-neck powder funnel
- Dropping device (stand, stand pincers or ring, rubber base) for setting a standardized drop height of 10 cm

4 Carrying out the testing

Counterbalance the empty measurement cylinder. Fill the sample material loosely up to the rim with the help of the powder funnel. Let the measurement cylinder drop 10 times from a height of 10 cm vertically onto the rubber base with the help of the dropping device. The dropping device has to be constructed in such a way that, with a raised measurement cylinder, the lower edge of the measurement cylinder is exactly 10 cm over the rubber base. Read the settled volume of the sample material, accurate to 10 ml, and exactly determine the mass of the filled measurement cylinder accurate to 1 g. The determination is to be carried out with threefold repetition with loosely filled sample material each time.

5 Calculation of the results

The mean value of the three repetitions is to be stated, accurate to 10 g, in the dimension [g/l]. A more exact evaluation can not be carried out on account of the insufficient reading accuracy of the volume.

$$RD_{FS} = [M_{FS} / Vol_{FS}] \cong 10^3 \text{ [g/l]}$$

RD_{FS} : Raw unit weight of the fresh substance in [g/l]

M_{FS} : Mass of the filled fresh substance in [g]

Vol_{FS} : Volume of the settled fresh substance in [ml]

6 Remarks

For any calculation of the raw unit weight of the dry substance (RD_{DS}), one is to see to it that the water content to be used in the calculation has to be determined on a sample of the same sieving (< 10 mm).

If the raw unit weight is to be determined from unscreened large-grain compost (0 - 20 or 0 - 30 mm), a 2 liter measurement cylinder is to be used for this. The described method is not appropriate for materials with an even larger grain (e.g. raw compost materials).

The piled weight of raw compost materials has to be determined by weighing a certain volume, for example with a 10 l bucket. The suitable vessel size is, in isolated cases, dependent on the nature of the raw compost material.

7 Literature

VDLUFA, Methods Book, Volume I (1991): The investigation of soils

Determination of the degree of rotting in a self-heating test

1 Preliminary remark

The self-heating capability of the fresh compost substance in the Dewar vessel makes a statement possible on the degree of rotting. The self-heating test has to be carried out with an optimum and standardized water content of the samples. Too dry or too moist sample material leads to an underestimate of the self-heating capability and consequently to an overestimate of the degree of rotting. A standardized, optimum water content, adjusted to the water capacity of the respective compost, can be set using the so-called 'fist test'.

2 Sample preparation

Sieving the fresh original sample to ≤ 10 mm.

Adjustment of the water content using a 'fist test'.

3 Materials

- Dewar vessel (volume 1.5 liters, inside diameter 100 mm)
- Straight enclosed-scale thermometer with a maximum display or continuously writing temperature measurement device
- Drying chamber and suitable vessels for the determination of the water content (see also [chapter II.1](#))
- Scales (0.1 g reading)

Kommentar [jb1]: prüfen ob das stimmt

4 Carrying out the testing

The self-heating test has to be made as soon as possible after taking the sample. If a delay of the start of testing cannot be avoided for organizational reasons, it is necessary after the cool storage to bring the temperature of the cooled sample material onto room temperature at the beginning of the test.

The testing substrate is to be adjusted to a moisture content that is optimum for microbial events, depending on the material, before the start of the test. In the case of composts with a low content of organic substances, the favorable water content is lower than in the case of composts with a high content of organic substances.

4.1 Fist test

The optimum water content is adjusted using a '*fist test*'. A compost sample is pressed in the fist. If water beads escape between the fingers, the sample is too wet. If the sample crumbles without further action when the fist is opened, the sample is too dry. There is a suitable moisture content if the pressed sample crumbles with light pressure; if, on the contrary, it is only deformed, it is too wet. This suitable moisture is also appropriately characterized by the comparison "moist like a well-squeezed sponge". It corresponds to the stage "slightly humid " of the [method 12.3, chapter IV](#). During the moistening of the sample material that is too dry, the water has to be evenly mixed in the compost, sample material that is too moist has to be carefully dried.

After the adjustment of an optimum moisture content, the Dewar vessels are filled up to the rim with compost in a loose pouring, lightly shaking the test containers on a base, and the sensor of the temperature measurement device is placed in the lower third of the vessel. The vessels are set up openly at room temperature (approx. 20°C). As a rule the temperature maximum is reached after 2 to 5 days.

The test ends after temperatures exceed the maximum and are clearly falling, at the latest, after 10 days.

If continuous measurement writers or devices with digital measurement value recording (measurement interval 4 hours) are not available, at least 2 measurements with a time difference of at least 8 hours are to be carried out within 24 hours.

5 Calculation and evaluation of the results

The assignment of the rotting degree takes place on the basis of the temperature maximum (T_{\max}).

Table II-1: Assignment of the rotting degree on the basis of the temperature maximum

Rotting degree	T_{\max} [° C]	Product name
I	> 60	Compost raw material
II	50.1 - 60.0	Fresh compost
III	40.1 - 50.0	Fresh compost
IV	30.1 - 40.0	Mature compost
V	= 30	Mature compost

6 Statistics

The measurement fluctuations at the adjustment of the temperature maximum for repeated measurements are indicated by BECKER (1997) and JOURDAN (1988) with < 4 %. The measurement inaccuracy for fresh composts increases with a decreasing rotting degree.

Frequent range of values: 20 - 75 ° C

Repeatability limit: 11.2 %

7 Remarks

The water content of the compost adjusted by using the 'fist test' is to be determined. This is possible without much expenditure, as the water content is determined anyway (compare chapter II.1). If the fist test proves that water has to be added, the water amount and the used compost weight (ml or g) is recorded and added to the water content of the fresh substance. Is the compost to be tested too wet the weight difference before and after drying is to be recorded and subtracted from the actual water content of the sample.

8 Literature

BECKER;G., 1997

Measurement exactitude of the determination of the rotting degree, abstract from thesis, Münster

JOURDAN, B., 1988:

For determination of the rotting degree from composts from waste and waste sludges, Stuttgarter Berichte zur Abfallwirtschaft 30, ESV Erich-Schmidt-Verlag, Berlin

Determination and evaluation of the plant compatibility of compost in the germinating plant test with spring barley

1 Preliminary remark

The testing of the plant compatibility of compost takes place in a germinating plant test with spring barley. As a mixing component and as reference substrate serves the Standard Soil 0 (Einheitserde EE0) from the German Standardized Soil Association. The start of the plant test has to take place directly after the arrival of the fresh compost sample. If several days lie between the arrival of the sample and the start of the test, the statements on the test are no longer in accordance with the necessary certainty.

2 Sample preparation

Sieving the fresh original sample to ≤ 10 mm.

3 Materials

- Plastic mixing tub
- Plastic measurement bowls (2 l, 1 l, 0.5 l)
- Plastic pots 500 ml with bottom perforation and basins underneath
- Water-soluble multi-nutrient fertilizer
- Certified seeds from spring barley, germinating capability $> 90\%$
- Laboratory scales (10 mg reading)
- Standard Soil 0 (EE0) from the Standardized Soil Association (pH value 5.5 - 6.5) as a reference substrate and mixing component (reference address, see chapter II.5.8).

4 Carrying out the testing

The test substrate (compost) and reference substrate (EE0) are screened through a 10 mm sieve; the sieve overflow is thrown out. During the screening process larger compost aggregates are rubbed under light pressure so that they break apart.

Test mixtures with 25% vol. and 50% vol. test substrate proportion are produced from the test substrate and EE0. For the variation with 25% compost proportion, 0.5 l compost are mixed with 1.5 l EE0, for the variation with 50% compost proportion, 1 l compost is mixed with 1 l EE0.

After this, 400 ml each of the test mixture (3-fold striking of the measurement bowl) are filled into the corresponding vessel. Because a three-fold test start takes place for security, 3 vessels are filled (repetitions) per test mixture (variations). The contents of all pots are lightly compressed during the filling because of the 3-fold striking of the vessel. 400 ml each of reference substrate (EE0 unmixed) are also filled in 3 test vessels.

If all 9 test vessels are filled, 100 ml of a liquid multi-nutrient fertilizer is evenly poured on them. The concentration is calculated in such a way that 110 mg nitrogen make it into each vessel (corresponds to 220 mg N/l substrate, incl. cover).

50 seeds of spring barley per vessel are evenly distributed on the substrate surface. They can, as a choice, be counted out or, after a repeated determination of weight of 50 grains, weighed out (accurate to 10 mg). It is to be guaranteed by visual inspection that no broken or stunted grains make it into the seed.

After the sowing, a covering takes place with 100 ml of the respective test mixture or with EE0 in the case of the reference substrate.

The remaining test substrate is thrown out. The well-pressed covering layer is subsequently evenly moistened with approx. 60 ml of water (pipeline water with drinking water quality). A wetting of the covering layer has definitely to be avoided, though, because otherwise the germination of the spring barley can be impeded. The test substrate in the plant test should not be moister than the fresh compost substance during the determination of the degree of rotting (fist test).

Setting up the vessels takes place in an air-conditioned room at 18-20°C and a luminous strength of at least 3,000 lux at least 12 h/day. Fluorescent tubes connected in parallel suggest themselves for the lighting.

Desalinized water has to be poured on the vessel 2 - 3 times, as a rule, in the test period of 10 - 12 days. The pouring takes place according to the needs of the plants, strong growth uses more water than weak growth. The water requirement can be estimated by experienced personnel using a 'finger sample'; otherwise, the water used in the individual vessels can be replaced corresponding to their weight loss (weigh vessels incl. the basins underneath at the start of the test and each time during watering). The substrate is to constantly be kept well moistened, without leakage water coming out. If leakage water nevertheless comes out into the basin underneath, this is to be put back.

The cutting of the test plants takes place when the majority of the second leaves have overgrown the first ones in the checkpoint (comparison substrate). The stalks are cut off pot-wise with scissors directly over the substrate surface and immediately weighed (in [g], accurate to two decimal places).

