INTERNATIONAL STANDARD

ISO 23381

First edition
2020-09

Determination of salt out (crystallization) temperature of liquid fertilizers

Détermination de la température de désolubilisation (cristallisation) des engrais liquides

Reference number
ISO 23381:2020(E)

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Published in Switzerland

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Foreword

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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 of 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 www.iso.org/iso/foreword.html.

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The global liquid fertilizers market size is estimated to account for a value of USD 2,5 billion in 2019 and is projected to grow at a CAGR of 3,7 % from 2019, to reach a value of USD 3,1 billion by 2025. North America is the leading consumer of the liquid fertilizers with 32 % of the market, followed by Europe (25 %), Asia Pacific (21 %), Latin America (13 %), and the rest of the world at 9 %[7]. Liquid fertilizers cover inorganic nitrogen, phosphorus, potash, and micronutrients as well as organic, and synthetic fertilizers applied to soil, as foliar, in fertigation, and as starter solutions and aerial applications and in crops such as cereals, grains, fruits, vegetable, oilseeds, pulses, turf, ornamentals, forage, and plantation crops. Liquid fertilizers are the most efficient way of delivering the required nutrients to the plants at the correct time and in optimal concentration.

Urea ammonium nitrate (UAN 28-32) is an example of a widely used liquid fertilizer. Ammonium polyphosphate (APP, 10-34-0, and 11-37-0), Ammonium thiosulfate (ATS, 12-0-0-26S), potassium thiosulfate (KTS, 0-0-25-17S), magnesium thiosulfate (0-0-0-10S-4Mg), calcium ammonium nitrate (CAN 17), and solutions of water soluble fertilizers such as potassium nitrate (13-0-46), and ammonium chloride solution (6-0-0-16Cl) are few examples of liquid fertilizers.

Limitations of liquid fertilizers

Liquid fertilizers, although easier to use, and are versatile in application, they have the disadvantage of salting out (crystalize) at cold climate. This phenomenon creates limitation for transportation, storage, and handling during the cold season and proper steps is recommended to avoid crystallization.

Accurate crystallization data and an accurate, easy to use, and a universal method for the determination of salt out temperature (SOT) of liquid fertilizers is a helpful reference for stakeholders to avoid or prevent crystallization of these popular fertilizers during the cold season[8].

Currently, there is no uniform and standard method for the determination of SOTs of liquid (fluid) fertilizers. There are quite a few methods used internally by several manufacturing companies. ASTM D6660 for the freezing point determination of aqueous ethylene glycol base engine coolants by atomic phase transition method, ASTM D1177-17 for the determination of freezing point of aqueous engine coolants, and the ASTM D97 (pour point) have also been cited in the literature for SOT measurements of liquid (fluid) fertilizers. Pour point of a liquid is defined as the temperature below which the liquid loses its flow characteristic. [4]to[6]

In addition to these cited methods, there are ISO documents that provide methods to determine the melting/freezing temperature of chemicals. ISO 1392 provides a method for the determination of the crystallization point of chemicals, and ISO 3016 provides a method for petroleum oils for the determination of pour points. [1],[2]

Differential scanning calorimetry is a thermoanalytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference is measured as a function of temperature. Both the sample and reference are maintained at nearly the same temperature throughout the experiment. The temperature increase as a function of time linearly and heat flow curve vs. the temperature increase is the instrument output. These instruments are expensive and the price tag is over $50,000 (USD).

Moreover, OECD Guidelines refers to the term “melting range” for the transition of solid to liquid.

The proposed method is based on cooling/thawing of the liquid fertilizer. The liquid (fluid) fertilizer is cooled in a dry ice/methanol bath until the liquid is crystallized. It then warms at the ambient temperature. Normally, the SOT is the temperature at which the last crystal dissolves.

Determination of salt out (crystallization) temperature of liquid fertilizers

1 Scope

This document specifies the test procedure for the determination of the salt out temperature (SOT), also known as the crystallization temperature (CT) of liquid (fluid) fertilizers, using an inexpensive and simple technique.

This method might not be applicable to the binary and ternary fertilizers, especially with regards to the last crystal to disappear (LCTD). Some of these exceptions are discussed in the procedure (Clause 8).

2 Normative references

The following referenced documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696:1987, *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 https://www.iso.org/obp

4 Principles

Liquid fertilizers have been defined in ISO 8157:2015.

An aliquot of the original liquid fertilizer sample is placed into a glass test tube and is then immersed into an alcohol-dry ice bath until it crystallizes. The alcohol could be chosen from methanol, ethanol, or isopropanol (IPA). Acetone could also be used instead of alcohols; however, IPA (rubbing alcohol) is preferred due to its higher boiling point. If dry ice is not available, other lowering temperature mixtures such as salt-ice could be used to achieve the proper cooling and crystallization of the fertilizer test sample.

Once the precipitation (formation of salt crystals) is complete, the cooling source is removed and the glass test tube and its contents are allowed to warm up to room temperature. The SOT in most cases is defined when the last crystal to dissolve (LCTD) dissolves (i.e. is no longer visible in the solution). Exceptions to the LCTD rule are covered below and in 8.11.

The underlying principal of the technique can be illustrated with the following cooling curve[9] shown in Figure 1.