



BSI Standards Publication

**Fertilizers — Determination
of trace elements —
Determination of arsenic by
inductively coupled plasma-
atomic emission spectrometry
(ICP-AES) after aqua regia
dissolution**

National foreword

This British Standard is the UK implementation of EN 16317:2013. It supersedes PD CEN/TS 16317:2012 which is withdrawn.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.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2013. Published by BSI Standards Limited 2013

ISBN 978 0 580 81509 6

ICS 65.080

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 October 2013.

Amendments issued since publication

Date	Text affected
------	---------------

EUROPEAN STANDARD

EN 16317

NORME EUROPÉENNE

EUROPÄISCHE NORM

October 2013

ICS 65.080

Supersedes CEN/TS 16317:2012

English Version

Fertilizers - Determination of trace elements - Determination of arsenic by inductively coupled plasma-atomic emission spectrometry (ICP-AES) after aqua regia dissolution

Engrais - Dosage des éléments traces - Détermination de l'arsenic par spectrométrie d'émission atomique avec plasma induit par haute fréquence (ICP-AES) après digestion à l'eau régale

Düngemittel - Bestimmung von Elementspuren - Bestimmung von Arsen mit Atomemissionsspektrometrie mit induktiv gekoppeltem Plasma (ICP-AES) nach Königswasseraufschluss

This European Standard was approved by CEN on 29 August 2013.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of 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 United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

Contents

Page

Foreword.....	3
1 Scope.....	4
2 Normative references.....	4
3 Terms and definitions	4
4 Principle.....	4
5 Sampling and sample preparation	4
6 Reagents.....	4
7 Apparatus	5
8 Procedure	6
8.1 General	6
8.2 Preparation of the test solution	6
8.2.1 General	6
8.2.2 Preparation.....	6
8.3 Preparation of the test solution for the correction of matrix effects by spike recovery	7
8.4 Preparation of the blank test solution.....	7
8.5 Preparation of the calibration solutions for the analysis of arsenic.....	8
8.6 Determination of arsenic by ICP-AES	8
8.6.1 General	8
8.6.2 Determination by ICP-AES.....	8
9 Calculation and expression of the results	9
9.1 External calibration.....	9
9.2 Correction for spike recovery.....	9
9.3 Standard addition method	10
9.4 Calculation of the element content in the sample	11
10 Precision.....	11
10.1 Inter laboratory tests.....	11
10.2 Repeatability.....	11
10.3 Reproducibility.....	11
11 Test report	12
Annex A (informative) Results of the inter-laboratory test.....	13
A.1 Inter-laboratory tests	13
A.2 Statistical results for the determination of arsenic by ICP-AES	13
Bibliography.....	14

Foreword

This document (EN 16317:2013) 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 16317:2012.

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.

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

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 arsenic in fertilizers using inductively coupled plasma-atomic emission spectrometry (ICP-AES) after aqua regia dissolution. Limits of quantification are dependent on the sample matrix as well as on the instrument, but can roughly be expected to be 1,5 mg/kg for As.

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

Arsenic is extracted from the sample with aqua regia and conventional boiling. The concentration in the extract is measured by inductively coupled plasma-atomic emission spectrometry (ICP-AES) with axial or radial viewing.

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, according to EN ISO 3696, grade 2.

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

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

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 arsenic standard stock solution, e.g. $\rho = 1\ 000$ mg/l in arsenic.

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 a commercially available standard stock solution for arsenic.

6.6 Working standard solutions.

Depending on the scope, different working standard solutions may be necessary.

6.6.1 Working standard solution I, $\rho = 100$ mg/l for arsenic.

Dilute 10,0 ml of the stock solution of arsenic (6.5) to 100,0 ml with the mixed acid solution (6.4) in the same 100 ml flask. This solution is used to prepare spiked test solutions and standard and calibration solutions.

6.6.2 Working standard solution II, $\rho = 10$ mg/l for arsenic.

Dilute 10,0 ml of the working standard solution I (6.6.1) to 100,0 ml with the mixed acid solution (6.4) in a 100 ml flask. This solution is used to prepare spiked test solutions and calibration solutions.