5 Calculation and evaluation of the results

The evaluation of the plant compatibility of a test substrate takes place on the basis of the mean value of the above-ground fresh mass yields of the 3 repetitions with a 25% or, as the case may be, with a 50% compost proportion. The yields are expressed percent-wise in relation to the mean yield of the reference substrate.

$$FM(r)_{25/50\%} = [FM_{25/50\%} / FM_{EE0}] \cong 100 [\%]$$

$FM(r)_{25/50\%}$: Relative yield of the variations with respect to the reference substrate EE0 in [%]

$FM_{25/50\%}$: Mean fresh mass yields of the variations in [g]

FM_{EE0} : Mean fresh mass yield of the reference substrate in [g]

Compost is considered to be plant tolerant if no visible chloroses or necroses appear on the leaves and the fresh mass yield of the variation with 25% compost proportion reaches at least 90% of the yield of the comparison substrate. If these requirements are fulfilled, compost can be recommended for use as a soil improver and fertilizer.

If the variation with the 50% compost proportion also reaches at least 90% of the fresh mass yield of the reference substrate, the tested compost can furthermore also be recommended in principle for use as a mixing component in garden moulds and cultivated substrates. The mixing proportions to be recommended depend, in the process, above all on the salt content, the content of soluble plant nutrients (especially potassium) and the pH value of the compost, as well as the intended plant cultivation.

6 Statistics

Frequent value range:	95 - 142 % for test mixture with 25 vol.-% compost
	74 - 135 % for test mixture with 50 vol.-% compost
Repeatability limit:	22.4 %

7 Remarks

During the execution of the test, one has to see to it that the material is not moistened too heavily. No leakage water should come out, especially after the addition of the fertilizer solution, the subsequent moistening of the covering layer with water and during watering.

8 Reference addresses for Standard Soil (EE0)

Gebr. Patzer KG Einheitserde- und Humuswerke, 36391 Sinntal-Jossa;
Tel.: +49 (0) 06665 / 8057; Fax: +49 (0) 6665 / 604

Balster Einheitserdenwerk GmbH, 58730 Fröndenberg;
Tel.: +49 (0) 2373 / 7398; Fax: +49 (0) 2373 / 71919

Einheitserdenwerk Uetersen, Werner Tantau GmbH & Co. KG, 25436 Uetersen;
Tel.: +49 (0) 4122 / 53308; Fax: +49 (0) 4122 / 53365

Einheitserdenwerk A. Stangenberg GmbH, 31789 Hameln;
Tel.: +49 (0) 5151 / 7631; Fax: +49 (0) 5151 / 22794

9 Literature

KEHRES, B. 1990: Quality of Compost from different original material. Thesis University of Kassel, Faculty Agriculture, Witzenhausen. To be ordered at: Bundesgütegemeinschaft Kompost e.V., www.kompost.de.

Determination of the pH value

1 Preliminary remark

The pH value is determined electrometrically in a suspension of the fresh compost substance in 0.01 molar CaCl₂ solution (ratio: 1 + 10).

2 Sample preparation

Sieving the fresh original sample to ≤ 10 mm.

3 Materials and reagents

- Scales (0.1 g reading)
- Suitable vessels (e.g. beakers), glass rod
- Thermometer
- Measuring device: pH meter with a suitable pH electrode
- 0.01 molar CaCl₂ solution
- Suitable pH buffer solutions for the calibration of the measuring device

4 Carrying out the testing

200 ml of 0.01 molar CaCl₂ solution are added to 20 g of sample material (FS ≤ 10 mm). The pH value in the suspension (stir repeatedly with a glass rod) is determined after 1 hour with a calibrated pH measuring device. The temperature of the suspension is to be taken into account during the measurement in accordance with the type of measuring device.

5 Calculation of the results

The results of the pH determination are to be stated accurate to one decimal point.

6 Remarks

A close ratio of fresh compost substance and CaCl_2 solution (e.g. 1 + 2.5) would ensure an approximation of the situation in the 'soil solution'. A ratio of 1 + 10 is chosen for the determination of the pH value on account of the slight change of the pH value in the case of a greater 'dilution' and of the simpler execution of the analysis.

7 Literature

VDLUFA, Methods Book, Volume I (1991): The investigation of soils

Determination of the salt content after extraction with distilled water

1 Preliminary remark

The determination of the salt content occurs after the extraction of the fresh compost substance with **distilled water** (ratio 1+10). The extraction with saturated gypsum solution still called for in the first edition of the methods book is connected with difficulties in the method and is replaced by the simpler extraction with distilled water (also see Point 6 Remarks).

2 Sample preparation

Sieving the fresh original sample to < 10 mm.

3 Materials and reagents

- Scales (0.1 g reading)
- PE shaking bottles (min. 250 ml)
- Suitable filtering vessels (e.g. beakers)
- Medium-porous folded filter and suitable funnels
- Conductivity measuring device with cell constant $C = 1$
- Thermometer (min. 0.5°C reading)
- Shaker
- Distilled water (conductivity < $1.0 \cdot 10^{-6}$ S/cm)

4 Carrying out the testing

Shake 20 g of sample material ($FS \leq 10$ mm) with 200 ml distilled water in the PE bottles for 2 hours. Following this, filter and determine the conductivity and temperature in the filtrate (in so far as a conductivity measuring device with an automatic temperature compensation is not used).

5 Calculation of the results

The corresponding calcium chloride concentration is calculated from the conductivity in the extract. The basis for the calculation is the specific conductivity of a 0.01 molar calcium chloride solution (745.6 mg KCl/l H_2O) of $14.12 \cong 10^{-4}$ S/cm at 25°C. The dependence of the electrical conductivity of a salt solution on the temperature has to be taken into consideration through a corresponding factor F_t (if a conductivity measuring device with an automatic temperature compensation is not used). The conversion factors F_t are published in VDLUFA, Methods Book, Volume I (1991): The investigation of soils (Method A 10.1.1). If the determination of the conductivity is carried out at a constant temperature of 25°C or using a measuring device with automatic temperature compensation (observe manufacturer details), the conversion of the conductivity into the corresponding salt concentration takes place with the constant factor $F_t = 52.80$.

The salt content relates to the volume of the fresh compost substance. The statement takes place in [g KCl/l FS], accurate to two decimal points.

$$SA_m = LF_p \cong F_t \text{ [mg/100 g]}$$

SA_m : Salt content with regard to the fresh mass in [mg KCl/100 g FS] (with an extraction ratio of 1 + 10, the value corresponds to the KCl concentration of the solution in [mg/l])

LF_p : Conductivity of the sample extract in [10^{-4} S/cm]

F_t : Factor for the calculation of the salt concentration from the conductivity, taking the temperature into consideration

$$\text{At } 25^\circ\text{C} : F = 745.6 \text{ [mg/l]} / 14.12 \text{ [} 10^{-4} \text{ S/cm]}$$

$$= \mathbf{52.80 \text{ [mg/l]} / [} 10^{-4} \text{ S/cm]}$$

Conversion of the volume of the fresh substance

$$SA_{vol} = SA_m \cong RD_{FS} \cong 10^{-5} \text{ [g/l]}$$

SA_{vol} : Salt content with regard to the volume of the fresh substance in [g KCl/l FS]

RD_{FS} : Raw unit weight of the fresh substance (< 10 mm) in [g/l]

During the conversion of the salt content from [mg KCl/100 g FS] to [g KCl/l FS], all dimensionally dependent changes in the factor 10^{-5} are integrated, so only the numerical values of SA_m and RD_{FS} have to be used in the formula.

6 Remarks

The influence of weak electrolytes is to be made impossible during the determination of the salt content through the extraction with saturated gypsum solution (1st edition of the methods book). Only the strong electrolytes with osmotic relevance should specifically be recorded, therefore, for the evaluation of the plant tolerance of the material. This procedure has proven itself for many garden soils for the evaluation of the conductivity (of the salt content) for the plant tolerance. A simple transfer to composts and compost substrates is not practical, however; the portion actually relevant for the plant tolerance can, for example, be overestimated for strong electrolytes (Na^+ and K^+ ions) through the strengthened exchange of cations with the use of saturated gypsum solution.

In addition, difficulties with the method can arise with the use of saturated gypsum solution as the extraction agent that can lead to an erroneous determination of the conductivity stemming from the compost sample.

The salt content derived from the conductivity measurement with the use of distilled water as the extraction agent can also only be a first, crude (and above all simple to determine) parameter for the evaluation of the plant tolerance of the examined compost material. The direct determination of the ions Na^+ , K^+ and Cl^- with osmotic effectiveness in the extract is substantially more convincing. The determination of the ions mentioned can be carried out without a problem when the equipment is available (flame photometer, chloride meter). Rules for the determination in the watery extract can be taken from the methods book, volume I of the VDLUFA.

7 Literature

VDLUFA, Methods Book, Volume I (1991): The investigation of soils

Determination of nitrate, ammonia and magnesium in the CaCl₂ extract

1 Preliminary remark

The soluble content of nitrate, ammonia and magnesium is determined after extraction of the fresh compost substance with 0.0125 molar CaCl₂ solution (ratio 1 + 10). In the extract, nitrate and ammonia are measured with a spectrophotometer (or with a comparable method), and magnesium using AAS. The nitrogen fractions determined in this way are designated as **soluble mineral nitrogen**. The **soluble organic nitrogen compounds** likewise obtained in the extract are not recorded with the described methods. No statement on the total nitrogen available to the plants can be made by the sole determination of nitrogen and ammonia either. As a crude rule of thumb, approx. 10% of the total nitrogen content determined according to Method 13.1 can be assessed for the nitrogen available to plants in a vegetation period.

2 Sample preparation

Sieving the fresh original sample to < 10 mm.

3 Materials and reagents

- Scales (0.1 g reading)
- PE shaking bottles (min. 250 ml)
- Suitable filtering vessels (e.g. conical flasks)
- Medium-porous folded filter and suitable funnels
- Shaker
- Measuring devices (spectrophotometer, AAS)
- 0.0125 molar CaCl₂ solution
- Standard solutions of the nutrients to be determined

4 Carrying out the testing

Shake 20 g of sample material (FS < 10 mm) with 200 ml of 0.0125 molar CaCl₂ solution in the PE bottles for 2 hours. Following this, filter and determine the nitrate and ammonia in the filtrate with a spectrophotometer and determine magnesium using AAS. The determination of the individual nutrients in the extract can also take place through comparable methods (e.g. titration for the determination of the ammonia content).

