





TECHNICAL SPECIFICATION  
SPÉCIFICATION TECHNIQUE  
TECHNISCHE SPEZIFIKATION

**FINAL DRAFT**  
**FprCEN/TS 16197**

November 2010

---

ICS

English Version

## Fertilizers - Determination of magnesium by atomic absorption spectrometry

Engrais - Dosage du magnésium par spectrométrie  
d'absorption atomique

Düngemittel - Bestimmung von Magnesium mit  
Atomabsorptionsspektrometrie

This draft Technical Specification is submitted to CEN members for formal vote. It has been drawn up by the Technical Committee CEN/TC 260.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

**Warning** : This document is not a Technical Specification. It is distributed for review and comments. It is subject to change without notice and shall not be referred to as a Technical Specification.



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

**Management Centre: Avenue Marnix 17, B-1000 Brussels**

<b>Contents</b>		Page
Foreword.....		3
1	Scope .....	4
2	Normative references .....	4
3	Terms and definitions .....	4
4	Principle.....	4
5	Sampling.....	5
6	Reagents.....	5
7	Apparatus .....	5
8	Preparation of the extraction solution.....	5
9	Procedure .....	6
10	Calculation and expression of the result .....	6
11	Precision.....	7
12	Test report .....	8
Annex A (informative) Statistical results of the inter-laboratory test .....		9
Bibliography .....		11

## Foreword

This document (FprCEN/TS 16197:2010) has been prepared by Technical Committee CEN/TC 260 "Fertilizers and liming materials", the secretariat of which is held by DIN.

This document is currently submitted to the Formal Vote.

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

## FprCEN/TS 16197:2010 (E)

### 1 Scope

This Technical Specification 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 CEN/TS 15960 and CEN/TS 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 [1] Annex I D relating to secondary nutrients:

- type 4 (kieserite);
- type 5 (magnesium sulfate) and type 5.1 (magnesium sulfate solution);
- type 7 (kieserite with potassium sulfate)

to which [1], method 8.8, applies.

NOTE Method 8.8 is covered by prCEN/TS 16198 (see Bibliography).

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 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 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*

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

CEN/TS 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.

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,  $c = 1$  mol/l.**

**6.2 Hydrochloric acid solution,  $c = 0,5$  mol/l.**

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

**6.3.1** Dissolve 1,013 g of magnesium sulfate ( $\text{MgSO}_4, 7\text{H}_2\text{O}$ ) in hydrochloric acid solution (6.2).

**6.3.2** Weigh 1,658 g of magnesium oxide ( $\text{MgO}$ ), 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, or take a commercial standard solution.

The laboratory is responsible for testing such solutions.

**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 CEN/TS 15960 or CEN/TS 15961.

## FprCEN/TS 16197:2010 (E)

### 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 CEN/TS 15960 or CEN/TS 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 spectrometer (7.1) 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 Equation (1).

$$w_{Mg} = \frac{(X_s - X_b)(200/10)D_2 500.100}{1\ 000.1\ 000\ M} \quad (1)$$

where

$X_s$  is the concentration of the solution to be analyzed 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 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	$\bar{x}$ %	$r$ %	$R$ %
CAN	CEN/TS 15960	4,75	0,15	0,82
	CEN/TS 15961	1,83	0,25	1,18
KALI ROH	CEN/TS 15960	6,28	0,25	0,80
	CEN/TS 15961	6,05	0,20	0,83
NPK2:12-11-18+2+8	CEN/TS 15960	3,38	0,13	0,74
	CEN/TS 15961	3,04	0,13	0,62