



**SLOVENSKI STANDARD**  
**kSIST-TS FprCEN/TS 16195:2011**  
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**Gnojila - Določevanje kloridov v odsotnosti organske snovi**

Fertilizers - Determination of chlorides in the absence of organic material

Düngemittel - Bestimmung von Chlorid bei Abwesenheit organischer Stoffe

Engrais - Dosage des chlorures en l'absence de matières organiques

**Ta slovenski standard je istoveten z: FprCEN/TS 16195**

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65.080            Gnojila    Fertilizers

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English Version

## Fertilizers - Determination of chlorides in the absence of organic material

Engrais - Dosage des chlorures en l'absence de matières organiques

Düngemittel - Bestimmung von Chlorid bei Abwesenheit organischer Stoffe

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

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

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

## Foreword

This document (FprCEN/TS 16195:2010) 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 16195:2010 (E)

### 1 Scope

This European Standard specifies a method for the determination of chlorides in the absence of organic material. The method is applicable to all fertilizers which are free from organic material.

### 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*

### 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

The chlorides, dissolved in water, are precipitated in an acid medium by an excess of standard solution of silver nitrate. The excess is titrated with a solution of ammonium thiocyanate in the presence of ferric ammonium sulfate (Volhard's method).

### 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

Use only reagents of recognized analytical grade.

**6.1 Water**, distilled or demineralized and free from chlorides.

**6.2 Nitrobenzene or diethyl ether**.

**6.3 Nitric acid**,  $c = 10 \text{ mol/l}$ .

#### 6.4 Indicator solution.

Dissolve 40 g of ferric ammonium sulfate  $\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$ , in water and make up to 1 l.

#### 6.5 Silver nitrate standard solution, $c = 0,1 \text{ mol/l}$ .

Preparation: Since this salt is hygroscopic and cannot be dried without risk of decomposition, it is advisable to weigh out approximately 9 g, dissolve in water and make up the volume to 1 l. Adjust to  $c = 0,1 \text{ mol/l}$  substance concentration by titration of  $\text{AgNO}_3$   $0,1 \text{ mol/l}$ .

#### 6.6 Diluted hydrochloric acid, one volume of $\text{HCl}$ , $\rho = 1,18 \text{ g/ml}$ plus one volume of water.

#### 6.7 Ammonium thiocyanate, standard solution, $c = 0,1 \text{ mol/l}$ .

### 7 Apparatus

#### 7.1 Standard laboratory equipment.

#### 7.2 Rotary shaker, 35 to 40 turns per minute.

#### 7.3 Burettes.

#### 7.4 Graduated flask, capacity 500 ml.

#### 7.5 Conical (Erlenmeyer) flask, capacity 250 ml.

### 8 Procedure

#### 8.1 Preparation of the test portion and the solution

Place 5 g of the laboratory sample, weighed to the nearest 0,001 g, in a graduated flask (7.4) and add 450 ml of water (6.1). Mix for 0,5 h on the shaker (7.2); make up to 500 ml with water (6.1); mix and filter into a beaker.

#### 8.2 Determination

Take an aliquot part of the test solution containing not more than 0,150 g of chloride: for example 25 ml (0,25 g), 50 ml (0,5 g) or 100 ml (1 g). If the amount of the test solution taken is smaller than 50 ml it is necessary to make up the volume to 50 ml with water (6.1).

Add 5 ml of nitric acid (6.3), 20 ml of the indicator solution (6.4), and two drops of ammonium thiocyanate standard solution (6.7) (a sample of this latter reagent is taken with a burette adjusted to zero for this purpose).

With a burette (7.3) then add silver nitrate standard solution (6.5) until there is an excess of 2 ml to 5 ml. Add 5 ml of nitrobenzene or 5 ml of diethyl ether (6.2) and shake well to agglomerate the precipitate. Titrate the excess silver nitrate with ammonium thiocyanate (6.7) until a red-brown colour appears which remains after the flask has been shaken slightly.

NOTE Nitrobenzene or diethyl ether (but above all nitrobenzene) prevents the silver chloride from reacting with thiocyanate ions. Thus a clear colour change is obtained.

## FprCEN/TS 16195:2010 (E)

### 8.3 Blank test

Carry out a blank test (omitting the test portion) under the same conditions and allow for it when calculating the final result.

### 8.4 Control test

Before carrying out the estimations check the accuracy of the method by using an aliquot part of a freshly prepared solution of potassium chloride, such that this part contains a known quantity in the order of 100 mg of chloride.

## 9 Calculation and expression of the results

Express the result of the analysis as a percentage of chloride contained in the sample as it has been received for analysis.

Calculate the mass fraction of chlorides (Cl),  $w_{\text{Cl}}$ , in percent according to Equation (1).

$$w_{\text{Cl}} = 0,003\,546 \times \frac{(V_Z - V_{\text{CZ}}) - (V_a - V_{\text{ca}})}{M} \times 100 \quad (1)$$

where

$V_Z$  is the amount of silver nitrate 0,1 mol/l, in millilitres;

$V_{\text{CZ}}$  is the amount of silver nitrate 0,1 mol/l, used in the blank test, in millilitres;

$V_a$  is the amount of ammonium thiocyanate 0,1 mol/l, in millilitres;

$V_{\text{ca}}$  is the amount of ammonium thiocyanate 0,1 mol/l, used in the blank test, in millilitres;

$M$  is the mass, in grams, of the test portion taken (8.1).

## 10 Precision

### 10.1 Inter-laboratory test

An inter-laboratory test has been carried out in 2009 with 14/13 participating laboratories and two different samples. The 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.

### 10.2 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.

### 10.3 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.



**Table 1 — Mean values, repeatability and reproducibility limits**

<b>Sample</b>	$\bar{x}$ %	<i>r</i> %	<i>R</i> %
Patent kali	2,781	0,066	0,284
NPK2 (12-11-18+4+8)	0,478	0,076	0,312

## 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) the test method used with reference to this document;
- c) the 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).