5 Calculation of the results

The soluble nutrient content is related to the volume of the fresh compost substance. The statements take place as Nitrate-N, Ammonia-N and Mg as whole numbers in [mg/l FS].

$$NS_{\text{vol}} = NS_{\text{m}} \cong RD_{\text{FS}} \cong 10^{-2} [\text{mg/l FS}]$$

NS_{vol} : Nutrient content with regard to the volume of the fresh substance in [mg/l FS]

NS_m : Nutrient content with regard to the fresh mass in [mg/100 g FS] (with an extraction ratio of 1 + 10, the value corresponds to the nutrient concentration of the solution in [mg/l])

RD_{FS} : Raw unit weight of the fresh substance (< 10 mm) in [g/l]

6 Remarks

The nutrients in the extract are determined in accordance with the methods mentioned in the methods book VDLUFA, Volume I (1991), A 6.1.4.1 and A 6.2.4.1 or using comparable methods.

Possibly arising dilution steps or the mixing of an aliquot of the extract with reagents are to be correspondingly taken into consideration in the determination of NS_m.

The self-coloring of the extracts can lead to substantial problems during the determination of the nitrate, especially with low nitrate content. That is why an examination of the nitrate determination with the spectrophotometer using nitrate indicator rods is to be recommended. As a rule, these quick nitrate determinations alone already supply sufficiently accurate results for most compost applications. Strongly colored extraction solutions can be compensated by suitable decolorizing agents (e.g. activated charcoal), by blind measurements of the extracts or by alternative nitrate determination methods (more on this in the methods book of VDLUFA, Volume I (1991), Method A 6.1.4.1, Point 8 Remarks).

Besides the purely analytical problems mentioned for the determination of the soluble mineral nitrogen fractions, the **sample preparation and conservation** or, as the case may be, the **rapid execution of the analyses** have an outstanding significance! The soluble mineral nitrogen contents are subject to a strongly temperature-dependent dynamic. That is why the samples have to be examined directly after the taking of the sample for their soluble nitrogen contents. If delays that are not avoidable arise during the analysis, the samples have to be conserved by freezing. In connection with this, it is pointed out that the determination of the soluble mineral nitrogen contents can characterize compost charge ready for sale only at a certain point in time (that of the taking of the sample); the dynamic addressed takes its course further, of course, during the storage of the compost.

7 Literature

VDLUFA, Methods Book, Volume I (1991): The investigation of soils
DIN 38 406 Section 5 (1988)

Determination of phosphorus and potassium in the CAL extract

1 Preliminary remark

The soluble content of phosphorus and potassium is determined after extraction of the fresh compost substance with a solution of calcium acetate, calcium lactate and acetic acid buffered to pH 4.1 in the ratio 1 + 10. In the extract, phosphorus is measured with a spectrophotometer and potassium with flame photometry.

2 Sample preparation

Sieving the fresh original sample to < 10 mm.

3 Materials and reagents

- Scales (0.1 g reading)
- PE shaking bottles (min. 250 ml)
- Suitable filtering vessels (e.g. conical flasks)
- Medium-porous folded filter and suitable funnels
- Shaker
- Measuring devices (spectrophotometer, AAS)
- CAL solution (statements on the production in VDLUFA, Methods Book, Volume I (1991), Method A 6.2.1.1)
- Standard solutions of the nutrients to be determined
- Color reagents for the determination of phosphates (statements on the production in VDLUFA, Methods Book, Volume I (1991), Method A 6.2.1.1)

4 Carrying out the testing

Shake 20 g of sample material (FS < 10 mm) with 200 ml CAL solution in the PE bottles for 2 hours. Following this, filter and determine the phosphorus in the filtrate with a spectrophotometer and determine potassium with flame photometry.

5 Calculation of the results

The soluble nutrient content is related to the volume of the fresh compost substance. The statements take place as **oxide compounds (P₂O₅, K₂O)** as whole numbers in [mg/l FS].

$$NS_{\text{vol}} = NS_{\text{m}} \cong RD_{\text{FS}} \cong 10^{-2} \text{ [mg/l]}$$

NS_{vol} : Nutrient content with regard to the volume of the fresh substance in [mg/l FS]

NS_{m} : Nutrient content with regard to the fresh mass in [mg/100 g FS] (with an extraction ratio of 1 + 10, the value corresponds to the nutrient concentration of the solution in [mg/l])

RD_{FS} : Raw unit weight of the fresh substance (< 10 mm) in [g/l]

6 Remarks

The nutrients in the extract are determined in accordance with the methods mentioned in the methods book VDLUFA, Volume I (1991), A 6.2.1.1 or using comparable methods.

Possibly arising dilution steps or the mixing of an aliquot of the extract with color reagents for the determination of the phosphate are to be correspondingly taken into consideration in the determination of NS_m .

7 Literature

VDLUFA, Methods Book, Volume I (1991): The investigation of soils

Determination and evaluation of the content of seeds capable of germinating and plant parts capable of sprouting

1 Preliminary remark

The cultivation method is carried out for the determination of the content of seeds capable of germinating and plant parts capable of sprouting in the compost.

2 Sample preparation

Sieving the fresh original sample to ≤ 10 mm.

3 Materials and reagents

- Plastic basin with bottom perforation or equivalent testing containers
- Pouring mats
- Pinhole foil
- Conductivity measuring device and further materials (see [chapter II.7](#)) for the determination of the salt content
- Suitable mixing substrate (e.g. weakly decomposed high moor peat with approx. 4 g calcium carbonate per litre)

4 Carrying out the testing

The sieved test substrate (FS ≤ 10 mm) is exposed to a temperature of +4°C for 3 days. Following this, 3 liters of the test substrate are diluted with a mixing component in such a way that the test mixture has a salt content of < 2 g KCl/l FS.

The required dilution is to be determined by calculation from the salt content of the fresh substance and the mixing component is to be assessed a salt content of 0 g KCl/l FS here! The mixing component used has to be free from seeds capable of germinating and plant parts capable of sprouting. Weakly decomposed high moor peat with approx. 4 g of calcium carbonate per liter is suitable.

The test mixture is evenly distributed in a layer of approx. 10 mm in test basins with bottom perforation, lightly pressed down and watered to the full water capacity. First is a pouring mat laid out for the test containers and covered with a pinhole foil for protection against contamination.

After this, the test containers are set up over a period of 15 days with an illumination intensity of at least 1000 lux and a room temperature of 18 - 20°C without direct sunshine and the water loss is regularly compensated by spraying. In order to prevent desiccation to a great extent, the basins can be covered with glass or plastic plates (leaving an air gap open).

5 Calculation and evaluation of the results

After the end of the test, the germinating plants that have accumulated are counted and calculated per liter of test substrate, (not the test mixture from test substrate and mixing components!!) and exactly given up to two decimal places. If necessary, the species composition is determined and stated.

Compost is **free** of seeds capable of germinating and plant parts capable of sprouting if **fewer than 0.5 germinating plants** are determined per liter of test substrate.

Compost is **largely free** of seeds capable of germinating and plant parts capable of sprouting if **fewer than 2 germinating plants** are determined per liter of test substrate.

There is a **substantial content** of seeds capable of germinating and plant parts capable of sprouting if **more than 2 germinating plants** are determined per liter of test substrate.

A distinct content of germinating plants and sprouting plant parts is measured, if more than 2 germinating plants are determined per liter test substrate

Frequent range of values: 0 - 0.3 [1/l]

Repeatability limit: 50 %

6 Remarks

One proceeds according to chapter II.7 for the determination of the salt content of the fresh compost substance. The required salt content in the test mixture of < 2 g KCl/l FS is to be determined by way of calculation and not by measuring the conductivity of the test mixture, because the conductivity measurement would supply higher and higher values with the use of lime-white high moor peat as a mixing component. A salt content of 0 g KCl/l FS is to be assessed for the mixing component for the calculation of the required dilution of the fresh substance.

Compost has to be **largely free** of seeds capable of germinating and plant parts capable of sprouting for the **use as a mulch, fertilizer and soil conditioner**.

Compost has to be **free** of seeds capable of germinating and plant parts capable of sprouting for the use as a blending component for cultivation substrates.

7 Literature:

KEHRES, B.. 1990: .Quality of Compost from different original material. Thesis University Kassel, Faculty Agriculture, Witzenhausen. To be ordered at www.kompost.de.

Determination and evaluation of the foreign matter and stone content

1 Preliminary remark

Foreign matter is material that is undesired in the composting. This is in principle all inorganic material, such as glass, plastic, metals, rubber, composite materials etc. Paper, stones, lava and clay granulates are no foreign matter.

Material produced from biologically degradable polymers BAW (bioplastics) are called foreign matter, as far as they can be selected as optically visible particles in the frame of the foreign matter selection of para 4 (see below).

Foreign matter is undesirable content material, because it impairs the optical appearance of compost and can be disturbing when compost is used. The degree of impurity is dependent on the optical conspicuousness of the foreign matter contained in the compost. Smaller foreign matter particles are less noticeable than larger foreign matter particles. Only foreign matter with a particle size > 2 mm is relevant for the optically-effective degree of impurity of compost.

Stones are likewise undesirable content material, but not foreign matter. The evaluation of the content of stones in compost is effected separately.. Only stones with a particle size > 5 mm are relevant for the evaluation.

2 Sample preparation

Drying the unscreened fresh substance at 105°C.

3 Materials

- Scales (10 mg reading)
- Sieve with 5 mm mesh width
- Sieve with 2 mm mesh width
- Tweezers.

4 Test performance

For fine-grained compost (0 to 10 mm), 1 liter of dried (105°C) test substrate is required for the examinations, for medium-grained compost (0 to 20 mm), 2 liters, and for coarse-grained compost, 3 liters.

The dry sample is weighed and subsequently grated by hand through a 5 mm sieve and the sieve through fraction through a 2 mm sieve. The sieve through fraction < 2 mm is thrown out. From the sieve fraction > 5 mm, stones are picked out and weighed. Following this, the sieve fractions 2 - 5 mm and > 5 mm are joined. This united fraction is spread out on a flat base and the foreign matter is picked out with tweezers. During the selection, portions of not more than 100 ml are to be examined each time, in the process of which the selection of the foreign matter has to take place as thoroughly as possible. The mass of the foreign matter > 2 mm is weighed and separately recorded.

