

BS EN 16197:2012



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Fertilizers — Determination of magnesium by atomic absorption spectrometry

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

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Fertilizers - Determination of magnesium by atomic absorption spectrometry

Engrais - Dosage du magnésium par spectrométrie
d'absorption atomiqueDüngemittel - Bestimmung von Magnesium mit
Atomabsorptionsspektrometrie

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Foreword

This document (EN 16197:2012) 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of an identical text or by endorsement, at the latest by May 2013, and conflicting national standards shall be withdrawn at the latest by May 2013.

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 16197:2011.

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.

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1 Scope

This European Standard specifies a method for the determination of the magnesium content in fertilizer extracts using atomic absorption spectrometry (AAS).

This method is applicable to EC fertilizer extracts obtained according to EN 15960 and EN 15961, for which a declaration of the total magnesium and/or water soluble magnesium content is required, with the exceptions of the following fertilizers according to [4], Annex I D relating to secondary nutrients:

- type 4 (kieserite);
- type 5 (magnesium sulfate) and type 5.1 (magnesium sulfate solution);

and with the exception of the following fertilizer listed in [4], Annex I A.3 relating to potassic fertilizers:

- type 7 (kieserite with potassium sulfate)

to which [4], method 8.8, applies.

NOTE Method 8.8 is covered by EN 16198 [2].

The method applies to all fertilizer extracts containing elements in quantities that might interfere with the complexometric determination of magnesium.

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 15960, *Fertilizers — Extraction of total calcium, total magnesium, total sodium and total sulfur in the forms of sulfates*

EN 15961, *Fertilizers — Extraction of water soluble calcium, magnesium, sodium and sulfur in the form of sulfates*

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

Determination of magnesium by atomic absorption spectrometry (AAS) after appropriate dilution of the extract.

5 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN 1482-1 [1].

Sample preparation shall be carried out in accordance with EN 1482-2. Grinding of the laboratory sample is recommended for homogeneity reasons.

6 Reagents

6.1 Hydrochloric acid solution, $c = 1 \text{ mol/l}$.

6.2 Hydrochloric acid solution, $c = 0,5 \text{ mol/l}$.

6.3 Standard solution of magnesium, $\rho = 1,00 \text{ mg/ml}$.

6.3.1 Preferably use a commercially available certified stock solution of magnesium.

6.3.2 Dissolve 1,013 g of magnesium sulfate ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) in hydrochloric acid solution (6.2), transfer quantitatively into a graduated flask (7.3) and make up the volume with the same acid and mix.

6.3.3 Weigh 1,658 g of magnesium oxide (MgO), previously calcined to remove all traces of carbonation.

Place in a beaker with 100 ml of water and 120 ml of hydrochloric acid (6.1). When it has dissolved, decant quantitatively into a graduated flask (7.5). Make up the volume by adding and mix.

The concentration of magnesium in the stock solution shall be checked on a regular basis.

6.4 Strontium chloride solution.

Dissolve 75 g of strontium chloride ($\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$) in a hydrochloric acid solution (6.2) and make up to 500 ml with the same acid solution.

7 Apparatus

7.1 Spectrometer, fitted for atomic absorption, with a magnesium lamp, set at 285,2 nm.

7.2 Air-acetylene flame.

7.3 100 ml graduated flask.

7.4 200 ml graduated flask.

7.5 1 000 ml graduated flask.

8 Preparation of the extraction solution

The extraction solutions shall be prepared according to EN 15960 or EN 15961.

9 Procedure

9.1 Preparation of the test solution

9.1.1 If the fertilizer has a declared magnesium (Mg) content of more than 6 % (i.e. 10 % as MgO), take 25 ml (V_1) of the extraction solution (see Clause 8). Transfer into a graduated flask (7.3), and make up to volume with water and mix. The dilution factor is $D_1 = 100/V_1$.

