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Sludge, treated biowaste and soil — Determination of Kjeldahl nitrogen

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Foreword

This document is a working document.

This document TF WI has been prepared by CEN/BT/Task Force 151 – Horizontal Standards in the Field of Sludge, Biowaste and Soil, the secretariat of which is held by Danish Standards.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

This standard is applicable and validated for several types of matrices. The table below indicates which ones.

Material	**Validated for** **(type of sample, e.g. municipal sludge, compost)**	**Reference:**
Sludge		JRC report
Soil		JRC report
Biowaste		JRC report
Waste		

Annex A and Annex B are informative.

Introduction

This document is developed in the project 'Horizontal'. It is the result of a desk study "DS 16: Determination of total phosphorus, total nitrogen and nitrogen fractions" and aims at evaluation of the latest developments in assessing Kjeldahl nitrogen in sludge, treated biowaste and soil. After an evaluation study, in which e.g. the ruggedness of the method was studies, a European wide validation of the draft standard has taken place. The results of the desk studies as well as the evaluation and validation studies have been subject to discussions with all parties concerned in CEN. The standard is part of a modular horizontal approach in which the standard belongs to the analytical step.

Until now test methods determining properties of materials were often prepared in Technical Committees (TCs) working on specific products or specific sectors. In those test methods often steps as sampling, extraction, release or other processing, analyses, etc were included. In this approach it was necessary to develop, edit and validate similar procedural steps over and over again for every material or product. Consequently this has resulted in duplication of work. To avoid such duplication of work for parts of a testing procedure references to parts of test methods from other TCs were introduced. However the following problems are often encountered while using references in this way: 1) The referenced parts are often not edited in a way that they could easily be referred to, 2) the referenced parts are often not validated for the other type of material and 3) the updates of such test standards on products might lead to inadequate references.

In the growing amount of product and sector oriented test methods it was recognised that many steps in test procedures are or could be used in test procedures for many products, materials and sectors. It was supposed that, by careful determination of these steps and selection of specific questions within these steps, elements of the test procedure could be described in a way that can be used for all materials and products or for all materials and products with certain specifications.

Based on this hypothesis a horizontal modular approach is being investigated and developed in the project 'Horizontal'. 'Horizontal' means that the methods can be used for a wide range of materials and products with certain properties. 'Modular' means that a test standard developed in this approach concerns a specific step in assessing a property and not the whole ” chain of measurement” (from sampling to analyses). A beneficial feature of this approach is that “modules” can be replaced by better ones without jeopardizing the standard “chain”.

The use of modular horizontal standards implies the drawing of test schemes as well. Before executing a test on a certain material or product to determine certain characteristics it is necessary to draw up a protocol in which the adequate modules are selected and together form the basis for the test procedure.

The modules that relates to this standard are specified in section XX Normative references.

An overview of modules and the manner, in which modules are selected will be worked out later, at which time proper reference in this standard will be provided.

Sludge, treated biowaste and soil — Determination of Kjeldahl nitrogen

WARNING — Waste and sludge samples may contain hazardous and inflammable substances. They may contain pathogens and be liable to biological action. Consequently, it is recommended that these samples should be handled with special care. Concentrated sulphuric acid is used in Kjeldahl digestion. Sulphuric acid causes severe damages to skin and eyes, therefore protective gloves and glasses have to be worn. Special instructions of the manufacturer of the digestion apparatus have to be followed. National regulations should be followed with respect to microbiological and chemical hazards with this method.

1 Scope

This method is applicable to determine Kjeldahl nitrogen according to the Kjeldahl procedure in sludge, treated biowaste and soil. Nitrate and nitrite are not included. Compounds with nitrogen bound in N-N, N-O linkages and some heterocycles (pyridines) are only partially determined.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12880, *Characterization of sludges — Determination of dry residue and water content*.