5 Calculation and evaluation of the results

Foreign matter and stone content relate to the total mass of the dried sample each time (before the sieving) and to be stated in [% DS], accurate to two decimal places.

$$F_s = [M_{F_s > 2\text{mm}} / P_{\text{tot}}] \cong 100 [\%]$$

$$S_t = [M_{S_t > 5\text{mm}} / P_{\text{tot}}] \cong 100 [\%]$$

F_s : Foreign matter content in [% DS]

$M_{F_s > 2\text{mm}}$: Foreign matter > 2 mm in [g]

S_t : Stone content in [% DS]

$M_{S_t > 5\text{mm}}$: Stones > 5 mm in [g]

P_{tot} : Total mass of the examined sample before the sieving in [g]

10.5.1 Evaluation of the foreign matter content:

- Compost is **free of foreign matter** if the content is **less than 0.1** [% DS].
- Compost is **practically free of foreign matter** if the content is **less than 0.5** [% DS].
- Compost has a **noticeable content of foreign matter** if this is **more than 0.5** [% DS].
- Compost has a **substantial content of foreign matter** if the content is **more than 2** [% DS].

As far as "bioplastics" i.e. materials from "biologically degradable polymers" are admitted or used as original material for composting or admitted or used for the production of compost both the foreign matter content and the degree of impurities must be determined according to **chapter IV 12.2**. A distinct content of foreign matter is existent if the top view of the area sum of the selected foreign matter is larger than $800 \text{ mm}^2 / 1$ of the dried sample.

10.5.2 Evaluation of the stone content

- Compost has a **low content** of stones if this is **less than 5** [% DS].
- Compost has a **noticeable content** of stones if this is **more than 5** [% DS].

10.6 Statistics

Frequent range of values:	0 - 0.4 for foreign matter 0.5 - 4 % for stones
Repeatability limit:	80 % for foreign matter 6 % for stones

10.7 Remarks

The gravimetric content of foreign matter is a scale for the evaluation of the degree of impurities only in those cases where a structure of specific heavy foreign matter (e.g. glass) and specific light matter (e.g. plastics), like common in biowaste composting, can be assumed.

The evaluation of the degree of impurities in the sense of the quality and test regulations, regarding an increased portion of specifically light foreign matter, can only be carried out if not only the gravimetric content of foreign matter (chapter II.10) is determined and evaluated quantitatively but also the optically visible degree of impurities (chapter IV.12.2).

10.8 Literature

KEHRES, B. 1990:

Quality of compost from different original material. Thesis University of Kassel, Faculty Agriculture, Witzenhausen. To be ordered: Bundesgütegemeinschaft Kompost e.V. at www.kompost.de.

Determination of the loss on ignition

1 Preliminary remark

The loss on ignition supplies a measure for the content of organic substances in composts. It is to be observed that inorganic compounds and water of constitution can also evaporate through this determination method and that they are added to the content of organic substances in the evaluation. During the examination of composts, this possible error is negligibly small as a rule, however, so the loss on ignition represents a practical and sufficiently accurate measure for the content of organic substance, especially on account of the simple execution of the method.

2 Sample preparation

Drying the unscreened fresh substance at 105°C.

Grinding of at least 30 g DS with a suitable grinder to < 0.25 mm.

3 Materials

- Analytical scales (1 mg reading)
- Box-type furnace
- Porcelain crucible
- Dessicator

4 Carrying out the testing

Weigh in approx. 5 g of the dried and ground sample material into a dry, weighed porcelain crucible (make red hot and keep in the dessicator) and burn up at 550°C in the box-type furnace until the weight is constant. Let the porcelain crucible cool down in the dessicator and weigh back in. The weighings are to be determined accurate to 1 mg.

5 Calculation of the results

The loss on ignition relates to the dry mass. The results are stated in [% DS], accurate to one decimal place.

$$GV = [(M_{vdG} - M_{ndG}) / (M_{vdG} - M_{tara})] \cong 100 \text{ [%]}$$

GV : Loss on ignition in [% DS]

M_{tara} : Mass of the empty crucible in [g]

M_{vdG} : Sample weight + M_{tara} in [g]; before the burning

M_{ndG} : Sample + M_{tara} in [g]; after the burning

6 Remarks

A generally valid time span up to point where constancy of weight is reached can not be given; in case of doubt, a sample has to be burned still further after an initial reweighing. Ventilation with approx. 2 l air/minute has proven itself for the removal of faulty oxidation.

The organic carbon content can be calculated by multiplication with the factor 0.58 from the loss on ignition as a measure of the organic substance in composts. The C/N relationship then results from the division of the carbon content calculated in this way by the total nitrogen content determined according to Method 13.1.

7 Literature

DIN 38 414 (S2), German uniform proc. for water, waste water and slurry investigation; slurry and sediments: Determination of the loss on ignition and of the burning residue

Schaller, K. (1988): Practical training for soil science and plant nutrition; Geisenheimer reports, Volume II

Determination of heavy metals in the aqua regia treatment

1 Preliminary remark

The dry compost substance is treated with aqua regia for the determination of the total content of the heavy metals lead, cadmium, chromium, copper, nickel, mercury and zinc. The elements mentioned are determined in the treatment solution with AAS or an equivalent procedure.

2 Sample preparation

Drying the unscreened fresh substance at 105°C.

Grinding of at least 30 g DS with a suitable grinder to < 0.25 mm. During the grinding procedure, no contamination of the sample may take place with the heavy metals to be determined.

3 Materials and reagents

- Scales (1 mg reading)
- Reaction vessels made of glass with suitable reflux condensers and heating devices
- 100 ml volumetric flask
- Fine to medium-porous folded filters (acid-proof, free of trace elements) and suitable glass funnels
- Sealable filtering vessel (100 ml volumetric capacity)
- Measuring device: AAS (ICP)
- Hydrochloric acid_{conc} ($\rho = 1.19$ g/ml, degree of purity: at least to a.)
- Nitric acid_{conc} ($\rho = 1.40$ g/ml, degree of purity: at least to a.)
- Nitric acid_{dil} ($c = 2$ mol/l, degree of purity: at least to a.)
- Heavy metal standard solutions

4 Carrying out the testing

Approx. 5 g of the sample material is weighed into the reaction vessel (note the weigh-in in [g], accurate to 3 decimal points), subsequently 18 ml of hydrochloric acid_{conc} and 6 ml of nitric acid_{conc} are carefully added (aqua regia: 3 parts hydrochloric acid_{conc} + 1 part nitric acid_{conc}). Let the reaction vessel stand at least 12 hours (over night) at room temperature. After that, render the solution soluble for 2 hours with light boiling under reflux condensation. After cooling, partially rinse the condenser with nitric acid_{dil} (max. 25 ml) and subsequently transform the contents of the reaction vessel with nitric acid_{dil} into a 100 ml volumetric flask (fill up to the marking with acid). Filter the contents of the volumetric flask after thorough mixing through a suitable filter into various filtering vessels.

The determination of the individual heavy metals in the treatment solution takes place using AAS (ICP) in [mg/l], applying the corresponding standard calibration series. It is to be ensured that the standard calibration solutions contain the same proportion of acid as the measurement solution.

5 Calculation of the results

The heavy metal contents relate to the dry mass, the statement takes place in [mg/kg DS], in the process of which contents < 10 mg/kg are to be given to two decimal points, contents between 10 - 100 mg/kg DS to one decimal point and contents > 100 mg/kg DS are to be given as whole numbers.

$$SM = SM_{al} \cong F_{dil} \cong V_{mk} / E \text{ [mg/kg]}$$

SM : Heavy metal content in [mg/kg DS]

SM_{al} : Heavy metal concentration in the treatment solution in [mg/l]

F_{dil}: Possibly necessary dilution factor, in so far as the standard measurement range was exceeded with individual elements and the treatment solution had to be diluted

V_{mk} : Volume of the treatment solution (volumetric flask) in [ml]

E : Weigh-in in [g]

6 Remarks

After the treatment of the dry compost substance with aqua regia, the total nutrient content of phosphorus, potassium, calcium and magnesium can also be determined in the treatment solution, in addition to the heavy metals mentioned. Phosphorus is determined using ICP (or with a spectrophotometer), the other elements using AAS.

If a different procedure than AAS is used for the determination of individual elements in the treatment solution, this is to be explicitly stated together with the respective analytical result!

A scaling of the heavy metal contents to 30% of loss on ignition (organic substance) takes place according to the formula:

$$SM_{30\%} = SM_p \cong 70 / (100 - GV_p)$$

SM_{30%} : Heavy metal content in [mg/kg DS], scaled to 30% of loss on ignition

SM_p : Heavy metal content in [mg/kg DS] in the sample

GV_p : loss on ignition in [% DS] of the sample

7 Literature

VDLUFA, Methods Book, Volume I (1991): The investigation of soils

DIN 38 414, Section 7, German uniform proc. for water, waste water and slurry investigation; slurry and sediments: Treatment with aqua regia for the subsequent determination of the acid-soluble proportion of metals

DIN 38 406, Section 12 (1988)

DIN 38 406, Section 22 (1988)

Determination of the total content of nitrogen according to Kjeldahl

1 Preliminary remark

The organically-bound total nitrogen content can be recorded through the Kjeldahl treatment of the dry compost substance. The organically-bound nitrogen is transformed with concentrated sulfuric acid in the presence of salts and suitable catalysts into the ammonia form. After alkalization, distillation and collection of the stripped ammonia in a suitable acid pattern, the ammonia content can be determined with volumetric analysis.

2 Sample preparation

Drying the unscreened fresh substance at 105°C.

Grinding of at least 30 g DS with a suitable grinder to < 0.25 mm.