9.1.2 Using a pipette, take 10 ml of the extraction solution (see Clause 8) or the solution (9.1.1). Transfer into a graduated flask (7.4). Make up to volume with hydrochloric acid solution (6.2) and mix. The dilution factor is 200/10.

9.1.3 Dilute this solution (9.1.2) with hydrochloric acid solution (6.2) so as to obtain a concentration in the optimum working field of the spectrometer (7.1). V_2 is the volume of the sample in 100 ml. The dilution factor is $D_2 = 100/V_2$.

9.1.4 The final solution should contain 10 % (volume fraction) of the strontium chloride solution (6.4).

9.2 Preparation of the blank solution

Prepare a blank solution by repeating the whole procedure from the extraction according to EN 15960 or EN 15961, omitting only the test sample of fertilizer.

9.3 Preparation of the calibration solutions

By diluting the standard solution (6.3) with the hydrochloric acid (6.2), prepare at least five calibration solutions of increasing concentration within the optimum measuring range of the apparatus (7.1).

These solutions should contain 10 % (volume fraction) of the strontium chloride solution (6.4).

9.4 Measurement

Set up the atomic absorption spectrometer (7.1) according to the manufacturer's instructions at a wavelength of 285,2 nm.

Spray, successively, the calibration solutions (9.3), the sample solution (9.1) and the blank solution (9.2), washing the instrument through with the solution to be measured next. Repeat this operation three times. Plot the calibration curve using the mean absorbencies of each of the calibrations (9.3) as the ordinates and the corresponding mass concentration of magnesium in micrograms per millilitre as the abscissas. Determine the concentration of magnesium in the sample (9.1), X_s and blank (9.2), X_b , by reference to the calibration curve.

10 Calculation and expression of the result

Calculate the amount of magnesium (Mg) or magnesium oxide (MgO) in the sample by reference to the calibration solutions and taking into consideration the blank.

Calculate the magnesium content in the fertilizer, w_{Mg} , as a mass fraction in percent according to Formula (1).

$$w_{\text{Mg}} = \frac{(X_s - X_b)D_1(200/10)D_2 500 \times 100}{1000 \times 1000 M} \quad (1)$$

where

X_s is the concentration of the solution to be analysed recorded on the calibration curve, in micrograms per millilitre;

X_b is the concentration of the blank solution as recorded on the calibration curve, in micrograms per millilitre;

D_1 is the dilution factor when the solution is diluted (9.1.1):

— it is equal to 4 if 25 ml are taken;

— it is equal to 1 when the solution is not diluted;

D_2 is the dilution factor (9.1.3);

M is the mass, in grams, of the test sample at the time of extraction.

$$\text{MgO (\%)} = \text{Mg (\%)} / 0,6$$

11 Precision

11.1 Inter-laboratory test

Repeated inter-laboratory tests have been carried out in 2007 and 2009 with different numbers of participating laboratories and several different samples (see Table A.1 to Table A.4). Repeatability and reproducibility were calculated according to ISO 5725-2.

The values derived from these inter-laboratory tests may not be applicable to concentration ranges and matrices other than those given in Annex A.

11.2 Repeatability

The absolute difference between two independent single test results, obtained with 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 not more than 5 % of the cases exceed the values of r given in Table 1 and Table 2.

11.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of the cases exceed values of R given in Table 1 and Table 2.

Table 1 — Results ring test 2007

Sample	Extraction method	\bar{x} %	<i>r</i> %	<i>R</i> %
CAN	EN 15960	4,75	0,15	0,82
	EN 15961	1,83	0,25	1,18
KALI ROH	EN 15960	6,28	0,25	0,80
	EN 15961	6,05	0,20	0,83
NPK2:12-11-18+2+8	EN 15960	3,38	0,13	0,74
	EN 15961	3,04	0,13	0,62

Table 2 — Results ring test 2009

Sample	Extraction method	\bar{x} %	<i>r</i> %	<i>R</i> %
CAN	EN 15960	4,38	0,11	1,65
	EN 15961	2,38	0,30	3,23
NPK:12-12-17S+2	EN 15960	1,88	0,08	0,32
	EN 15961	1,58	0,08	0,52