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods.*

CSS99031 Sludge, treated biowaste, and soils in the landscape – Sampling – Framework for the preparation and application of a sampling plan

CSS99058 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 1: Guidance on selection and application of criteria for sampling under various conditions

CSS99057 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 2: Guidance on sampling techniques

CSS99032 Sludge, treated biowaste, and soils in the landscape – Sampling - Part 3: Guidance on sub-sampling in the field

CSS99059 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 4: Guidance on procedures for sample packaging, storage, preservation, transport and delivery

CSS99060 Sludge, treated biowaste, and soils in the landscape – Sampling – Part 5: Guidance on the process of defining the sampling plan

CSS99034 Soil, sludge and treated biowaste – Guidance for sample pre-treatment

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1
Kjeldahl nitrogen
amount of nitrogen that is determined after Kjeldahl digestion and titration

3.2
dry residue
dry mass fraction of the sample obtained after the specified drying process. It is expressed as percent or in grams per kilogram

4 Principle

The dried and homogenised, moist or liquid material is digested in a suitable Kjeldahl tube with sulphuric acid.

To rise the temperature potassium sulphate is added and titanium dioxide or copper sulphate is used as a catalyst. After adding sodium hydroxide to the digestion solution the produced ammonium from all nitrogen species is evaporated by distillation as ammonia. This is condensed in the cooling system and rinses into a conical flask with boric acid solution. This solution is titrated with sulphuric acid until the endpoint, which is detected potentiometrically or using an indicator.

5 Interferences and sources of errors

The Kjeldahl method in principle does not capture all nitrogen compounds. The nitrogen, that occurs in N-N and N-O linkages (e.g. azo-, nitro- and nitroso compounds, hydrazines, hydrazones, oximes, pyrazolones, isooxazoles, dia- and triazines) is not completely recorded. Furthermore the inorganic fraction (nitrate and nitrite) is not determined. Another source of error include impurities in the apparatus. Therefore the apparatus has to be rinsed after each analytical series and blank determinations have to be carried out. The amount of sulphuric acid used in digestion depends on the composition of the sample. A ratio of sample to acid of at least 1:10 (w/V) shall be used for samples with high content of organic matter. Digestion temperature shall not rise above 380 °C - 400 °C to avoid analyte losses.

Table 1 — Amounts of sulphuric acid consumption by various materials during Kjeldahl digestion

Material	**Consumption of sulphuric acid (36 mol/l) during digestion** ml/g
Soil organic C	10,0
Soil organic matter	5,8
Al_2O_3	1,63
Fe_2O_3	1,04
Clay	0,60
$CaCO_3$	0,55
Silt	0,33
Sand	0
Salicylic acid	6,76
$Na_2S_2O_3$	0,58
Reduced Fe	1,50

6 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

6.1 Water, complying with grade 2 as defined in EN ISO 3696.

6.2 Sulphuric acid, H_2SO_4, ρ = 1,84 kg/l.

6.3 Catalyst mixture

Grind and thoroughly mix 200 g of potassium sulphate, 20 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$) and 20 g of titanium dioxide (TiO_2), with the crystal structure of anatase.

6.4 Sodium hydroxide, *c*(NaOH) = 10 mol/l.

6.5 Boric acid solution, H_3BO_3, ρ = 20 g/l.

6.6 Mixed indicator

Dissolve 0,1 g of bromocresol green and 0,02 g of methyl red in 100 ml ethanol.

6.7 Sulphuric or hydrochloric acid solution, $c(H^+)$ = 0,01 mol/l – 0,50 mol/l.

7 Apparatus

Usual laboratory apparatus, and in particular the following:

7.1 Kjeldahl digestion flasks or tubes, of nominal volume 50 ml, suitable for digestion stand (7.2).

7.2 Digestion stand, suitable for digestion of samples with sulphuric acid at a temperature near to 400 °C.

7.3 Distillation apparatus, e.g. of the Parnas-Wagner type or other suitable distillation apparatus with steam generator.

8 Sampling and sample pre-treatment

8.1 Sampling

pling shall be carried out in accordance with sampling standards CSS99031-32 and 99057-60.

Samples shall be stored in suitable containers with an appropriate closure material, for example PE.

8.2 Sample pre-treatment

All samples shall be pre-treated according to the special standard (CSS99034) in the field of sludge, treated biowaste and soil. Normally, they are dry, homogeneous and of a defined grain size, liquid or moist. Results are referred to dry residue, so that in case of liquid or moist samples a special sample has to be used for the determination of dry residue.

During the digestion procedure care has to be taken not to lose amounts of nitrogen. Therefore, digestion temperatures exceeding 400 °C shall be avoided.

Dry residue of the sample is determined by the specified drying process according to EN 12880.