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 to the manufacturer's instructions. Radial viewing of the plasma may be used if it can be shown that the limit of quantification for arsenic is below the required legal limit values.

The use of axial orientation of the viewing optics requires good control of the matrix effects coming from "easily ionisable elements" (i.e. the influence of easily ionisable elements in varying concentrations on the signal intensities of the analytes). For alkali-elements, this can be achieved by adding caesium-chloride solution (CsCl). In general, matrix matching of calibration solutions or calibration by standard additions with several calibration standards will correct accurately for these matrix effects. Spike recovery of one known standard combined with external calibration can, if used properly, also correct sufficiently for matrix effects (see 8.1). Correction by internal standardisation is also a good option, but the accuracy of the measurements after internal standard correction should be validated properly prior to use on unknown fertilizer samples.

7.4 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.

7.5 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.

Correction by internal standardisation is also a good option, but the accuracy of the measurements after internal standard correction should be validated properly prior to use on unknown fertilizer samples.

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 ICP-AES.

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 or by correction for matrix effects with internal standardization may also be used.

8.2.2 Preparation

8.2.2.1 Weigh ($3 \pm 0,003$) 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 ($21 \pm 0,1$) ml of hydrochloric acid (6.2) followed by ($7 \pm 0,1$) 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 further with 10 ml of water (6.1) (action 3).

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.

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 then 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 (7.5).

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 Dilute the test solutions with the mixed acid solution (6.4) using a dilutor (7.4) to obtain a concentration of arsenic between 0,03 mg/l and 5 mg/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 concentration of arsenic in the solution in action 5 (8.2.2.5) is chosen so that it is above the typical limits of quantification, and that the concentration 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 arsenic.

a) If the test solution (from 8.2.2.4, action 4 or from 8.2.2.5, action 5) contains 1,00 mg/l to 5,00 mg/l of arsenic, a spike addition corresponding to 1,00 mg/l of a multi-element standard solution containing arsenic may be done while diluting the sample 5 times. Thus, take 2,00 ml of test solution and add 1,00 ml of the 10 mg/l working standard solution II (6.6.2) and 7,00 ml mixed acid solution (6.4). The test solution (from 8.2.2.4, action 4 or from 8.2.2.5, action 5) shall also be 5 times diluted with the mixed acid solution (6.4) prior to analysis by ICP-AES.

b) If the test solution (from 8.2.2.4, action 4) contains 0,03 mg/l to 1,00 mg/l arsenic it should be analysed without further dilution. Add 0,10 ml of a suitable standard solution (e.g. 100 mg/l working standard solution I (6.6.1) corresponding to an addition of 1,00 mg/l of arsenic) to 9,90 ml test solution, thus preparing a spiked test solution of 10,00 ml without changing the matrix of the test solution significantly. The test solution (from 8.2.2.4, action 4) is measured using the same dilution (9,90 ml test solution and 0,100 ml mixed acid solution (6.4)) by 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 best possible detection capability. Contaminations from arsenic 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 for the analysis of arsenic

Prepare the calibration solutions by dilution of suitable working standard solutions (6.6.1 and 6.6.2) and calibration standards with the mixed acid solution (6.4).

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

As: 0 mg/l, 0,05 mg/l, 0,1 mg/l, 0,25 mg/l, 1 mg/l, and 2 mg/l.

8.6 Determination of arsenic by ICP-AES

8.6.1 General

Set up the instrument according to the recommendations of the manufacturer and with a suitable background correction system in operation.

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 ICP-AES

Aspirate the calibration solutions, the blank test solution (8.4), the test solution (8.2) and the spiked test solution (8.3) separately into the plasma, and measure the emission of arsenic. Perform at least two replicate measurements for each solution. Average the values if the values fall within an acceptable range. After each measurement, wash with the mixed acid solution (6.4) (e.g. for 60 s). For prolonged analytical runs, the calibration should be checked (e.g. every 10th solution) by a suitable QC standard (e.g. a 0,50 mg/l standard solution containing arsenic or a digested control sample with a well-defined content of arsenic).

As an analytical control, reference sample(s) having a reliable known arsenic content 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.

