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Determination of urea content in urea-based fertilizers by high performance liquid chromatography (HPLC)

Détermination de la teneur en urée dans les engrais à base d'urée par chromatographie liquide à haute performance (CLHP)

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*.

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Introduction

Urea is the most widely used source of nitrogen fertilizers worldwide and is used in a variety of forms such as pure urea, in combination with other nutrients, in complex fertilizers, and as reacted or modified ureas[2].

Due to the rapid hydrolysis of urea in the environment, especially when applied at the soil surface[3], efforts have been made to modify urea to slow down this loss process. Slow release nitrogen fertilizers such as methylene urea compounds, controlled release N fertilizers such as sulfur-coated urea (SCU) or polymer-coated urea (PCU), and stabilized N fertilizers containing additives (urease inhibitors and nitrification inhibitors) are examples of products aimed at containing the rapid hydrolyses of urea in the soil.

Accurate determination of urea in urea-based fertilizers is desirable for regulatory and product quality purposes. This is especially true for those fertilizers in which the urea content is physically or chemically modified. Most of these modified fertilizers contain some amounts of free and unreacted urea which is readily available N and therefore could not be accounted as part of the slow or controlled release N component[5].

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Determination of urea content in urea-based fertilizers by high performance liquid chromatography (HPLC)

1 Scope

This document specifies the test procedure for determining the urea content in urea-based fertilizers, including urea, urea aldehydes [methylene urea fertilizers, isobutylene diurea (IBDU), crotonylidene diurea (CDU)], urea triazone fertilizers, urea ammonium nitrate (UAN), sulfur- and polymer-coated urea (SCU and PCU), as well as compound fertilizers containing urea. The method is based on High Performance Liquid Chromatography (HPLC).

The proposed method is an extension of the AOAC Official Method 2003.14 which was collaboratively studied for the "Determination of Urea in Water-Soluble Urea-Formaldehyde Fertilizer Products and in Aqueous Urea Solutions" in 2003. The method was published in the Journal of AOAC in 2004[4] and was granted the First Action in 2003 and the Final Action in 2008.

This method also applies to the determination of biuret content in urea containing fertilizer with the results published in the J. AOAC in 2014[5]. This method was adopted by the International Organization for Standardization (ISO) as a Committee Draft (ISO/CD 18643) in 2014, and after review and the Ring Test Analyses[6].

NOTE This HPLC method can also be utilized to analyse Crotonylidene diurea (CDU) and Isobutylidene diurea (IBDU) contents within those above-mentioned fertilizers, in addition to EN 15705[1].

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

4 Principles

The urea content in urea-based fertilizer is extracted by aqueous acetonitrile mobile phase and separated from other contents by High Performance liquid chromatography (HPLC) on an aminopropyl column. The urea peak is detected by a UV detector attached to the HPLC.

5 Reagents

WARNING — Acetonitrile is flammable and toxic. The related operations shall be performed in a laboratory fume hood. This document does not point out all possible safety problems, and the user shall bear the responsibility to take proper safety and health measures, and ensure the

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