9 Procedure

9.1 General

Homogeneity of the laboratory sample and the test sample has to be guaranteed.

9.2 Digestion

Place a test portion of the dried sample, of about 0,2 g to 1 g or undried sample with the corresponding dry matter to the nearest of 0,1 % accuracy in the digestion flask or tube (7.2). To use the semimicro- or the macromethod respective flasks or tubes have to be used with suitable volumes. Add 10 ml sulphuric acid (6.2).

NOTE 1 The amount of sulphuric acid may be adapted to the size of the flask or tube.

Swirl until the acid is thoroughly mixed with the sample. Allow the mixture to stand for cooling. Add 2,5 g of the catalyst mixture (6.3).

NOTE Other catalyst mixtures, that contain mercury or selenium, are not allowed due to their toxicity towards human health and the environment. The effectiveness of other catalyst mixtures has to be proven by an expert group, before using them in the Kjeldahl procedure.

To avoid spattering of samples the digest may be left at room temperature for 24 h or a longer time before digestion.

Heat until the digestion mixture becomes clear. Boil the mixture gently up to 5 h so that the sulphuric acid condenses about 1/3 of the way up to the neck of the flask or the end of the tube. During the digestion procedure care has to be taken not to lose amounts of nitrogen. Therefore, avoid temperatures exceeding 400 °C. The use of a temperature programme, that ensures gentle heating before reaching the boiling point is recommended, especially for liquid samples or samples with high content of organic matter.

The time of boiling period may be different and depends on the sample material. The solution has to be clear at the end of boiling. The amount of test material and added chemicals and catalysts can be changed in the ratio described in the working instructions. The semimicro and the macro version of the Kjeldahl procedure are suitable for some materials.

9.3 Titration

After completion of the digestion step, allow the flask or tube to cool and add 20 ml of water (6.1) slowly while shaking. Then swirl the flask or tube to bring any insoluble material into suspension and transfer the contents to the distillation apparatus (7.3). Rinse three times with water (6.1) to complete the transfer.

Add 50 ml of boric acid solution (6.5) to a 200 ml conical flask and place the flask under the condenser of the distillation apparatus in such a way that the end of the condenser dips into the solution. Add 20 ml of sodium hydroxide (6.4) to the funnel of the apparatus and run the alkali slowly into the distillation chamber.

Modern Kjeldahl apparatus use the digestion tubes for distillation and the addition of chemicals is programmed. The distillation is done automatically. A potentiometric titration with an endpoint of pH = 5,0 is possible.

The best way of distillation is steam distillation. A rate of up to 25 ml/min is applicable.

Stop the distillation when 100 ml of distillate have been collected (the amount for quantitative results depends on the dimensions of the apparatus), rinse the end of the condenser, add a few drops of mixed indicator (6.6) to the distillate and titrate with sulphuric or hydrochloric acid solution (6.7) to a violet endpoint.

The concentration of the sulphuric or hydrochloric acid solution (6.7) shall be chosen according to the expected amount, that is consumed in titration of different sample matrices. For sludges and treated biowaste the use of $c(H^+)$ = 0,50 mol/l is recommended, due to nitrogen contents > 0,5 %. For soil samples the use of

other acid concentrations decreases analytical error but care has to be taken on the contamination of titration acids by carbon dioxide, which changes the acid concentration and therefore reduces its stability.

NOTE The final determination method can be done by other validated methods than titration (spectrophotometric determination of ammonium, manually or by automated methods).

9.4 Calibration

Calibration substances with known and unchangeable content of nitrogen are used to control the digestion and the apparatus. This may be ammonium sulphate, acetanilid, l-asparaginacid, sulfanilacid or other aminoacids with known nitrogen content. Besides these substances certified reference materials can be used to control the whole procedure.

9.5 Blank determination

Carry out at least two blank determinations in each series and use the average blank value for subsequent calculations.