The emission line given in Table 1 is a recommendation, which has to be checked for each individual instrument considering the performance and working parameters of the instrument. It is possible to use other emission lines if the suitability for analysing arsenic in fertilizers has been validated.

Table 1 — General analytical conditions for the determination of arsenic by ICP-AES

Analyte	Arsenic
Wavelength	193,76 nm
Background correction	Yes
Working range	0,03 mg/l to 10 mg/l
Main interferences	Cr
Typical limit of detection for axial viewing	0,01 mg/l

Spectral lines which are not interfered by other elements should be selected when the instrumental measuring protocol is set up. Lists of spectral interferences can be found in the scientific literature or in datasets delivered with the software of the emission spectrometer; or they shall be identified in practical trials using mixtures of standard solutions containing the elements typically contained in fertiliser samples in varying concentrations.

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):

$$S_{st} = c_{st} \times b + a \quad (1)$$

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.

Calculate the analyte concentration, c_f , in the filtrate of the test portion using the slope, b , and the intersection, a , given in Formula (1) according to Formula (2):

$$c_f = \frac{S_f - a}{b} \quad (2)$$

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) and 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):

$$R_s = \frac{c_{fs} - c_f}{\left(\frac{c_s \times V_s}{V_{fst}} \right)} \times 100 \quad (3)$$

where

c_s is the actual concentration of the standard solution, in milligrams 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):

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

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):

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

In this particular case of three standard additions,

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

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

where

c_s is the concentration of the standard solution, in milligrams 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 the values of b and a according to Formula (8) and Formula (9):

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

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

where

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

Calculate the analyte concentration, c_f , in milligrams per litre, of the filtrate of the test portion according to Formula (10):

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

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 2.

10.3 Reproducibility

The absolute difference between two independent 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 2.

Table 2 — Mean values, repeatability and reproducibility limits

Sample	\bar{x} mg/kg	r mg/kg	R mg/kg
Triple Super Phosphate (TSP)	17,9	2,6	8,5
Rock phosphate	6,0	1,9	4,3

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 16317);
- 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

A.1 Inter-laboratory tests

The precision of the method has been determined in the year 2010 in an inter-laboratory trial with 12 respectively 13 laboratories participating and carried out on two samples of fertilizers. The statistical results are given in Table A.1.

A.2 Statistical results for the determination of arsenic by ICP-AES

Table A.1 — Statistical results for arsenic

Parameter	TSP	Rock phosphate
Year of the test	2010	2010
Number of laboratories	12	13
Number of laboratories retained after elimination of outliers	10	13
Number of outliers	2	0
Mean value, \bar{x} (mg/kg)	17,9	6,0
Repeatability standard deviation s_r (mg/kg)	0,9	0,7
Relative repeatability standard deviation RSD_r (%)	5,0	11,0
Repeatability limit r [$r = 2,8 \times s_r$] (mg/kg)	2,6	1,9
Reproducibility standard deviation s_R (mg/kg)	3,0	1,5
Relative reproducibility standard deviation RSD_R (%)	17,0	26,0
Reproducibility limit R [$R = 2,8 \times s_R$] (mg/kg)	8,5	4,3

Bibliography

- [1] *Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers*, Official Journal L 304, 21/11/2003 P. 0001-0194 and amendments available from: <http://eur-lex.europa.eu/LexUriServ/LexUriServ.do?uri=OJ:L:2003:304:0001:0194:EN:PDF>
- [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*

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards-based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email bsmusales@bsigroup.com.

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Copyright

All the data, software and documentation set out in all British Standards and other BSI publications are the property of and copyrighted by BSI, or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI. Details and advice can be obtained from the Copyright & Licensing Department.

Useful Contacts:

Customer Services

Tel: +44 845 086 9001

Email (orders): orders@bsigroup.com

Email (enquiries): cservices@bsigroup.com

Subscriptions

Tel: +44 845 086 9001

Email: subscriptions@bsigroup.com

Knowledge Centre

Tel: +44 20 8996 7004

Email: knowledgecentre@bsigroup.com

Copyright & Licensing

Tel: +44 20 8996 7070

Email: copyright@bsigroup.com



...making excellence a habit.™