BS EN 16320:2013

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Fertilizers — Determination of trace elements — Determination of mercury by vapour generation (VG) after aqua regia dissolution

National foreword

This British Standard is the UK implementation of EN 16320:2013. It supersedes PD CEN/TS 16320:2012 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilisers and related chemicals.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Düngemittel - Bestimmung von Elementspuren - Bestimmung von Quecksilber mit Verdampfungstechnik (VG) nach Königswasseraufschluss

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Contents

Page

Foreword

This document (EN 16320:2013) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2014, and conflicting national standards shall be withdrawn at the latest by April 2014.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 16320:2012.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

The following changes have been made to the former edition:

a) the CEN Technical Specification has been adopted as a European Standard;

b) the document has been editorially revised.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of the content of mercury in fertilizers after extraction with aqua regia and the detection of mercury by vapour generation (VG) coupled to an atomic absorption spectrometer or an inductively coupled plasma-atomic emission spectrometer. A limit of quantification of 0,01 mg/kg is to be expected.

2 Normative references

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

EN 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999 and EN 12944-2:1999 apply.

4 Principle

Mercury is extracted from the sample with aqua regia and conventional boiling. The concentration of mercury in the extract is measured by (cold) vapour generation (VG) coupled to a suitable detector, such as an atomic absorption spectrometer (AAS) or an inductively coupled plasma-atomic emission spectrometer (ICP-AES).

5 Sampling and sample preparation

Sampling is not part of the methods specified in this European Standard. A recommended sampling method is given in EN 1482-1.

Sample preparation shall be carried out in accordance with EN 1482-2.

6 Reagents

Use only reagents of recognised analytical grade.

Commercially available stock solutions shall be replaced according to the specifications from the supplier or after one year if prepared in the laboratory from available salts. Standard solutions shall be renewed monthly as a general rule.

6.1 **Water**, conforming to grade 2 of EN ISO 3696.

6.2 **Hydrochloric acid**, $c(\text{HCl})$ = 12 mol/l; 37 % volume fraction; $\rho \approx$ 1,18 g/ml.

6.3 **Nitric acid**, $c(\text{HNO}_3)$ = 16 mol/l; not less than 65 % volume fraction, $\rho \approx$ 1,42 g/ml.

6.4 Mixed acid solution of 0,8 mol/l nitric acid and 1,8 mol/l hydrochloric acid.

Mix 150 ml of hydrochloric acid (6.2) and 50 ml nitric acid (6.3) to 1,0 l of water (6.1).

6.5 Standard stock solution, mercury standard stock solution, e.g. ρ = 1 000 mg/l.

Use suitable stock solutions. Single-element stock solutions with adequate specification stating the acid used and the preparation technique are commercially available. It is recommended to use commercially available standard stock solutions for mercury.

6.6 Working standard solutions.

Depending on the scope, different working standard solutions may be necessary. In general, the stability of mercury working standard solutions should be checked.

6.6.1 Working standard solution I, ρ = 10 mg/l for mercury.

Dilute 1,00 ml of the standard stock solution for mercury (6.5) to 100,0 ml with the mixed acid solution (6.4). This solution is used to prepare 200 µg/l mercury working standard solution.

6.6.2 Working standard solution II, ρ = 200 µg/l for mercury.

Dilute 2,00 ml of the 10 mg/l mercury working standard solution I (6.6.1) to 100,0 ml with the mixed acid solution (6.4). This solution is used to prepare spiked test solutions and calibration solutions.

6.7 Reducing agents.

6.7.1 General.

Tin(II) chloride or sodium borohydride may be used as the reducing agent, but it is not advisable to use the two reagents alternately. Observe the instructions of the manufacturers of the apparatus. The concentration by mass of the reducing agent solutions may be varied to suit the system, and the relevant information provided by the manufacturer of the apparatus shall be observed.

6.7.2 Tin(II) chloride solution, $\rho(SnCl_2 \cdot 2H_2O)$ = 100 g/l.

Dissolve 50 g of tin(II) chloride in approximately 100 ml of hydrochloric acid (6.2) in a 500 ml volumetric flask and dilute to the mark. Prepare a fresh solution daily.

6.7.3 Sodium borohydride solution, e.g. ρ = 2 g/l.

Dissolve 2 g of sodium hydroxide pellets in water, add 2 g of sodium borohydride and dilute to 1 000 ml with water (6.1). Prepare a fresh solution daily and, when necessary, filter before use. When the analysis procedure is of a longer time, it is recommended to cool the sodium borohydride solution (i.e. with ice around the flask) during its use in the vapour generation ICP-AES or AAS measurement.