3 Materials and reagents

- Scales (1 mg reading)
- Kjeldahl apparatus (heatable treatment block, treatment vessel, distillation apparatus)
- Burette or titration machine
- Suitable glass vessel for acid reception (beaker or conical flask)
- Concentrated sulfuric acid ($\rho = 1.84 \text{ g/ml}$)
- Kjeldahl tablets or catalysts (selenium, titanium oxide) and potassium sulfate
- Soda lye ($w = \text{approx. } 40\%$)
- Acid receiver (boric acid, hydrochloric acid) and indicator
- Suitable acid measurement solution (e.g. hydrochloric acid $c = 0.1 \text{ mol/l}$ or 0.01 mol/l)

4 Carrying out the testing

The sample amount to be weighed in depends on the nitrogen content to be expected. For composts that contain approx. 1-2% nitrogen in the dry substance as a rule, a weigh-in of approx. 250 mg DS has proven itself. With a greater content of nitrogen, the weigh-in is to be reduced in accordance with Table 12.1.1.

Table 12.1.1: Favorable sample weigh-ins based on the total N content expected in the dry substance

N content in [%]	Sample weigh-in in [mg]
1-2	200-250
3-4	100-150
5-10	50-100

The required sample amount is weighed into a treatment vessel (note accurate to 1 mg), a Kjeldahl tablet is added and treated with 15 ml concentrated sulfuric acid. The addition of the sulfuric acid is to be calculated in such a way that a residual still remains after the treatment. Instead of the Kjeldahl tablet, selenium reaction mixture (0.5 g) and potassium sulfate (0.2 - 0.3 g / ml H₂SO₄) can also be added.

The treatment takes place at 360°C until the liquid is bright and clear (duration approx. 60 - 90 min.). When the sample has cooled down to room temperature, approx. 20 ml of distilled water are carefully mixed in.

In so far as a direct distillation is possible from the treatment vessels, soda lye is carefully added in a clear surplus (approx. 30 ml) (otherwise transfer the content of the treatment vessel quantitatively into a suitable distillation vessel beforehand). Following this, the distillation of the ammonia takes place with water steam into an acid receiver.

The ammonia amounts collected in the acid receiver are titrated with a suitable measurement solution with the help of a burette of a titration machine up to the transition of the indicator.

A reagent blind value is also to be determined for every sample series.

5 Calculation of the results

The weighed-in sample amount is to be noted accurate to 1 mg and the measurement solution used is to be noted in ml accurate to 2 decimal places. The results are to be stated in [% DS], accurate to two decimal places.

$$N = (M_{lp} - M_{lb}) \cong 100 \cong F_N / E \text{ [%]}$$

N : Nitrogen content in [% DS]

M_{lp} : Measurement solution used in [ml] for the sample treatment

M_{lb} : Measurement solution used in [ml] for the blind value

F_N : Factor (in [mg/ml]) for the calculation of the nitrogen equivalent in mg of 1 ml of measurement solution used. F_N is dependent on the concentration of the measurement solution used (1.4 for 0.1 molar or 0.14 for 0.01 molar HCl solution).

E : Sample weigh-in in [mg].

6 Remarks

Only the **organically-bound total nitrogen** can be determined with the described method, strictly speaking, because ammonia - nitrogen is stripped through the drying at 105°C and nitrates are not, or only insufficiently, recorded through the Kjeldahl treatment. **Nevertheless (in accordance with the definition), the designation total nitrogen is used for the characterization of composts.** The soluble mineral nitrogen compounds determined according to Method 8.1 are **not** calculated in with the Kjeldahl nitrogen determined here and remain disregarded in the calculation of the C/N ratio as well.

7 Literature

VDLUFA, Methods Book, Volume I (1991): The investigation of soils

Determination of P, K, Ca, Mg in the aqua regia treatment

1 Preliminary remark

The total content of phosphorus, potassium, calcium and magnesium are determined together with the heavy metals after treatment of the dry compost substance with aqua regia. Phosphorus is determined in the treatment solution using ICP (or with a spectrophotometer), the other elements are determined using AAS.

2 Sample preparation

See Method 12.

3 Materials and reagents

- Standard solutions of the elements to be determined
- Otherwise see Method 12

4 Carrying out the testing

The treatment of the sample and the determination of the individual elements in the treatment solution takes place in accordance with Method 12.

5 Calculation of the results

The total content of phosphorus, potassium, calcium and magnesium relate to the dry substance. The statement takes place each time as an **oxide compound (P₂O₅, K₂O, CaO, MgO)** in [% DS], accurate to two decimal points.

$$NS = NS_{al} \cong F_{dil} \cong V_{mk} \cong 10^{-4} / E \text{ [%]}$$

NS : Nutrient content in [% DS]

NS_{al} : Nutrient concentration in the treatment solution in [mg/l]

F_{dil} : Possibly necessary dilution factor, in so far as the standard measurement range was exceeded with individual elements and the treatment solution had to be diluted

V_{mk} : Volume of the treatment solution (volumetric flask) in [ml]

E : Weigh-in in [g]

6 Remarks

If a different procedure than AAS or ICP (phosphorus) was used for the determination of individual elements in the treatment solution, this is to be explicitly stated together with the respective analytical result!

7 Literature

VDLUFA, Methods Book, Volume I (1991): The investigation of soils

DIN 38 414, Section 7, German uniform proc. for water, waste water and slurry investigation; slurry and sediments (1983): Treatment with aqua regia for the subsequent determination of the acid-soluble proportion of metals

DIN 38 406, Section 22 (1988)

Determination of the degree of rotting through the exchange activity with the help of a respirometer

1 Preliminary remark

The exchange activity allows a statement on the content of biologically degradable materials in composts. An assessment of the degree of rotting takes place on the basis of the oxygen consumption.

2 Sample preparation

Sieving the fresh original sample to < 10 mm.

3 Materials

- Respirometer
- Scales
- Drying chamber

4 Carrying out the testing

The determination of the exchange activity takes place in the respirometer. The fresh compost sample is adjusted with the help of the 'fist test' (see Chap. II, Method 4) and allowed to stand for approx. 1 hour while being repeatedly turned. Following this, approx. 30 - 50 g of this mixture (depending on the respirometer) are weighed into the reaction vessels. The reaction vessels are placed into the respirometer into a water bath at 20°C and connected.

The registration of the oxygen used should take place in a 6 hour rhythm. The testing runs over 4 days (AT₄).

5 Calculation and evaluation of the results

A conversion of the units given in mg O₂/l is necessary. With an estimate of 166 ml of waste water required specific to the device, 1 counter unit (CU) = 0.166 mg O₂ consumption.

The result after 4 days is to be given in mg O₂ /g ODS as AT₄, taking the dry substance and the loss on ignition of the sample into consideration.

Calculation (following the determination of waste water samples):

$$AT_4 = CU \text{ after 4 d} \times 0.166 \text{ mg} / (\text{weigh-in DS in g} \times \text{BL in \%}) \times 100$$

Units : mg O₂ /g ODS

AT₄ : Exchange activity after 4 days

CU : Counter units

BL : Loss on ignition

DS : Dry substance

ODS : Organic dry substance

6 Literature

Notices of the State Working Group, Waste (LAGA), Instruction Leaflet 10 (M 10) "Quality criteria and application recommendations for compost from garbage and garbage/sewage sludge"; Erich Schmidt Verlag

Cress test

1 Preliminary remark

The chemical-physical examinations of the compost for carbon, nitrogen, self-heating and biological oxygen needs only give insights into the degree of aging (rotting). They say nothing about its plant tolerance, however. For this reason, a vegetation test is carried out (see plant tolerance with barley, Chap. II, Method 5).

The garden cress (*Lepidium sativum*) growing quickly in the sprouting-leaf stage has proven itself for a quick orientation, because the result is already available after 5 - 7 days.

2 Sample preparation

The fresh compost sample is sieved to < 10 mm.

3 Materials

- Plant basins (dia. 12 cm, height 7 cm)
- Standard soil EE0
- Multi-nutrient dung (see Chap. II, Method 5)

4 Carrying out the testing

The basins (dia. 12 cm, height 7 cm) are loosely filled with the substrate, almost up to the rim, patted smooth and watered. After a short period of waiting, in which an even moistening takes place and surplus water can run off, 1 g of cress is evenly scattered over the surface of the seed.

The seed should have contact with the bottom overall, if necessary, press down.

The basins are covered with a glass plate (evaporation protection), in the process of which a small gap should remain, that ensures the exchange of gas. The plant basins remain in the greenhouse approx. 7 days (the day of the sowing is included in the counting); the glass plates are removed if the germination plants touch them.

The plants are cut off exactly between the root and stalk for the harvest. The harvest should take place quickly, because the plants start to dry out immediately at the cutting surfaces. Following this, the plants are dried at 105°C and the water content is determined in the process.

The burning residual is determined at 550°C in the box-type furnace with the dried plant mass (see Chap. II, Method 11).

A cress cultivation on standard soil EE0 with corresponding additions of dung serves as a comparison.

5 Calculation and evaluation of the results

First, an observation is made as to whether the cress even starts to sprout. In the process, it can quickly be recognized whether the compost is an impediment to germination for sensitive plants or not. The cress reacts with a low capability for germinating or poor growth in the case of raised salt content. In comparison with the cultivation on EE0 substrate, it can be recognized how strong the impediment to sensitive plants is with the examined compost.

6 Literature

Abfall-Now, Number 1, PP. 14 - 16, Stuttgart

Determination of the air void volume in compost

1 Preliminary remark

For the determination of the air void volume in compost input materials and in composts.