10 Calculation

Calculate the content of nitrogen w_N, in milligram per kilogram, using the equation

$$w_N = \frac{(V_1 - V_0) \times c(H^+) \times M_N \times 100}{m \times w_{dm}} \quad (1)$$

where

V_1 is the numerical value of the volume of the sulphuric acid (6.2) used in the titration of the sample, in millilitres;

V_0 is the numerical value of the volume of the sulphuric acid (6.2) used in the titration of the blank test, in millilitres;

$c(H^+)$ is the numerical value of the exact concentration of H^+ in the sulphuric acid solution or hydrochloric acid solution (6.7), in moles per litre (e.g. if 0,01 mol/l sulphuric acid solution is used, $c(H^+)$ = 0,02 mol/l);

M_N is the numerical value of the molar mass of nitrogen, in gram per mole (M_N = 14);

m is the numerical value of the mass of the test sample, in kilogram;

w_{dm} is the dry mass portion, expressed as g/100 g on the basis of oven dried material determined in accordance with EN 12880.

11 Expression of results

The result shall be expressed in mg/kg or % dry matter and reported to two significant figures.

12 Precision data

The performance characteristics of the method (Annex C) data have been evaluated. Table 1 gives the resulting typical values for repeatability and reproducibility limits as their observed ranges. The typical value is derived from the data in Table C.2 in Annex C by taking the median value and rounding the numbers.

Table C.1 — Typical values and observed ranges of the repeatability and reproducibility limits

The reproducibility limit provides a determination of the differences (positive and negative) that can be found (with a 95 % statistical confidence) between a single test result obtained by a laboratory using its own facilities and another test result obtained by another laboratory using its own facilities, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard and the two laboratory samples are obtained from the same primary field sample and prepared under identical procedures. Conversely, the repeatability limit refers to measurements obtained from the same laboratory, all other conditions being identical. The reproducibility limit and the repeatability limit do not cover sampling but cover all activities carried out on the laboratory sample including its preparation from the primary field sample.		
Results of the validation of the determination of Kjeldahl nitrogen in soil, sludge and treated biowaste	Typical value %	Observed range %
Repeatability limit, r	9	*4 – 20*
Reproducibility limit, R	45	12 – 68

NOTE 1. The above results refer to the difference that may be found between two test results performed on two laboratory samples obtained under the same conditions. In the case when reference is made to the dispersion of the values that could reasonably be attributed to the parameter being measured, the above typical reproducibility values and observed reproducibility ranges should be divided by √2 to obtain the corresponding typical dispersion limit and its observed range. In the example of Kjeldahl N in Sludge 2 the result and its dispersion limit is 35.3 ± 4.36 g/kg (2 * sR = 12.3 % of 35.3). This means that with a 95 % statistical confidence, the values reasonably attributable to the measured parameter are larger than 30.9 g/kg and lower than 39.6 g/kg.

NOTE 2. The repeatability limit (r) and the reproducibility limit (R) as given in Table C.2 (Annex C) and in this table are indicative values of the attainable precision if the determination of Kjeldahl nitrogen is performed in accordance with this standard [CSS99021].

NOTE 3 A limited number of materials and parameters were tested. Consequently, for other materials and parameters, performance characteristics may fall outside the limits as derived from the validation of the the determination of Kjeldahl nitrogen in soil, sludge and treated biowaste.

NOTE 4 In particular for relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Table C.2 (Annex C) and this table.

13 Test report

The test report shall contain the following information:

a) a reference to this European Standard including its date of publication;

b) all information necessary for identification of the sample tested;

c) the method used for the determination of Kjeldahl nitrogen;

d) the results, calculated as specified in 10 and expressed according to 11;

e) any deviation from this standard, and any facts which may have influenced the result.

Where the test is not carried out in accordance with this standard, reference may only be made to EN xxxx:200x in the report in case all deviations from the procedures prescribed in this standard are indicated in the report stating the reason for deviation.

Annex A
(informative)

Validation of methods

The Kjeldahl procedure is validated in ISO 11261 for soils. The precision data according to ISO 5725 were determined from an experiment conducted in 1992 involving 14 laboratories and 4 soil samples. The results obtained are given in table A.1.

Table A.1 — Validation data for soil (ISO 11261)

Sample No.	**Nitrogen content, w_N** mg/g	**S_r** %	**S_R** %
1	0,98	6,1	27,0
2	3,11	3,9	18,6
3	6,70	2,8	15,9
4	10,88	2,4	8,2
S_r is the XXXXXXXXXXXXX, in %; S_R is the XXXXXXXXXXXXX, in %.			

Annex B
(informative)

Data from Desk study 16

Performance data using air dried samples in terms of repeatability and reproducibility have been determined during desk study 16 [4] using statistical data from 6 repeated measurements of one sample analysed on two days. The results obtained for soil are given in table B.1. The results obtained for treated biowaste and sludge are given in table B.2.