WARNING — It is essential to observe the safety instructions for working with sodium borohydride. Sodium borohydride forms hydrogen with acids and this can result in an explosive air/hydrogen mixture. A permanent extraction system shall be provided at the point where measurements are carried out.

7 Apparatus

7.1 Common laboratory glassware.

7.2 Analytical balance, capable of weighing to an accuracy of 1 mg.

7.3 Inductively coupled plasma-atomic emission spectrometer, with axial or radial viewing of the plasma and with suitable background correction.

The settings of the working conditions (e.g. gas flows, RF or plasma power, sample uptake rate, integration time, number of replicates) shall be optimised according the manufacturer's instructions. Radial viewing of the plasma may be used if it can be shown that the limit of quantification for mercury is below the required legal limit value.

7.4 Atomic absorption spectrometer, equipped with a heated quartz cuvette or a mercury absorption cell, or optionally with an amalgamation system.

7.5 Vapour generation equipment.

7.5.1 Continuous flow or flow-injection cold-vapour system.

7.5.2 Element-specific lamp for mercury.

7.6 Dilutor.

Instrument used for automated volumetric dilutions or other appropriate equipment (e.g. pipettes and volumetric glassware) to perform dilutions. The precision and accuracy of this type of equipment for volumetric dilutions shall be established, and controlled and documented regularly.

7.7 Ash-free filter paper, i.e. Whatman 589/2®1) or equivalent quality.

8 Procedure

8.1 General

Calibrations by standard additions with several standards or by matrix matching are very powerful calibration techniques and can be used to accurately correct for matrix effects from easy-ionisable elements (multiplicative matrix effects). Additive matrix effects (i.e. spectral interferences) are not corrected for with standard additions calibration. For matrix matching, additive matrix effects will be corrected for when the added matrix is the cause of the matrix effect. The main drawback of calibration by standard addition with several standards is the requirement for a calibration function for each sample type, which is a time consuming process. For matrix matching, a profound knowledge of the sample matrix is needed, which is not always necessarily available. These two techniques may thus not be practical to use in routine fertilizer laboratories.

It is therefore suggested that calibrations are to be performed by means of external calibration and correction of matrix effects by addition of one known spike of a standard solution (spike recovery). The method of external calibration and correction for spike recovery allows for the analysis of fertilizers with unknown matrix composition or with a matrix that cannot be easily imitated synthetically. This calibration technique may not be as precise as calibration by standard additions with several standards but the increased uncertainty is small compared to the total uncertainty of the method, if the total analyte concentration is in the linear working range after the spike and the added spike corresponds to at least a doubling of the analyte concentration. Many matrix errors can be compensated for by this procedure, if they are not additive (e.g. spectral interferences). Aliquots of the sample solution are analysed by the means of external calibration and then one aliquot is spiked with known concentrations of the analytes without changing the matrix of the sample solution. The calculated spike recovery is then used to correct the concentration calculated from the external calibration function. The concentration of the spikes shall be in the linear working range of the analytical detection technique used.

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1) Whatman 589/2® is an example of a suitable product available commercially. This Information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

8.2 Preparation of the test solution

8.2.1 General

The following extraction procedure leads, in most cases for mineral fertilizers, to trace element results which correspond to the total contents of these elements.

Calibration with several standard additions and external calibration after matrix matching may also be used without any problems.

8.2.2 Preparation

8.2.2.1 Weigh (3 ± 0,003) g of the prepared sample and transfer to a suitable reaction vessel (action 1).

8.2.2.2 Moisten the sample with about 0,5 ml to 1,0 ml of water (6.1) and add, whilst mixing, (21 ± 0,1) ml of hydrochloric acid (6.2) followed by (7 ± 0,1) ml of nitric acid (6.3), drop by drop if necessary to reduce foaming. Connect a condenser to the reaction vessel and let the mixture stand at room temperature until any effervescence almost ceases to allow for slow oxidation of any organic mass in the sample (action 2).

8.2.2.3 Transfer to the heating device and raise the temperature of the reaction mixture slowly to reflux conditions. Maintain for 2 h, ensuring that the condensation zone is lower than 1/3 of the height of the condenser, then allow to cool. Rinse the condenser with further with 10 ml of water (6.1) (action 3).

If the digested sample contains particulates which can clog nebulisers or interfere with the injection of the sample, the sample should be centrifuged and allowed to settle, or filtered before transferring into a suitable sized volumetric flask. For example, the solution should be allowed to pass through the filter paper and the insoluble residue washed onto the filter paper with a minimum of water (6.1). The method used shall be reported in the test report. Filter paper may cause contaminations (e.g. lead) and it may be necessary therefore to use ash-free filter paper (7.7).