2 Sample preparation

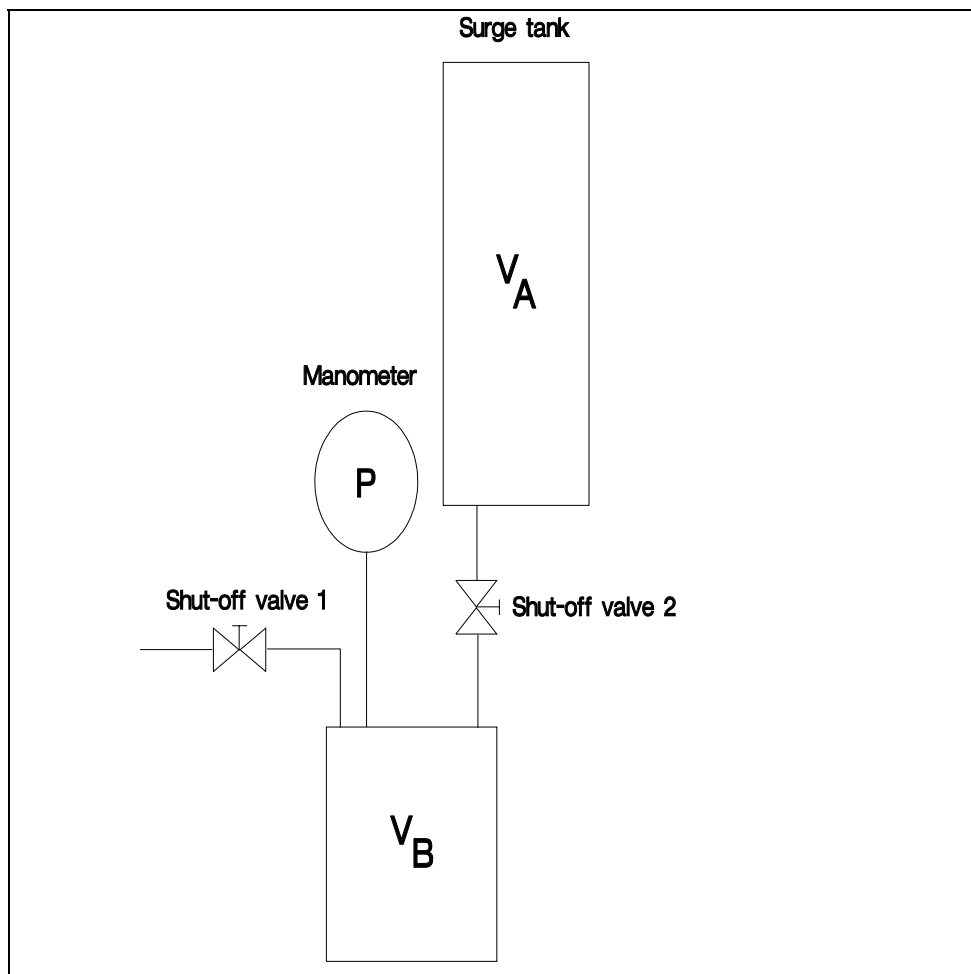
The sample should be filled into the pressure tank with as little disturbance as possible.

3 Materials

- Pressure tank with pressure display (e.g. manometer)
- Two shut-off valves
- Surge tank

4 Carrying out the testing

Open shut-off valve 2 to the surge tank. The fresh sample is filled in the pressure tank (V_B) (full). Shut-off valve 2 to the surge tank (V_A) is closed. The pressure tank is filled with 2 bar of compressed air through shut-off valve 1. Shut-off valve 1 is closed. The pressure is read (P_1). Shut-off valve 2 to the surge tank is opened. The second pressure value is read (P_2).



Illus.1: Measurement of the air void volume according to the pressure change method

5 Calculation and evaluation of the results

The relation

$$p \cong V = \text{const.}$$

delivers, at a constant temperature with

$$V_p = P_2 \cong V_A / P_1 - P_2$$

the air void volume V_p . The proportion of the air void volume is calculated with

$$O_K = (V_p / V_B) \cong 100$$

The result should be given accurate to 0.1%.

V_p	:	Air void volume [l]
V_A	:	Volume of the surge tank [l]
V_B	:	Volume of the pressure tank [l]
P_1	:	Pressure before opening shut-off valve 2 [Pa]
P_2	:	Pressure after opening shut-off valve 2 [Pa]
O_K	:	Porosity of the material [%]

6 Remarks

If the volumes of the pressure tank and the surge tank deviate a lot from each other, special attention is to be directed to a constant temperature. I.e. if $V_A \ll V_B$, even a light heating of V_A can cause a substantial measurement error.

Determination of the water capacity

1 Preliminary remark

By water capacity of soil, the water amount is meant that a soil sample can absorb up to full capillary saturation.

2 Sample preparation

A naturally moist sample is sieved to < 10 mm.

3 Materials

- Glass cylinder, height 120 mm, diameter inside 35.7 mm, with a close-meshed plastic net bottom
- Filter paper
- Watch glass
- Beaker 1 l, high form
- Scales

4 Carrying out the testing

The sieve bottom of the glass cylinder is covered up with moistened filter paper. Following this, the entire cylinder is weighed (m_0). The naturally moist sample material, < 10 mm, is filled into the cylinder with light shaking and weighed anew (m_e). The glass cylinder is then placed into the beaker. Following this, tap water is slowly added up to a height appropriate for the substrate, so the material can soak itself full with water from the bottom.

If the substrate is well moistened up to the surface, as much water is added again until the substrate is overdammed by approx. 1 cm. This coating is allowed to

stand for 24 h.

After this, the cylinder is taken out of the water, dried from the outside and placed on a water-saturated cellulose base covered with a watch glass. After 2 h, the glass cylinder is weighed back in (m_{moist}), then drained again and finally weighed back in anew.

This is repeated until no serious weight change can be determined any longer. Simultaneously with this determination, the water content of the fresh sample used has to be determined (WC).

5 Calculation of the results

Max. Water Capacity WK_{max} given in [% DS]:

$$WK_{\text{max}} = (E_{\text{moist}} - E_{\text{dr}}) / E_{\text{dr}} \cong 100\%$$

$$\text{Mass of the dry sample: } E_{\text{dr}} = (m_e - m_0) \cong (1 - WC/100)$$

$$\text{Mass of the wet sample: } E_{\text{moist}} = (m_{\text{moist}} - m_0)$$

WC: Water content of the fresh sample (naturally moist) in [%FS]

m_0 : Mass cylinder + wet filter paper

m_e : Mass cylinder + weighed-in sample

m_{moist} : Mass cylinder + wet sample

6 Literature

Laboratory method of the Münster Advanced Technical College, Laboratory for waste economy, Housing development water resources policy and environmental chemistry, 1993

Determination of organic contaminants

1 Polychlorinated dibenzodioxin and polychlorinated dibenzofurane (AbfKlärV - Sewage Sludge Decree - of April 15, 1992)

1.1 Preliminary remark

The following determination procedure is to be used for the PCDD and PCDF compounds selected for the determination stipulated for precautionary reasons.

It represents an investigation concept and is arranged in such a way that it integrates the necessary and possible elements of an analytical method; when it is followed and applied in laboratories experienced in trace analysis and when the measures for quality assurance and control for the realization of the AbfKlärV are regularly carried out, sufficiently dependable results are obtained.

Short description: The dried and ground sample is treated with ^{13}C -tagged PCDD and PCDF standards and extracted with toluene. The standards added and the PCDD/PCDF compounds possibly contained in the sample are freed from the interfering attendant material to a great extent, undone through capillary gas chromatography and subsequently determined with a mass spectrometer according to the MID (Multiple Ion Detection) technique, in the process of which the quantification step takes place after the isotope dilution method.

1.2 Sample preparation

The individual steps of the multi-stage sample preparation can be completely different at the qualified and experienced investigation departments. This is permissible, because the comparability, with the quality assurance and control accompanying the investigation, of the results obtained at the various investigation departments is ensured. There is an example for a procedure (1) set down in the following that is proven and used in many investigation laboratories (Official note: **Variations that make do without the dangerous working material benzene are to be given preference in the procedure described here, in so far as the attendant material interfering with the PCDD/PCDF analysis is sufficiently separated out and the comparability of the results is ensured.**):

50 g (in individual cases more) of the dried and ground sample are treated with the following ¹³C-tagged PCDD and PCDF: 5 ng each of 2,3,7,8-TetraCDD, 2,3,7,8-TetraCDF, 1,2,3,7,8-PentaCDD, 1,2,3,7,8-PentaCDF, 1,2,3,6,7,8-HexaCDD and 1,2,3,6,7,8-HexaCDF, as well as 10 ng each of 1,2,3,4,6,7,8-HeptaCDD, 1,2,3,4,6,7,8-HeptaCDF, OctaCDD and OctaCDF.

The sample is subsequently extracted in a Soxhlet apparatus for 20 h with toluene. The toluene extract is concentrated to approx. 25 ml. In some cases, in which the extract can only be concentrated to around 40 ml, benzene is used to fill up to 200 ml. The values given in the following in parentheses refer to the samples that are absorbed in 200 ml benzene. 50 g (or 75 g as the case may be) of aluminum oxide are filled into a chromatography column (60 x 4 cm) and provided with a layer of 50 g of sodium sulfate.

The extract is applied to the column and eluted with 300 ml (or 400 ml) of benzene and 300 ml (or 500 ml) of n-hexane/dichloromethane (98:2). The eluates are thrown out. Following this, the PCDD/PCDF fraction is eluted with 300 ml n-hexane/dichloromethane (1.1).

After a solution agent change in n-hexane, the samples are chromatographed on a "mixed" column of silica gel (2 g), silica gel/NaOH (5 g), silica gel (2 g), silica gel/H₂SO₄ (10 g), silica gel (2 g) and silica gel/AgNO₃ (5 g). Elution is done with 300 ml n-hexane.

The eluate is concentrated to approx. 5 ml and subsequently chromatographed on a column (30 x 2.5) filled with BioBeads S-X3, with cyclohexane/ethyl acetate (1:1) as the elution agent. The fraction from 100-160 ml contains the PCDD/PCDF. It is concentrated to a few millimeters, transferred to a 3 ml sample glass, the solvent is blown off in the nitrogen stream and the "residual" is absorbed with approx. 50 µl of toluene. After the wall of the sample glass is carefully rinsed with the solvent, 5 ng of ¹³C₆-1,2,3,4-TetraCDD are added and the volume of the sample solution is reduced to approx. 20 µl.

1.3 Materials

All of the devices coming into contact with the sample and its solutions/extracts have to be free of PCDD and PCDF within the scope of the verification limit of the procedure. All chemicals have to have a degree of purity that the mass-spectroscopic determination of PCDD and PCDF allows within the scope of the verification limit of the procedure. This is to be checked and guaranteed by regular blind-value examinations.

