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Fertilizers and soil conditioners — Determination of crude (free) water content of ammoniated phosphate products — DAP, MAP — by gravimetric vacuum oven at 50 °C

Engrais — Détermination de la teneur en eau (libre) brute des produits phosphatés ammoniacaux — DAP, MAP — par étuve sous vide gravimétrique à 50 °C

ICS: 65.080

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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.

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO ____ was prepared by Technical Committee ISO/TC 134 for fertilizers and soil conditioners.

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Introduction

The moisture content of ammoniated phosphate fertilizers is an important factor in the manufacturing process and an important metric of fertilizer quality in international trade. Specifications for ammoniated phosphate products generally reference moisture content. There have been many recent developments in the phosphate fertilizer market through both new manufacturers and shifts in trading partners. It is important that there is an internationally recognized standard for testing crude (free) moisture in ammoniated phosphate fertilizer. Especially since concerns have arisen that moisture specifications are being misconstrued, due in large part to improper testing.

Accepted industry standards specify a moisture content determination by drying in a vacuum oven at 50 °C for 2 hours. The use of higher temperatures will result in the loss of ammonia from these products.

This proposed standard will prevent erroneous analysis of crude (free) moisture which can result in incorrect specification and/or misinterpretation of analysis on trade documentation. This method will conform to accepted industry standards, benefitting manufacturers through quality control, labeling, registration, sales and marketing; purchasers of DAP, MAP products who currently have no means of evaluating and comparing products, and governmental agencies requiring regulatory standards for these products.

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Fertilizers and soil conditioners — Determination of Crude (Free) water content of Ammoniated Phosphate products — DAP, MAP — by gravimetric vacuum oven at 50 °C

1 Scope

This International Standard establishes a method for the determination of crude (free) moisture in ammoniated phosphate products – specifically Di-ammonium phosphate (DAP) and Mono-ammonium phosphate (MAP) fertilizer materials. Moisture is determined by drying a representative portion of the sample under vacuum and at a temperature of 50 °C.

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.

ISO 8157, *Fertilizers and soil conditioners — Vocabulary*

3 Terms and Definitions

3.1

Crude (Free) Water

Moisture found on the surface of the material, i.e. not chemically bound.

Note 1 to entry: This definition to be included in revision to ISO 8157

3.2

DAP

Di-ammonium phosphate

A product principally composed of ammonium phosphates, di-ammonium phosphate, resulting from the ammoniating of phosphoric acid.

3.3

MA

Mono-ammonium phosphate

A product composed of ammonium phosphates, principally mono-ammonium phosphate, resulting from the ammoniating of phosphoric acid.

4 Principle

A weighed portion of an unground sample is dried in a vacuum oven at 48-53 cm Hg and a temperature of 50 °C for 2 hours. The loss in weight expressed as a percentage of the original sample weight is deemed to be the crude (free) moisture.

5 Safety

5.1 General Requirements: A minimum of standard laboratory personal protective equipment including safety glasses, gloves, and lab coats should be worn at all times.

6 Reagents

6.1 Drying agents:

6.1.1 Sulfuric acid 18M - is a strong oxidizing agent and should be used with caution. Note: Handling of Concentrated Sulfuric Acid: Gloves, safety goggles, face shields, and lab coats should always be worn when handling concentrated sulfuric acid. Sulfuric acid is extremely corrosive and dehydrating, causing severe burns. Care must be taken to avoid eye or skin contact. If contact is made with eyes or skin, flush immediately with clean tap water and seek immediate medical attention. To avoid the potential for fuming and spattering of concentrated sulfuric acid during dilution, always add sulfuric acid to water and do not add water to sulfuric acid.

6.1.2 Silica gel or Calcium chloride suitable for use as a drying agent.

7 Apparatus

7.1 Vacuum oven – capable of maintaining vacuum of 48-53 cm (19-21”) Hg and temperature control of 50 +/- 1 °C

7.2 Weighing dish with close-fitting lid

7.3 Analytical balance – accuracy 0,1mg

7.4 Drying trap with suitable drying agent (See 7.1)

7.5 Desiccator with suitable drying agent.

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8 Procedure

8.1 Tare weighing dish and lid previously dried in oven. Record weight to nearest 0,1 mg.

8.2 Accurately weigh a two gram sample into the tared weighing dish. Record weight to nearest 0.1 mg (See Note regarding ground / unground sample).

8.3 Place the weighing dish in vacuum oven. Note: do not cover dish while in oven.

8.4 Dry the sample for two hours +/-10 minutes at 50 ° C +/-1,0 with the oven under a vacuum of 48-53 cm (19-21 inches) of mercury (Hg). Temperature must be controlled within the specified limits throughout the oven chamber. Maintain the vacuum by passing desiccated air (which has been passed through a drying trap) through the chamber.

8.5 After the allotted time, remove the weighing dish containing the sample from oven, immediately place the lid tightly on top and place in desiccator to cool.

8.6 After cooling, reweigh the weighing dish and sample, record the weight thus obtained.

8.7 Report the loss in weight as percent free water.

9 Calculations

9.1 Calculate the crude (free) moisture in the fertilizer product test sample, using the following equation.

$$\% \text{ Crude (Free) Water} = \text{Loss in Weight} \times 100 / \text{Weight of Sample}$$

10 Precision

10.1 Inter laboratory tests: Table 1 summarizes the results of an inter-laboratory test protocol. Repeatability and reproducibility were calculated according to ISO 5725-1 and ISO 5725-2.

Table 1 — Mean values, repeatability and reproducibility

Product	No. Labs	Average Moisture Value - %	r	R
DAP - 1	10	2,947	0,063	0,465
DAP - 2	8	2,186	0,156	1,638
DAP - 3	9	3,277	0,072	0,475
DAP - 4	9	1,586	0,058	0,323
DAP - 5	11	2,165	0,102	0,709
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MAP - 1	8	0,915	0,06	0,29
MAP - 2	8	0,787	0,109	0,311
MAP - 3	10	2,247	0,161	0,617
MAP - 4	12	0,673	0,086	0,424
MAP - 5	9	1,116	0,12	0,95