Fertilizers - Determination of water content - (Karl Fischer methods) - Part 1: Methanol as extracting medium

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EESTI STANDARDI EESSÕNA

NATIONAL FOREWORD

Käesolev Eesti standard EVS-EN 13466-1:2002 sisaldab Euroopa standardi EN 13466-1:2001 ingliskeelset teksti. Käesolev dokument on jõustatud 19.04.2002 ja selle kohta on avaldatud teade Eesti standardiorganisatsiooni ametlikus väljaandes. Standard on kättesaadav Eesti standardiorganisatsioonist.	This Estonian standard EVS-EN 13466-1:2002 consists of the English text of the European standard EN 13466-1:2001. This document is endorsed on 19.04.2002 with the notification being published in the official publication of the Estonian national standardisation organisation. The standard is available from Estonian standardisation organisation.

Käsitlusala: This standard specifies a Karl Fischers titrimetric method for the determination of the water content of fertilizers based on the use of methanol as extracting medium.	**Scope:** This standard specifies a Karl Fischers titrimetric method for the determination of the water content of fertilizers based on the use of methanol as extracting medium.

ICS 65.080

Võtmesõnad: chemical analysis and testin, determination of content, extraction, extraction agents, fertilizers, interference factors, karl fischer, karl fischer method, materials testing, methanol, reaction, sampling, sampling methods, testing, titrimetry, water, water content

EUROPEAN STANDARD

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English version

Fertilizers - Determination of water content - (Karl Fischer methods) - Part 1: Methanol as extracting medium

Engrais - Détermination de la teneur en eau (Méthodes Karl Fischer) - Partie 1: Le méthanol comme milieu d'extraction

Düngemittel - Bestimmung des Wassergehaltes (Karl-Fischer-Verfahren) - Teil 1: Methanol als Extraktionsmittel

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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 Management Centre has the same status as the official versions.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This European Standard 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 2002, and conflicting national standards shall be withdrawn at the latest by April 2002.

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

Introduction

The water content of fertilizers has a significant effect on their quality and, especially, their storage and handling properties.

Water can be present in a number of forms such as free water, bound water and water of crystallization. It is often important to be able to distinguish between these forms of water. The gravimetric methods for determination of water standardized in EN 12048 and EN 12049 have only limited applicability.

The Karl Fischer method is applicable to a wide range of fertilizers. However, there are several variations to the basic technique, different formulations of the Karl Fischer reagents are commercially available and a number of different solvents can be used. In this standard, methanol and 2-propanol are used as extracting media to distinguish between the different forms of water present in fertilizers.

EN 13466 "Fertilizers – Determination of water content (Karl Fischer methods)" consists of two parts:

— *Part 1 : Methanol as extracting medium*

— *Part 2 : 2-propanol as extracting medium*

As examples of the difference between methanol and 2-propanol as extracting media methanol gives a result which is a combination of free water and extracted water of crystallization from the following components of fertilizers: calcium nitrate tetrahydrate ($Ca(NO_3)_2 \cdot 4H_2O$); calcium hydrogen phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$); calcium sulfate dihydrate (gypsum, $CaSO_4 \cdot 2H_2O$) calcium sulfate hemihydrate ($CaSO_4 \cdot 0{,}5H_2O$); magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$); potassium chloride magnesium sulfate water (1/1/2,75, Kainite, $KCl \cdot MgSO_4 \cdot 2{,}75H_2O$); potassium magnesium sulfate hexahydrate (Schoenite, $K_2SO_4 \cdot MgSO_4 \cdot 6H_2O$); potassium magnesium sulfate tetrahydrate (Leonite, $K_2SO_4 \cdot MgSO_4 \cdot 4H_2O$); potassium sulfate calcium sulfate monohydrate (Syngenite, $K_2SO_4 \cdot CaSO_4 \cdot H_2O$); potassium chloride magnesium chloride hexahydrate (Carnallite, $KCl \cdot MgCl_2 \cdot 6H_2O$); magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$).

Extraction with 2-propanol gives a result which is a combination of free water and extracted water of crystallization from the following components of fertilizers: calcium nitrate tetrahydrate ($Ca(NO_3)_2 \cdot 4H_2O$); magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$); potassium chloride magnesium chloride hexahydrate (Carnallite, $KCl \cdot MgCl_2 \cdot 6H_2O$); magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$).

1 Scope

This European Standard specifies a Karl Fischer titrimetric method for the determination of the water content of fertilizers based on the use of methanol as extracting medium.

The method is applicable to all solid mineral fertilizers. The result (KFM water) includes "free" water and extracted water of crystallization from the following components of fertilizers: calcium nitrate tetrahydrate ($Ca(NO_3)_2 \cdot 4H_2O$); calcium hydrogen phosphate dihydrate ($CaHPO_4 \cdot 2H_2O$); calcium sulfate dihydrate (gypsum, $CaSO_4 \cdot 2H_2O$), calcium sulfate hemihydrate ($CaSO_4 \cdot 0{,}5H_2O$); magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$) ; potassium chloride magnesium sulfate water (1/1/2,75, Kainite, $KCl \cdot MgSO_4 \cdot 2{,}75H_2O$); potassium magnesium sulfate hexahydrate (Schoenite, $K_2SO_4 \cdot MgSO_4 \cdot 6H_2O$); potassium magnesium sulfate tetrahydrate (Leonite, $K_2SO_4 \cdot MgSO_4 \cdot 4H_2O$); potassium sulfate calcium sulfate monohydrate (Syngenite, $K_2SO_4 \cdot CaSO_4 \cdot H_2O$); potassium chloride magnesium chloride hexahydrate (Carnallite, $KCl \cdot MgCl_2 \cdot 6H_2O$); magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$).

Metal oxides and hydroxides soluble in methanol and pyridine will have an effect which can be corrected for, if their content is known.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1482, *Sampling of solid fertilizers and liming materials.*

3 Principle

Extraction of water from the fertilizer into methanol and titration of the water with a Karl Fischer reagent, previously standardized by titration with a known mass of water.

4 Reagents

4.1 General

All reagents shall be of recognized analytical grade.

4.2 Methanol, containing no more than a mass concentration of 500 mg/l water.

4.3 Karl Fischer reagent, equivalence from 1 mg to 5 mg water/ml reagent (see Tables 1 and 2).

4.4 Sodium tartrate dihydrate $Na_2C_4H_4O_6 \cdot 2H_2O$ (15,66 % mass fraction of water).

Sodium tartrate dihydrate $Na_2C_4H_4O_6 \cdot 2H_2O$ can be stored over 60 % H_2SO_4 in a desiccator. Check the water content by drying at about 150 °C.

5 Apparatus

Ordinary laboratory apparatus and glassware and in particular the following:

5.1 Balance, capable of weighing to the nearest 0,0001 g.