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Fertilizers and soil conditioners — Water soluble fertilizer — General requirements

Matières fertilisantes — Engrais soluble dans l'eau — Exigences générales

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Foreword

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 134, *Fertilizers and soil conditioners.*

Fertilizers and soil conditioners — Water soluble fertilizer — General requirements

1 Scope

This International Standard specifies the requirements for testing methods, sampling and preparation of test sample, marking and labelling, as well as package, transport, and storage of water soluble fertilizers.

This International Standard is applicable to water soluble fertilizers which are completely soluble in water and are suitable for fertigation and sprinkling irrigation, as well as for foliar application (foliar feeding).

2 Normative references

The following 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, *Water for analytical laboratory use — Specification and test methods*

ISO 5315, *Fertilizers — Determination of total nitrogen content — Titrimetric method after distillation*

ISO 6353-1:1982, *Reagents for chemical analysis — Part 1: General test methods*

ISO 6598, *Fertilizers — Determination of phosphorus content — Quinoline phosphomolybdate gravimetric method*

ISO 7409, *Fertilizers — Marking — Presentation and declarations*

ISO 7410, *Fertilizers and soil conditioners — Final samples — Practical arrangements*

ISO 7742:1988, *Solid fertilizers — Reduction of samples*

ISO 8633, *Solid fertilizers — Simple sampling method for small lots*

ISO 17318, *Fertilizers and soil conditioners — Determination of arsenic, cadmium, chromium, lead and mercury contents*

ISO 17319, *Fertilizers and soil conditioners — Determination of water-soluble potassium content — Potassium tetraphenylborate gravimetric method*

ISO 18643, *Fertilizers and soil conditioners — Determination of biuret content of urea-based fertilizers — HPLC method*

EN 16195, *Fertilizers — Determination of chlorides in the absence of organic material*

CEN/TS 16196, *Fertilizers — Manganimetric determination of extracted calcium following precipitation in the form of oxalate*

EN 16198, *Fertilizers — Determination of magnesium by complexometry*

EN 13366, *Fertilizers — Treatment with a cation exchange resin for the determination of the chelated micro-nutrient content and of the chelated fraction of micro-nutrients*

EN 13368-1, *Fertilizers — Determination of chelating agents in fertilizers by chromatography —Part 1: Determination of EDTA, HEEDTA and DTPA by ion chromatography*

EN 13368-2, *Fertilizers — Determination of chelating agents in fertilizers by chromatography — Part 2: Determination of Fe chelated by o,o-EDDHA and o,o-EDDHMA by ion pair chromatography*

EN 15451, *Fertilizers — Determination of chelating agents —. Determination of iron chelated by EDDHSA by ion pair chromatography*

EN 15452, *Fertilizers — Determination of chelating agents — Determination of iron chelated by o,p-EDDHA by reversed phase HPLC*

EN 15749, *Fertilizers — Determination of sulfates content using three different methods*

EN 15950, *Fertilizers — Determination of N-(1,2-dicarboxyethyl)-D,L-aspartic acid (Iminodisuccinic acid, IDHA) using high-performance liquid chromatography (HPLC)*

EN 15961, *Fertilizers — Extraction of water-soluble calcium, magnesium, sodium and sulfur in the form of sulfates*

EN 15962, *Fertilizers — Determination of the complexed micro-nutrient content and of the complexed fraction of micro-nutrients*

EN 16109, *Fertilizers — Determination Of complexed micro-nutrient ions in fertilizers — Identification of lignosulfonates*

EN 16847, *Fertilizers — Determination of complexing agents in fertilizers — Identification of heptagluconic acid by chromatography*

EC Regulation No. 2003/2003

3 Requirement

3.1 Appearance: the product (including single fertilizers, complexed micronutrients, and/or products obtained chemically) is a mixture of powders, prills, and/or granules free of foreign matter.

3.2 Water soluble fertilizer products shall be tested to demonstrate the conformance with the requirements specified in Table 1, when applicable, and the values declared on containers.