12 Test report

The test report shall contain at least the following information:

- a) all information necessary for the complete identification of the sample;
- b) the test method used with reference to this document;
- c) the method of preparation of the extraction solution (EN 15960 or EN 15961);
- d) the test results obtained;
- e) date of sampling and sampling procedure (if known);
- f) date when the analysis was finished;
- g) whether the requirement of the repeatability limit has been fulfilled;
- h) 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)

Statistical results of the inter-laboratory test

The precision of the method was established in 2007 and in 2009 by Working Group 7 “Chemical analysis” of CEN/TC 260 “Fertilizers and liming materials” in several inter-laboratory tests evaluated in accordance with ISO 5725-2. The statistical results are given in Table A.1 to Table A.4.

Table A.1 — Statistical results of the inter-laboratory test in 2007 – Extraction method EN 15960

Parameter	Sample		
	CAN	KALI ROH	NPK2:12-11-18+2+8
Number of participating laboratories	13	14	14
Number of laboratories after elimination of outliers (accepted test results)	13	12	12
Mean value \bar{x} (%)	4,75	6,28	3,38
Repeatability standard deviation s_r (%)	0,05	0,09	0,05
RSD_r (%)	1,1	1,4	1,4
Repeatability limit r (%)	0,15	0,25	0,13
Reproducibility standard deviation s_R (%)	0,29	0,29	0,27
RSD_R (%)	6,2	4,6	7,9
Reproducibility limit R (%)	0,82	0,80	0,74

Table A.2 — Statistical results of the inter-laboratory test in 2007 – Extraction method EN 15961

Parameter	Sample		
	CAN	KALI ROH	NPK2:12-11-18+2+8
Number of participating laboratories	13	14	14
Number of laboratories after elimination of outliers (accepted test results)	12	14	14
Mean value \bar{x} (%)	1,83	6,05	3,04
Repeatability standard deviation s_r (%)	0,09	0,07	0,05
RSD_r (%)	5,0	1,2	1,6
Repeatability limit r (%)	0,25	0,20	0,13
Reproducibility standard deviation s_R (%)	0,42	0,30	0,22
RSD_R (%)	23,0	4,9	7,2
Reproducibility limit R (%)	1,18	0,83	0,62

Table A.3 — Statistical results of the inter-laboratory test in 2009 – Extraction method EN 15960

Parameter	Sample	
	CAN	NPK:12-12-17S+2
Number of participating laboratories	10	10
Number of laboratories after elimination of outliers (accepted test results)	8	10
Mean value \bar{x} (%)	4,38	1,88
Repeatability standard deviation s_r (%)	0,04	0,03
RSD_r (%)	0,9	1,5
Repeatability limit r (%)	0,11	0,08
Reproducibility standard deviation s_R (%)	0,59	0,12
RSD_R (%)	13,5	6,2
Reproducibility limit R (%)	1,65	0,32

Table A.4 — Statistical results of the inter-laboratory test in 2009 – Extraction method EN 15961

Parameter	Sample	
	CAN	NPK:12-12-17S+2
Number of participating laboratories	9	9
Number of laboratories after elimination of outliers (accepted test results)	8	9
Mean value \bar{x} (%)	2,38	1,58
Repeatability standard deviation s_r (%)	0,11	0,03
RSD_r (%)	4,0	1,8
Repeatability limit r (%)	0,30	0,08
Reproducibility standard deviation s_R (%)	1,15	0,18
RSD_R (%)	48,0	11,6
Reproducibility limit R (%)	3,23	0,52

Bibliography

- [1] EN 1482-1, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*
- [2] EN 16198, *Fertilizers — Determination of magnesium by complexometry*
- [3] 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*
- [4] *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, Annex I and Annex IV, method 8.7, method 8.8

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