



SLOVENSKI STANDARD

SIST EN 14787:2005

01-december-2005

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Fertilizers and liming materials - Determination of water content - Guidelines and recommendations

Düngemittel und Calcium-/Magnesium-Bodenverbesserungsmittel - Bestimmung des Wassergehaltes - Leitlinien und Empfehlungen

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Engrais et amendements minéraux basiques - Détermination de la teneur en eau - Guide et recommandations

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Ta slovenski standard je istoveten z: EN 14787:2005

ICS:

65.080

Gnojila

Fertilizers

SIST EN 14787:2005

en,fr,de

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EUROPEAN STANDARD

EN 14787

NORME EUROPÉENNE

EUROPÄISCHE NORM

May 2005

ICS 65.080

English version

Fertilizers and liming materials - Determination of water content - Guidelines and recommendations

Engrais et amendements minéraux basiques -
Détermination de la teneur en eau - Guide et
recommandations

Düngemittel und Calcium-/Magnesium-
Bodenverbesserungsmittel - Bestimmung des
Wassergehaltes - Leitlinien und Empfehlungen

This European Standard was approved by CEN on 21 March 2005.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

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

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Foreword

This document (EN 14787:2005) 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 November 2005, and conflicting national standards shall be withdrawn at the latest by November 2005.

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

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Introduction

There are, at present, four standard methods (EN 12048, EN 12049, EN 13466-1 and EN 13466-2) available for the determination of the water content in fertilizers and liming materials. Product quality characteristics of fertilizers, especially physical and bulk behaviour properties, with effects on storing and handling, are to a major extent governed by the water content of the solid fertilizer. Consequently the water content is a very important parameter in production and product quality control. The term "moisture content" is often used to describe the amount of water present in a fertilizer.

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1 Scope

This document gives guidance on the choice for the appropriate method for the determination of the water content, considering the form under which it is present in a specific fertilizer type, and on the interpretation of the results.

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 12048, *Solid fertilizers and liming materials - Determination of moisture content - Gravimetric method by drying at (105 +/- 2)°C (ISO 8190:1992, modified)*.

EN 12049, *Solid fertilizers and liming materials - Determination of moisture content - Gravimetric method by drying under reduced pressure (ISO 8189:1992, modified)*.

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

EN 13466-1, *Fertilizers - Determination of water content (Karl Fischer methods) - Part 1: Methanol as extracting medium*.

EN 13466-2, *Fertilizers - Determination of water content (Karl Fischer methods) - Part 2: 2-propanol as extracting medium*.

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3 Terms and definitions

For the purposes of this European Standard, the following terms and definitions apply.

NOTE Water can be present in a number of forms (see [1]). It can be present as a contaminant from the atmosphere or it may be chemically bound within the sample.

3.1

essential water

water, in stoichiometric quantities, that forms an integral part of the molecular or crystal structure

3.2

water of crystallisation

essential water present in a stable solid hydrate

3.3

water of constitution

essential water that is released when the solid decomposes

3.4

non-essential water

water, in non-stoichiometric quantities, that is retained as a consequence of physical forces

NOTE The term "free water" is often used to describe non-essential water and best correlates to the physical properties and bulk behaviour.

EN 14787:2005 (E)**3.5****adsorbed water**

non-essential water that is retained on the surface of solids in a humid environment

NOTE The amount of water that is adsorbed depends on the fertilizer type, humidity, temperature and the specific surface area of the solid.

3.6**absorbed water**

non-essential water that is held as a condensed phase in the capillaries of the solid

NOTE The quantity contained in the solid is greatly dependent upon temperature and humidity.

3.7**occluded water**

liquid water entrapped in microscopic pockets throughout solid crystals

4 Methods for water determination**4.1 General**

The extent to which essential waters are removed from the sample by drying procedures, or even chemical methods such as the Karl Fischer method, is strongly dependent upon the properties of the water of crystallization and the crystal structure itself, although the Karl Fischer method is considered to be more effective in its determination. In any determination, some water will be completely removed, some will be partly removed and some might not be removed at all. This makes the interpretation of the results of the water determination in fertilizers even more complex as there are an enormous variety of components present in fertilizers. As is described in EN 13466-1 and EN 13466-2, the extracting medium also plays an important role in the Karl Fischer methods for determination of water contents.

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4.2 Gravimetric methods**4.2.1 General**

In general, drying of the sample in an oven is a quite commonly used method for the determination of the water content in a solid sample. The loss of mass, until the mass of the sample becomes constant, will then be a measure for the amount of water. The great virtue of this kind of analysis is its simplicity, but as there are some other processes besides the evolution of water being possible during the heating, the results are not necessarily the most reliable for all kinds of products. The most probable side effects are the volatilisation of other components and decomposition of one or more constituents resulting in gaseous products.

Adsorbed water is quite easily removed at temperatures above 100 °C and the equilibrium is already reached after a relatively short time. Heating at 105 °C, as is done in EN 12048, therefore will remove practically all the adsorbed water of the sample. There might even be, to a certain extent, a removal of some essential water.

Absorbed water and occluded water are not so readily removed by drying at 105 °C. In case of these forms of water it can take much more time to reach a constant mass or much higher temperatures. And even then, the removal of the water often is quite incomplete.

There are two European Standard methods available.

4.2.2 EN 12048 *Solid Fertilizers and liming materials - Determination of moisture content - Gravimetric method by drying at (105 ± 2) °C.*

The principle of the method is drying a test portion at (105 ± 2) °C for 5 h and determination of the resulting loss in mass.

The result is expressed as mass fraction in percent.

4.2.3 EN 12049 *Solid Fertilizers and liming materials - Determination of moisture content - Gravimetric method by drying under reduced pressure.*

The principle of the method is drying a test portion at a pressure of (66×10^3) Pa and a temperature of 25 °C for 24 h and determination of the resulting loss in mass.

The result is expressed as mass fraction in percent.

4.3 Extraction methods

4.3.1 Introduction

There are two European Standard methods available.

4.3.2 EN 13466-1 *Fertilizers - Determination of water content - Karl Fischer methods - Part 1: Methanol as extracting medium.*

The principle of the method is extraction of water from the fertilizer into methanol and titration of the extracted water with a Karl Fischer reagent, previously standardized by titration with a known mass of water. The method is differentiated for fertilizers dispersible or soluble in methanol, and those not dispersible in methanol.

The result, expressed as mass fraction in percent, includes "free water" and extracted water of crystallization of a number of components, possibly present in fertilizers.

4.3.3 EN 13466-2 *Fertilizers - Determination of water content - Karl Fischer methods - Part 2: 2-Propanol as extracting medium*

The principle of the method is extraction of water from the fertilizer into 2-propanol, separation of the clear solution and titration of the extracted water with a Karl Fischer reagent, previously standardized by titration with a known mass of water.

The result, expressed as a mass fraction in percent, includes "free water" and extracted water of crystallization of a limited number of components, possibly present in fertilizers.

4.4 Overview of existing methods

Table 1 gives an overview of the four existing standard methods for the determination of the water content in fertilizers and liming materials. For fertilizer types that are not listed, the applicability and precision of the methods should be further examined.

It is vital to mention the applied method when reporting any results for water.