1. Usual laboratory devices
2. Gas chromatograph for capillary chromatography
3. Mass spectrometer with evaluation unit
4. Gas chromatographic separation columns
 - polar column, e.g. SP 2331 or SP 2330, 60 m
 - non-polar column, e.g. DB-5, 25 m
5. Separation columns/packing materials for multi-stage column chromatography
6. Calibration substances

For the quantification to be carried out according to the isotope dilution method, a solution of ^{13}C -tagged PCDD and PCDF standards is used that contains one PCDD or PCDF isomer each per homologous group.

1.4 Carrying out the testing

The identification and quantification of the 17 PCDD/PCDF compounds to be used for the TCDD toxic equivalent calculation takes place with capillary gas chromatography and mass spectrometric detection. The VDI Guideline 3499 (2) is to be applied during the execution of this step.

1.5 Calculation and evaluation of the results

The results are formed as the arithmetic mean value from two separate determinations (extractions). In the process, the mass concentrations of the 17 PCDD/PCDF compounds to be used for the TCDD toxic equivalent calculation are individually stated in ng/kg dry mass, rounded off to 1 ng/kg. The respective mass concentrations are multiplied with the following factors and the products added for the calculation of the sum of the 2,3,7,8-TCDD toxic equivalents (TE).

Toxic equivalents according to the BImSchG Decree (I-TE)

2,3,7,8-TCDD	= 1	2,3,7,8-TCDF	= 0.1
1,2,3,7,8-PeCDD	= 0.5	1,2,3,7,8-PeCDF	= 0.05
		2,3,4,7,8-PeCDF	= 0.5
1,2,3,6,7,8-HxCDD	= 0.1	1,2,3,6,7,8-HxCDF	= 0.1
1,2,3,4,7,8-HxCDD	= 0.1	1,2,3,4,7,8-HxCDF	= 0.1
1,2,3,,7,8,9-HxCDD	= 0.1	1,2,3,7,8,9-HxCDF	= 0.1
		2,3,4,6,7,8-HxCDF	= 0.1
1,2,3,4,6,7,8-HpCDD	= 0.01	1,2,3,4,6,7,8-HpCDF	= 0.01
		1,2,3,4,7,8,9-HpCDF	= 0.01
OCDD	= 0.001	OCDF	= 0.001

The so-called international toxic equivalents (I-TE values) are generally used for the calculation of the "toxic equivalents". The BGA values are likewise calculated and stated for a general comparability:

Toxic equivalents according to the BGA

2,3,7,8-TCDD	= 1	2,3,7,8-TCDF	= 0.1
1,2,3,7,8-PeCDD	= 0.1	1,2,3,7,8-PeCDF	= 0.1
		2,3,4,7,8-PeCDF	= 0.1
		1,2,3,6,7,8-HxCDF	= 0.1
1,2,3,6,7,8-HxCDD	= 0.1	1,2,3,4,7,8-HxCDF	= 0.1
1,2,3,4,7,8-HxCDD	= 0.1	1,2,3,7,8,9-HxCDF	= 0.1
1,2,3,7,8,9-HxCDD	= 0.1	2,3,4,6,7,8-HxCDF	= 0.1
		1,2,3,4,6,7,8-HpCDF	= 0.01
1,2,3,4,6,7,8-HpCDD	= 0.01	1,2,3,4,7,8,9-HpCDF	= 0.01
Sum TCDD	= 0.01	Sum TCDF	= 0.01
Sum PeCDD	= 0.01	Sum PeCDF	= 0.01
Sum HxCDD	= 0.01	Sum HxCDF	= 0.01
Sum HpCDD	= 0.001	Sum HpCDF	= 0.001
OCDD	= 0.001	OCDF	= 0.001

1.6 Remarks

Quality assurance and quality control

- The magnitude and reproducibility of the recovery rate (RR) of the ¹³C-tagged PCDD and PCDF standards for the selected separation steps are to be checked regularly; for OCDD/OCDF, the RR has to be > 40%, for all other compounds at > 70%.
- The efficiency of the measurement system (GC/MS) is to be checked and calibrated by regular measurements (e.g. maintaining inspection cards).

1.7 Literature

- (1) Hagenmaier, H Investigations of selected soils and plants for dioxin and furane. Research Report UBA, No. 107010100, Tübingen, 1988
- (2) VDI (Publ) VDI 3499, Sheet 1, Measurement of PCDD and PCDF in purified and raw gas from furnaces with the dilution method. Determination in the filter dust, boiler ash and in slag. Draft March, 1990
- (3) Anonymous Sewage Sludge Decree (AbfKlärV), Federal Legislation Gazette I, P. 912, April 15, 1992

2 Polycyclical aromatic hydrocarbons PAH

2.1 Preliminary remark

The procedure described here is suitable for the determination of the 6 PAH (fluoranthene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, benzo(ghi)perylene, and indeno(1,2,3-cd)pyrene) in accordance with the drinking water decree in biowaste composts, as well as the starting material in mass concentrations > 10 ng/g DS (verification limit).

The expected range of the individual substances is 50 - 5000 ng/g DS.

2.2 Sample preparation

The sample material is extracted in the ultrasound bath with an ethyl acetate/cyclohexane mix. The extract is pre-clarified by solid phase extraction on silica gel and still-existing interfering attendant material is separated using gel chromatography on a Bio-Beads S-X3 phase.

The determination of the PAH takes place with the help of high-performance fluid chromatography with an UV and fluorescence detector.

2.3 Materials and reagents

Devices:

- Ultrasound device
- Centrifuge
- Solid phase extraction unit
- Rotary evaporator
- Gel chromatography column
- High-performance fluid chromatograph with gradient elution, as well as UV and fluorescence detectors
- Separating column, suitable for the determination of PAH (RP C-18 phase).

Reagents:

- Ethyl acetate for analysis
- Cyclohexane for residual analysis
- Acetonitrile gradient grade
- Water ultra pure
- Silica gel one-time separating columns (3 ml)
- BioBeads S-X3
- PAH standard solutions

2.4 Carrying out the testing

1 g of sample material is extracted 3 times with 10 ml ethyl acetate-cyclohexane 1:1 (v/v) each time for 15 min. in the ultrasound bath and centrifuged for 10 min. with a rotary speed of 4000/min.

The combined extraction solutions are concentrated and pre-clarified through a mini-silica gel column. The subsequent gel chromatographic isolation takes place on a Bio-Beads S-X3 column (250 mm length, 10 mm diameter) with a mixture of ethyl acetate/cyclohexane 1:1.

The eluate of the solid phase extraction, concentrated to approx. 1 ml, is given to the chromatography column and eluted with a flow rate of 2 ml/min with a slight nitrogen overpressure. The singling from 20 ml contains the strongly colored impurities, while the PAH are to be found in the main fraction between 20 - 45 ml.

The main fraction is concentrated to approx. 1 ml, treated with 2 ml methanol and 0.5 ml acetonitrile, and concentrated to 0.5 ml. This solution is diluted to 1 ml with acetonitrile and a determination can be made with the help of the HPLC.

If the mostly yellow-colored solution still contains undissolved components, they should be separated with a spray filter.

HPLC determination:

Mini-bore separating column: Nucleosil 5 C18-PAH (250/1/4"/2)

Injection volume: 10 µl

Elution: acetonitrile/water mixture

A = acetonitrile/water (50/50), B = acetonitrile

0 - 7 min. 88% A, 12% B, flow 0.2 ml/min., isocratic

7 - 8 min. 65% A, 35% B, flow 0.3 ml/min., linear gradient

8 - 25 min. 0% A, 100% B, flow 0.3 ml/min., linear gradient

25 - 59 min. 0% A, 100% B, flow 0.3 ml/min., isocratic

59 - 60 min. 88% A, 12% B, flow 0.2 ml/min., linear gradient

60 - 80 min. 88% A, 12% B, flow 0.2 ml/min., isocratic

Detection: UV (254 nm), fluorescence (Ex:260 nm, Em: 410 nm)

2.5 Evaluation

The calibration with an external standard takes place with a 6-component standard solution.

Component	Retention time [min.]	Recovery [%]
Fluoranthene	25.30	93
Benzo(b)fluoranthene	32.00	96
Benzo(k)fluoranthene	34.00	95
Benzo(a)pyrene	35.10	76
Benzo(ghi)perylene	39.50	94
Indeno(1,2,3-cd)pyrene	41.70	96

3 Polychlorinated biphenyls (PCB) and organochlorine pesticides

3.1 Preliminary remark

The method serves to determine 6 selected polychlorinated biphenyls (PCB-28, PCB-52, PCB-101, PCB-138, PCB-153 and PCB-180 according to Ballshmiter) and the selected pesticides HCB, alpha, beta, gamma HCH, heptachlor, heptachlor epoxy resin, aldrin, dieldrin, endrin and DDT/DDD/DDE and isomers.

The sample purification using solid-phase extraction, as well as the gas chromatographic determination take place following the methods DIN 51527, Section 1 and DIN 38407, Section 2.

3.2 Sample preparation

The sample material is extracted in the Soxhlet with hexane and subsequently purified using solid-phase extraction on the column combination benzene sulfonic acid/silica gel. The separation of the individual substance groups takes place through a step-wise elution with hexane and toluene/hexane mixtures of various compositions.

3.3 Materials and reagents

Devices:

- Soxhlet apparatus
- Rotary evaporator
- Solid phase extraction unit

Reagents:

- n-hexane for residual analysis
- Benzene sulfonic acid one-time separating column
- Silica gel one-time separating column
- Toluene for analysis
- PCB standard
- Pesticide standard

3.4 Carrying out the testing

10 g of compost sample are extracted with 200 ml hexane for 8 h in the Soxhlet. Following this, rinse the apparatus 3 times with 10 ml hexane each time and combine with the extract. The sample, concentrated almost up to dryness, is absorbed with 0.5 ml n-hexane and deposited on the frit of a dry benzene sulfonic acid separating column, that is placed on a 3 ml silica gel separating column using an adapter. The sample is brought to the packing of the upper column by the application of a slight underpressure and eluted twice with 1 ml n-hexane each time to the lower column. Following this, remove the benzene sulfonic acid column and transfer the sample into a receiver by eluting the silica gel column three times with 0.5 ml hexane.