8.2.2.4 Transfer the digested sample into a 150 ml volumetric flask and dilute to the mark with water (6.1). This yields an acid concentration approximately equal to that of the mixed acid solution (6.4). This test solution corresponds to a 50 times dilution of the solid sample (action 4).

8.2.2.5 Test solutions are diluted with the mixed acid solution (6.4) using a dilutor (7.6) to obtain a concentration of mercury between 0,2 µg/l and 10 µg/l (action 5).

NOTE 1 It is important that the total dilution of the test solution is equal to the dilution of the spiked test solution (see 8.3 on how to prepare the spiked test solution).

NOTE 2 The concentrations in the solutions in 8.2.2.5, action 5 are chosen so that they are above the typical limits of quantification, and that the concentrations fall within the linear working range of the analytical technique.

8.3 Preparation of the test solution for the correction of matrix effects by spike recovery

For each test solution analysed, a spiked test solution with a known addition of a standard solution is required to correct for matrix effects by correcting for the spike recovery measured under close to identical measurement conditions. The addition of a spike of the standard solution shall increase the analyte concentrations by at least 100 % without changing the matrix of the test solution (from 8.2.2.4, action 4) or the diluted test solution (from 8.2.2.5, action 5). See list entries a) and b) below for suggestions on how to spike a diluted and an undiluted test solution respectively when determining mercury.

a) If the test solution (from 8.2.2.4, action 4 or 8.2.2.5, action 5) contains 2,00 µg/l to10 µg/l of mercury, a spike addition corresponding to 2,00 µg/l of mercury is done while diluting the sample 5 times. Thus, take 2,00 ml of test solution and add 0,10 ml of the 200 µg/l mercury working standard solution II (6.6.2) and 7,90 ml mixed acid solution (6.4). The test solution (from 8.2.2.4, action 4 or 8.2.2.5, action 5) shall also be 5 times diluted with the mixed acid solution (6.4) prior to analysis by vapour generation AAS/ICP-AES.

b) If the test solution (from 8.2.2.4, action 4) contains 0,20 µg/l to 2,00 µg/l mercury it should be analysed without further dilution. Add 0,10 ml of the 200 µg/l mercury working standard solution II (6.6.2) to 9,90 ml test solution corresponding to addition of 2,00 µg/l mercury, thus preparing a spiked test solution of 10,00 ml without significant dilution or change in the matrix of the test solution. The test solution (from 8.2.2.4, action 4) is measured using the same dilution (9,90 ml test solution and 0,10 ml mixed acid solution (6.4)) by vapour generation AAS/ICP-AES.

8.4 Preparation of the blank test solution

Carry out a blank test at the same time as the extraction with only the reagents and follow the same procedure as for the samples. The blank test solutions should be analysed without further dilution to achieve the best possible detection capability. Contaminations from mercury in the mixed acid solution used for further dilutions of the sample test solutions should be checked before each analysis (i.e. by observing the corresponding analyte signals in the calibration blank solutions or acid blanks).

8.5 Preparation of the calibration solutions

8.5.1 Calibration solutions for the analysis of mercury

Prepare the calibration solutions by dilution of the working standard solution II (6.6.2) and calibration standards with the mixed acid solution (6.4).

8.5.2 Calibration standards

A suitable range of calibration standards covering the linear range of the calibration should be selected. Suggested calibration standards are:

Hg: 0 µg/l, 1 µg/l, 2 µg/l, 5 µg/l and 10 µg/l.

8.6 Determination of mercury

8.6.1 General

Set up the instrument according to the recommendations of the manufacturer.

For each instrument used, selectivity, limits of detection and quantification, precision, linear working range, and interferences shall be established separately.

8.6.2 Determination by VG-AAS

The test and calibration solutions are measured directly with an atomic absorption spectrometer with electrically heated quartz cuvette or a dedicated mercury absorption cell coupled to a suitable vapour generation system (e.g. continuous flow or flow-injection hydride-system).

The vapour generation system should be programmed in such a way that first the sample loop is filled with the test or calibration solution. The test- or calibration solution is then transferred to a mixing unit with the mixed acid solution (6.4) and mixed with sodium borohydride solution (6.7.3) or with tin(II) chloride solution (6.7.2). The resulting gas/liquid mixture is separated by an argon-flowed separator. The argon steam sorts out the mercury to the absorption cell for measurement of the atomic absorption of mercury.

Optionally, an amalgamation system could be used to increase sensitivity and to decrease matrix effects.