Table B.1 — Precision data: soil

Sample No.	**Nitrogen content, w_N %**	***S*** % N	**S_r** %
SO1	0,24	0,01	4
SO13	0,28	0,01	4
SO9	0,40	0,01	3
Repeatability is expressed by standard deviation in % N and as relative standard deviation : s_r *S* is the XXXXXXXXXXXXX, in % N; S_r is the XXXXXXXXXXXXX, in %.			

Table B.2 — Precision data: treated biowaste and sludge

Sample No.	**Nitrogen content, w_N %**	***S*** % N	**S_r** %
CW1	1,48	0,03	2
CW5	1,52	0,03	2
SL4	1,92	0,02	1
SL11	0,66	0,03	5
Repeatability is expressed by standard deviation in % N and as relative standard deviation : s_r *S* is the XXXXXXXXXXXXX, in % N; S_r is the XXXXXXXXXXXXX, in %.			

Linearity of standards and standard addition:

Glycine: r = 0,9997 % N up to 3,7 % N

Addition soil: *r* = 0,9997 % N up to 1,5 % N

Addition biowaste: *r* = 0,9977 % N up to 4,7 % N

Addition sewage sludge: *r* = 0,99996 % N up to 4,7 % N

LOQ = 0,1 % N ; LOD = 0,03 % N (for use of 0,25 N sulphuric acid in titration)

Recovery = 94 % – 103 %

Annex C (informative)

Repeatability and reproducibility data

C.1 Performance characteristics

C.1.1 Objective of the interlaboratory comparison

In a European wide interlaboratory comparison study according to ISO 5725-2, the performance characteristics of the standard “Determination of Kjeldahl nitrogen in soil, sludge and treated biowaste” were established.

C.1.2 Materials used in the interlaboratory comparison study

The interlaboratory comparison of Determination of Kjeldahl nitrogen in soil, sludge and treated biowaste was carried out with 11 - 12 European laboratories on 6 materials. The materials selected for the interlaboratory comparison were chosen to represent soil, sludge and biowaste as broad as possible, because the standard will find general application across different types of soil and soil related materials. (detailed information can be found in the final report on the Interlaboratory comparison study mentioned in the Bibliography).

In the interlaboratory comparison study the following starting points were used:

- The laboratory samples were all taken from one large batch of the different materials according to the normal practice. The normal size reduction and the normal repeated mixing were carried out as needed to obtain representative laboratory samples from the large batch sample (ref JRC).
    - Note : the samples provided for the validation should not be confused with reference samples provided for certification purposes, as the performance results obtained have to be directly applicable to daily practice (less rigorous sample preparation than for a reference material).
- The experimental plan was designed by project HORIZONTAL on the basis of each laboratory being given two laboratory samples of each material to be tested. This is in accordance with ISO 5725-2.
- The materials examined cover all the grain size classes to which the the Determination of Kjeldahl nitrogen in soil, sludge and treated biowaste applies: very fine grained materials (like sludge: 0 µm to about 125 µm) and fine-grained materials (soil and compost: 0 mm to 4 mm).

Table A.1 provides a list of the types of materials chosen for testing and the selected components.

Table A.BD.2 — Material types tested and components analysed in the interlaboratory comparison of the determination of Kjeldahl nitrogen in soil, sludge and treated biowaste.

Grain size class	Sample code	Material type tested	Parameters/congeners
Sludge (<0.5 mm)	Sludge 1	Mix 1 of municipal WWTP sludges from North Rhine Westphalia, Germany	Kjeldahl N
	Sludge 2	Mix 2 of municipal WWTP sludges from North Rhine Westphalia, Germany	Kjeldahl N
Fine grained (< 2 mm)	Compost 1	Fresh compost from Vienna, Austria	Kjeldahl N
	Compost 2	Compost from Germany	Kjeldahl N
	Soil 4	A sludge amended soil from Hohenheim, Germany	Kjeldahl N
	Soil 5	An agricultural soil from Reading, UK	Kjeldahl N

C.1.3 Interlaboratory comparison results

The statistical evaluation was conducted according to ISO 5725-2. The average values, the repeatability standard deviation (s_r) and the reproducibility standard deviation (s_R) were obtained (Table C.2).