This eluate (1st fraction) contains the PCB, as well as several organochlorine pesticides.

Further organochlorine pesticides can be isolated in a second fraction by a step-wise elution of the silica gel column with hexane/toluene mixtures.

3 ml m-hexane/toluene (95:5)

6 ml m-hexane/toluene (65:35)

One obtains a third fraction by eluting the separated benzene sulfonic acid separating column with 3 ml n-hexane/toluene (95:5).

The three fractions contain the following pesticides and PCB:

	Fraction			Recovery [%]
	1	2	3	
PCB 028	X	-	-	97
PCB 052	X	-	-	93
PCB 101	X	-	-	77
PCB 153	X	-	-	71
PCB 138	X	-	-	74
PCB 180	X	-	-	79
α -HCH	-	X	-	90
Hexachlorobenzene	X	-	-	59
β -HCH	-	-	X	87
Lindane	-	X	-	96
Heptachlor	X	-	-	94
Aldrin	X	-	-	95
Heptachlor epoxy resin	-	X	X	108
o,p'-DDE	X	-	-	113
p,p'-DDE	X	-	-	109
Dieldrin	-	-	X	105
o,p'-DDD	-	X	-	102
Endrin	-	-	-	0
p,p'-DDD	-	X	-	112
o,p'-DDT	X	-	-	109
p,p'-DDE	X	X	-	110

Endrin cannot be verified with this method.

The calibration of the method takes place with an external standard.

The calibration of the chromatographic determination with an internal standard takes place with PCB 209.

Determination methods:

The determination methods mentioned are to be understood as examples; in individual cases, the methods are to be adapted to the equipment in the laboratory.

GC/ECD method for PCB

Sample feed:	splitless
Injector temperature:	280°C
Detector temperature:	300°C
Separating column:	SE-54 (L = 50 m / ID = 0.32 mm / df = 0.35 µm)
Carrier gas:	Nitrogen
Temperature schedule:	1 min. at 80°C 25°C/min to 180°C 10°C/min to 210°C 5°C/min to 290°C 10 min. at 290°C

GC/ECD method for pesticides and PCB

Sample feed:	splitless
Injector temperature:	280°C
Detector temperature:	325°C
Separating column:	CP-SIL (L = 50 m / ID = 0.25 mm / df = 0.25 µm)
Carrier gas:	Nitrogen
Temperature schedule:	1 min. at 45°C 25°C/min to 195°C 10°C/min to 205°C 1°C/min to 230°C 15°C/min to 305°C 12 min. at 305°C

3.5 Literature

DIN 51 527, Section 1, Determination of polychlorinated biphenyls (PCB). Beuth Verlag, Berlin (1987)

DIN 38 407, Section 2 (draft), German uniform proc. for water, waste water and slurry investigation; Jointly ascertainable material groups (Group F); Gas chromatographic determination of halogen-organic compounds not easily volatilized (F2). Beuth Verlag, Berlin (1991).

4 Phenoxyalkane carboxylic acid herbicide 2,4-D and 2,4,5-T

4.1 Preliminary remark

The procedure described here is suitable for the determination of the herbicide 2,4-dichlorophenoxy ethanoic acid (2,4-D) and 2,4,5-trichlorophenoxy ethanoic acid (2,4,5-T) in biological waste composts, as well as in the starting materials in mass concentrations > 5 ng/g DS.

The expected range of the individual substances is 10 - 100 ng/g DS. Reference substances for the gas chromatographic determination are the methyl esters of the acids.

The enrichment in an RP-C18 phase, as well as obtaining the derivatives takes place following the draft of the method DIN 38407, Section 14 of 1990 on water, waste water and slurry investigations.

4.2 Sample preparation

The extraction of 10 g of sample material takes place with a watery methanol solution (2% H₂O) in the ultrasound bath. Following this, the contaminants are enriched in an inversion phase (octadecyl) of a watery agent at pH 2 and eluted with methanol. After the esterification with methanol and conc. sulfuric acid, the methyl ester derivatives are extracted with n-hexane and, if necessary, repurified through silica gel.

4.3 Materials and reagents

Devices:

- Various glass devices
- Ultrasound bath
- Filtration unit
- Solid phase extraction unit
- Rotary evaporator

Reagents:

- Methanol for analysis
- HCl solution (pH 2)
- Toluene for analysis
- Octadecyl one-time separating column
- Hexane for residual analysis
- Sulfuric acid conc.
- Sodium sulfate
- Sodium hydrogen carbonate
- Silica gel one-time separating column (3 ml)
- Standard solutions of the chlorphenoxy ethanoic acids and their methyl esters

4.4 Carrying out the testing

10 g of sample material is extracted 3 times each with 30 ml watery methanol solution (2% H₂O) in the ultrasound bath for 10 min. and subsequently filtered. For the complete isolation of the phenoxyalkane carboxylic acid in octadecyl phases, the extract is concentrated almost to dryness and the residual solution is diluted with diluted HCl (pH 2) to approx. 100 ml and sucked through the conditioned octadecyl column. Rewash the column with 10 ml of water and suck dry for approx. 5 min. After this, wash 2 times with 1 ml of hexane and lastly suck up completely. Elute the phenoxyalkane carboxylic acids subsequently twice with 1 ml of methanol each time.

For the esterification of the carboxylic acids, dilute the eluate to 4.5 ml with methanol and add approx. 0.5 ml conc. sulfuric acid drop by drop. Allow the solution to react in a closed flask with frequent twirling around for 10 min. Following this, add 50 ml of Na₂SO₄ solution (5%) and extract the methyl esters with n-hexane. Wash the organic phase with 50 ml NaHCO₃ solution (4%), dry through a little Na₂SO₄, and concentrate to a defined volume.

If a repurification is necessary, one brings the sample to a 3 ml silica gel column (conditioned with hexane) and elutes with toluene-hexane mixture 70:30. Concentrate the eluate almost to dryness, blow dry with nitrogen and absorb the residual into 1 ml of hexane.

The calibration of the gas chromatographic determination method with an external standard takes place with methyl ester reference solutions. The calibration of the overall procedure with an external standard takes place with the free acids.

GC/ECD determination method

Sample feed:	splitless
Injector temperature:	300°C
Detector temperature:	300°C
Separating column:	CP-SIL 8 CB (L = 50 m / ID = 0.25 mm / df = 0.25 µm)
Carrier gas:	Nitrogen
Temperature schedule:	Starting temperature 130°C 1st rate 1°C/min to 185°C 2nd rate 10°C/min to 300°C

4.5 Remarks**Column conditioning:**

Give a column filling, one after the other, of hexane, methanol, as well as water acidified to pH 2, to a 3 ml octadecyl column (500 mg) and suck through under vacuum, avoid dry running in the process.

4.5 Literature

DIN 38 407, Section 2 (draft), German uniform proc. for water, waste water and slurry investigation; Jointly ascertainable material groups (Group F); Determination of phenoxyalkane carboxylic acid using, and mass spectrometric detection according to, solid-fluid extraction and obtaining derivatives (F14). Beuth Verlag, Berlin (1991).

Determination of the total carbon content

Depending on the type of task, the course of analysis leads, starting with the determination of the inorganic TIC (total inorganic carbon), through the determination of the total carbon content TC (total carbon) to the calculation of the organically-bound carbon TOC (total organic carbon).

The following determinations can be cited:

- COULOMETRIC DETERMINATION
- DETERMINATION USING ELEMENTARY ANALYSIS

1 Coulometric determination

1.1 Preliminary remark

The determination method permits the analysis of more than 50 mg of sample material (50 - 1000 mg weigh-in).

1.2 Sample preparation

Drying the unscreened fresh sample (original sample) at 105°C.

Grinding of at least 30 g of dry substance with a suitable grinder to < 250 µm.

1.3 Materials

- Precision analytical scales
- Vitreous fused silica combustion boat for the weigh-in of the sample
- Combustion furnace or coulomat
Combustion device with integrated post-combustion
Carbon dioxide analyzer
- Combustion temperature in the vitreous fused silica tube: 950°C

1.4 Carrying out the testing

The sample is burned at approx. 950°C in the tube furnace in the oxygen stream, the gas mixture that arises is dried and the CO₂ is coulometrically titrated after quantitative absorption in the alkaline barium perchlorate solution.

The weigh-in is approx. 50 mg.

1.5 Calculation and evaluation of the results

The amount of CO₂ formed is determined through the pH value change. This measurement supplies the total carbon content TC.

Description: Total carbon content TC in [%DS]

1.6 Remarks

Approx. 3-5 g of dried sample are treated with a strong acid (approx. 10 ml HCl_{conc}) in a closed reaction vessel for the determination of the inorganic carbon content (TIC).

The carbon dioxide stripped from the carbonates in this way is fed to the analyzer and quantitatively absorbed in the barium perchlorate solution and coulometrically titrated.

The content of TC and TIC determined in this way serve in the calculation of the TOC:

$$\text{TOC} = \text{TC} - \text{TIC}$$

2 Determination using elementary analysis

2.1 Preliminary remark

The determination of the C_{tot} content using elementary analysis is a workable alternative for the simplification and streamlining of the analyses of a sample to be carried out (also see analysis of the N_{tot} content). On top of this is also the significant health aspect and the low use of chemicals that represent contamination of the environment.

2.2 Sample preparation

Drying the unscreened fresh sample at 105°C.

Grinding of at least 30 g of dry substance with a suitable grinder to < 250 µm.

2.3 Materials

Elementary analyzer

Carrier gas: Helium

Gas chromatograph with a thermal conductivity detector (TCD)

2.4 Carrying out the testing

10 - 80 mg of the sample, dried and ground fine, are oxidized at approx. 1000°C in an oxygen pulse while hermetically sealed. The gas is separated with gas chromatography and measured with a TCD.

2.5 Calculation and evaluation of the results

The determination supplies the TC content.

Description: Total carbon content TC in [%DS]

2.6 Literature

Various methods for the determination of organic, inorganic and total carbon content (TOC, TIC, TC) in selected solid waste templates; Kraschon G., C.U. Schmidt, A. M. Bahadir; Garbage and Waste 3/93