The calibration solutions are measured first, followed by the blank solution, test solution and lastly the spiked test solution. Check the linear range of the calibration function. If the concentration of the test solution is outside the linear range, dilute the test solution with the mixed acid solution (6.4). When carrying out prolonged series of measurements, it is advisable to check the zero and the calibration after every ten solutions.

Although background correction is seldom necessary in the case of the cold-vapour technique for analysing mercury, nevertheless this shall be checked for every type of sample. As an analytical control, reference sample(s) having reliable known mercury contents could be analysed in parallel with all the samples analysed, with the reference sample(s) being subjected to all the steps in the method starting from the digestion.

8.6.3 Spectrometer settings of VG-AAS

To devise a test schedule, first adjust the apparatus as specified in the operating manual of the manufacturer. Then, optimise the settings, paying particular attention to gas flow times and the amounts of tin(II) chloride or sodium borohydride introduced. Typical settings are given in Table 1.

Table 1 — Typical settings of VG-AAS for measuring mercury

Parameter	Typical settings
Temperature of the absorption cell	20 °C to100 °C
Wavelength	253,7 nm
Width length	0,7 nm
Signal processing	Signal height or signal area
Smoothing	0,5 s
Integration time	15 s
Working range	0,2 µg/l to 10 µg/l
Main interferences	As, Cu
Typical limit of detection	0,05 µg/l

8.6.4 Determination by VG-ICP-AES

The test and calibration solutions are measured directly with an ICP-AES coupled to a suitable vapour generation system (e.g. continuous flow or flow-injection hydride-system).

The vapour generation system should be programmed in such a way that first the sample loop is filled with the test or calibration solution. The test- or calibration solution is then transferred to a mixing unit with the mixed acid solution (6.4) and mixed with sodium borohydride solution (6.7.3) or with tin(II) chloride solution (6.7.2). The resulting gas/liquid mixture is separated by an argon-flowed separator. The argon stream sorts out the mercury to the ICP-AES for the measurement of the atomic emission of mercury.

The calibration solutions are measured first, followed by the blank solution, test solution and lastly the spiked test solution. Check the linear range of the calibration function. If the concentration of the test solution is outside the linear range, dilute the test solution with the mixed acid solution (6.4). When carrying out prolonged series of measurements, it is advisable to check the zero and the calibration after every ten solutions.

As an analytical control, reference sample(s) having reliable known mercury contents may be analyzed in parallel with all the samples analyzed, with the reference sample(s) being subjected to all the steps in the method starting from the digestion.

The emission line given in Table 2 is a recommendation which has to be checked for each individual instrument, considering the performance and working parameters of the instrument. Other emission lines may be used if the suitability for analyzing mercury in fertilizers has been validated.

Table 2 — Typical settings of VG-ICP-AES for measuring mercury

Parameter	Typical settings
Wavelength	253,65 nm
Background correction	Yes
Working range	0,2 µg/l to 10 µg/l
Main interferences	As, Cu
Typical limit of detection	0,05 µg/l

9 Calculation and expression of the results

9.1 External calibration

NOTE Net signal is defined as the number of counts per time unit at the selected wavelength, corrected for background contributions.

In the case of a linear calibration curve constructed with one blank calibration solution and one calibration solution, the calibration function can be described according to Formula (1):

$$S_{\text{st}} = c_{\text{st}} \times b + a \tag{1}$$

where

a is the intersection of the calibration curve;

b is the slope of the calibration curve;

S_{st} is the net signal of the calibration solution;

c_{st} is the concentration of the calibration solution in milligrams per litre or in micrograms per litre.

Calculate the analyte concentration, c_{f}, in the filtrate of the test portion using the slope, b, and the intersection, a, according to Formula (2):

$$c_{\text{f}} = \frac{S_{\text{f}} - a}{b} \tag{2}$$

where

S_{f} is the net signal of the test solution.

9.2 Correction for spike recovery

Calculate the analyte concentration, c_{fs}, in the spiked test portion from Formula (2). Then calculate the spike recovery, R_{s}, in percent from the analyte concentrations in the filtrate of the test portion, c_{f}, according to Formula (3):

$$R_{\text{s}} = \frac{c_{\text{fs}} - c_{\text{f}}}{\left(\dfrac{c_{\text{s}} \times V_{\text{s}}}{V_{\text{fst}}}\right)} \times 100 \tag{3}$$

where

c_s is the actual concentration of the standard solution, in milligrams per litre or in micrograms per litre;

V_s is the volume, in litres, of the standard solution used for spiking;

V_{fst} is the total volume, in litres, used to prepare the spiked test solution.