The repeatability is determined as an interval around a measurement result (i.e. "repeatability limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another, both test results being obtained under the following conditions: The tests are performed in accordance with all the requirements of the present standard by the same laboratory using

its own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures.

The repeatability limit was calculated using the relationship : $r_{test} = f \cdot \sqrt{2} \cdot s_{r,test}$ with the critical range factor f = 2.

For instance, the repeatability limit around a measurement result of 40 g Kjeldahl N/kg is ± 3.68 g Kjeldahl N/kg (i.e ± 9 % of 40).

NOTE The above relationship refers to the difference that may be found between two measurement results performed each on two laboratory samples obtained under the same conditions. The value f = 2 used in the factor $f \cdot \sqrt{2}$ corresponds to the theoretical factor of 1,96 for a pure normal distribution at 95 % statistical confidence. Also, this value f = 2 corresponds to the usual value k = 2 of the coverage factor recommended in the Guide to the expression of Uncertainty in Measurement (GUM). However it may be necessary to use a larger value for f in situation as described in clause 12.

The reproducibility, like repeatability is also determined as an interval around a measurement result (i.e. "reproducibility limit"). This interval corresponds to the maximum difference that can be expected (with a 95% statistical confidence) between one test result and another test result obtained by another laboratory, both test results being obtained under the following conditions : The tests are performed in accordance with all the requirements of the present standard by two different laboratories using their own facilities and testing laboratory samples obtained from the same primary field sample and prepared under identical procedures.

The reproducibility limit was calculated using the relationship: $R = f \cdot \sqrt{2} \cdot s_R$ with the critical range factor f = 2.

For instance, the reproducibility limit around a measurement result 40 g Kjeldahl N/kg is ± 17.9 g Kjeldahl N/kg (i.e ± 45 % of 40).

NOTE The above relationship refers to the difference that may be found between two measurement results performed each on two laboratory samples obtained under the same conditions. The value f = 2 used in the factor $f \cdot \sqrt{2}$ corresponds to the theoretical factor of 1,96 for a pure normal distribution at 95 % statistical confidence. Also, this value f = 2 corresponds to the usual value k = 2 of the coverage factor recommended in the Guide to the expression of Uncertainty in Measurement (GUM). In the case when reference is made to the dispersion of the values that could reasonably be attributed to the parameter being measured, the dispersion limit is equal to $k \cdot s_R$ with the usual value

k = 2, resulting in a dispersion limit lower than the reproducibility limit (i.e. a ratio of √2). However it may be necessary to use a larger value f · √2 (or k) in situation as described in clause 12 .

In case of relatively heterogeneous materials, the repeatability and the reproducibility limits may be larger than the values given in Tables C.2 (this means that the value chosen for the critical range factor f is larger than 2 as well as for the coverage factor k for dispersion). This is because the extreme results may have been obtained in accordance with the present standard and/or be caused by the variability within, or in between, the laboratory samples.

Table C.BD.3 — Results of the interlaboratory comparison studies of the determination of Kjeldahl nitrogen in soil, sludge and treated biowaste. All concentrations in g/kg.

Matrix	Parameter	Mean	sr	sR	r	R	p	Outliers	Used number of data	Number of data reported below detection	Total no of data reported
Sludge 1	Kjeldahl	38.02	1.41%	4.36%	1.499	4.638	8	1	42	0	48
Sludge 2	Kjeldahl	35.27	1.24%	6.18%	1.221	6.100	9	1	48	0	54
Compost 1	Kjeldahl	16.22	2.15%	15.5%	0.978	7.043	9	2	42	0	54
Compost 2	Kjeldahl	12.68	4.50%	16.4%	1.597	5.839	10	1	54	0	58
Soil 4	Kjeldahl	1.62	7.19%	24.3%	0.327	1.107	11	0	44	0	44
Soil 5	Kjeldahl	1.83	4.42%	16.6%	0.226	0.852	9	1	46	0	52

Abbreviations: sr Repeatability standard deviation; SR Reproducibility standard deviation; r Repeatability limit (comparing two measurements); R Reproducibility limit (comparing two measurements); p Number of labs.

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ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility for a Standard test method by inter-laboratory tests*.

ISO 11261, *Soil quality — Determination of total nitrogen — Modified Kjeldahl method.*

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