Correct the concentration of the analyte in the filtrate, c_f, from external calibration for the recovery to yield the corrected analyte concentration of the filtrate, $c_{f(R)}$, according to Formula (4):

$$c_{f(R)} = \frac{c_f}{R_s} \times 100 \qquad (4)$$

9.3 Standard addition method

In the case of several standard additions, use regression techniques on the linear model of variable y as a function of variable x to determine the elemental concentration of the test solution. Generally, this model can be indicated according to Formula (5):

$$y_i = a + b \times x_i \qquad (5)$$

In the particular case of three standard additions,

$$y_i = S_i \quad \text{(for } i = 0, 1, 2, 3\text{), and} \qquad (6)$$

$$x_i = c_s \times V_i \quad \text{(for } i = 0, 1, 2, 3\text{)} \qquad (7)$$

where

c_s is the concentration of the standard solution, in milligrams per litre or in micrograms per litre;

V_i is the various volumes, in litres, of the standard solution added;

S_i is the net signals after the various additions.

Calculate then the values of b and a according to Formula (8) and Formula (9):

$$b = \frac{n \times \sum x_i y_i - \sum x_i \sum y_i}{n \times \sum x_i^2 - \left(\sum x_i\right)^2} \qquad (8)$$

$$a = \frac{\sum y_i - b \times \sum x_i}{n} \qquad (9)$$

where

n is the number of solutions measured (n = 4 in the case of three additions).

Calculate the analyte concentration, c_f, (in mg/l or µg/l) of the filtrate of the test portion, according to Formula (10):

$$c_f = \frac{a/b}{V_f} \qquad (10)$$

where

V_f is the volume, in litres, of the filtrate of the test portion used to prepare the test solution.

9.4 Calculation of the element content in the sample

Calculate the analyte concentration in the sample or mass fraction of the analyte, w_E, expressed in milligrams of analyte per kilogram of fertilizer, according to Formula (11):

$$w_E = \frac{(c_f - c_{bl})}{m} \times V_t \quad (11)$$

where

c_f is the concentration, in milligrams per litre, of the filtrate of the test portion, as determined using Formula (2), Formula (4) or Formula (10) depending on the choice of calibration. $c_{f(R)}$ is used instead of c_f when using spike recovery (9.2);

c_{bl} is the concentration, in milligrams per litre, of the blank solution;

m is the mass of the sample, in kilograms, taken for the extraction, and corrected for water content;

V_t is the total volume, in litres, of extract (filtrate of the test portion).

10 Precision

10.1 Inter laboratory tests

Details of inter laboratory tests on the precision of the method are summarised in Annex A. Repeatability and reproducibility were calculated according to ISO 5725-1 and ISO 5725-2.

The values derived from this test may not be applicable to concentration ranges and matrices other than those given.

10.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in no more than 5 % of the cases be greater than the repeatability limit r given in Table 3.

10.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in no more than 5 % of the cases be greater than the reproducibility limit R given in Table 3.

Table 3 — Mean values, repeatability and reproducibility limits

Sample	$\bar{x}$ mg/kg	r mg/kg	R mg/kg
Triple Super Phosphate (TSP)	0,025 2	0,001 4	0,018 4
Rock phosphate	0,091 9	0,002 1	0,029 0

11 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) test method used with reference to this document (EN 16320);

c) test results obtained;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been fulfilled;

g) all operating details not specified in this document, or regarded as optional, together with details of any incidents occurred when performing the method, which might have influenced the test result(s).

Annex A
(informative)

Results of the inter-laboratory test

The precision of the method has been determined in the year 2009 in an inter-laboratory trial. The statistical results are given in Table A.1.

Table A.1 — Statistical results for mercury

Parameter	**TSP**	**Rock phosphate**
Number of laboratories	11	10
Number of laboratories retained after elimination of outliers	8	8
Number of outliers	3	2
Mean value, $\bar{x}$ (mg/kg)	0,025 2	0,091 9
Repeatability standard deviation s_r (mg/kg)	0,000 5	0,000 7
Relative repeatability standard deviation RSD_r (%)	2	0,8
Repeatability limit r [r = 2,8 × s_r] (mg/kg)	0,001 4	0,002 1
Reproducibility standard deviation s_R (mg/kg)	0,006 6	0,010 3
Relative reproducibility standard deviation RSD_R (%)	26	11,3
Reproducibility limit R [R = 2,8 × s_R] (mg/kg)	0,018 4	0,029 0

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[2] EN 1482-1, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*

[3] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[4] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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