Table 1 — Requirements of water soluble fertilizer

Items		Requirements
Mass fraction of total primary nutrient ($N + P_2O_5 + K_2O$)[a], % ≥		20
Mass fraction of secondary nutrients (represented in elementary substance or oxides)[b], %		Marked value
Mass fraction of micronutrients (represented in elementary substance)[b], %		Marked value
Chelated fraction of micro-nutrients[c], %		Marked value
Mass fraction of water insoluble matter, % ≤		0,5
pH value measured at 10 g/100 mL water		2,0~9,0
Mass fraction of biuret[d], % ≤		1,2
Mass fraction of chloride ion[e], %	Products not labeled with “contains chloride” ≤	2
	Products labeled with “contains chloride” >	2

a If the product contains primary nutrients (N, P_2O_5, K_2O), the product should contain at least one of the primary nutrients. For binary or ternary fertilizers, the declared primary nutrients of N should not be less than 3 %, the declared primary nutrients of P_2O_5 and K_2O should not be less than 5 %. For straight fertilizer, the mass fraction of single primary nutrient should refer to in EC Regulation No. 2003/2003.

b For detailed regulations, refer to EC Regulation No .2003/2003.

c This item should be only tested when the chelated form of micro-nutrients are declared.

d This item should be tested wherever the existence of urea nitrogen is listed on the containers; otherwise, it should be exempted.

e For the limitation of mass fraction of chloride and mass fraction of total primary nutrient, applicable local/regional legislations/laws/rules need to be followed.

NOTE 1 Regarding the biuret content, the related requirements specified by the countries or regions need to be followed.

NOTE 2 Regarding the content of As, Cd, Pb, Cr and Hg the related requirements specified by the countries or regions shall be followed.

4 Test method

4.1 Visual inspection for foreign matter

Visually inspect the product for the presence of contaminants and foreign matter.

4.2 Determination of nitrogen, phosphorus and potassium contents

Determine the nitrogen content in accordance with ISO 5315.

Determine the phosphorus content in accordance with ISO 6598.

Determine the potassium content in accordance with ISO 17319.

4.3 Determination of calcium, magnesium and sulphur contents

Prepare the test solution in accordance with EN 15961.

Determine the calcium content in accordance with CEN/TS 16196.

Determine the magnesium content in accordance with EN 16198.

Determine the sulphur content in accordance with EN 15749.

4.4 Determination of molybdenum, boron and manganese, zinc, copper and iron contents

Determine the molybdenum, boron and manganese, zinc, copper, and iron contents in accordance with EC Regulation No. 2003/2003, Annex IV, methods 9 and 10.

4.5 Determination of the chelated fraction of micro-nutrients

Determine the chelated fraction of micro-nutrients in accordance with the following: EN 13366, EN 13368-1, EN 13368-2, EN 15451, EN 15452, EN 15950, EN 15962, EN 16109, EN 16847.

4.6 Determination of the mass fraction of water insoluble matter

4.6.1 Principle

Dissolve/dilute the test sample in water, and determine the residual content that is insoluble in the water.

4.6.2 Reagents and materials

Use only water conforming to grade 3 of ISO 3696.

4.6.3 Apparatus

4.6.3.1 Common laboratory apparatus.

4.6.3.2 Glass crucible filter, No. 1, with volume of 30 ml.

4.6.3.3 Reduced-pressure suction filtration device.

4.6.3.4 Drying oven, with temperature controlled at (110 ± 2) °C.

4.6.4 Ambient temperature controlled at (20 ± 10) °C

4.6.5 Test procedure

Weigh 25 g test sample (accurately to 0,001 0 g) into a beaker, add 250 ml water and stir thoroughly for 3 min, standing in ambient temperature for (15 ± 3) mins.

Filter the solution with the glass crucible filter, which has already been dried to a constant weight in a drying oven at (110 ± 2) °C (dry until the weight of the two measurements is ≤0,3 mg). Transfer the residual content into the filter with a minimum amount of water.

Put the glass crucible filter with all the residual content into a drying oven at (110 ± 2) °C, keep it at 110 °C for 1 h, and then transfer the glass crucible filter into a desiccator, wait until it cools down to the room temperature and weigh it.

For the parallel blank test, follow the same procedure, except adding the test portion.

The glass crucible filter should be washed thoroughly after filtering, recommended washing process including soaking with potassium dichromate-sulphuric acid lotion overnight, then carefully flush with fresh water, reserved after suction filtering.

4.6.6 Calculation of the test result

The mass fraction of water insoluble material, *w*, represented in mass fraction (%), shall be calculated by Formula (1):

$$w = \frac{m_1 - m_0}{m} \times 100 \quad (1)$$

where

m_1 is the mass of water insoluble material, with the unit of gram (g);

m_0 is the mass of water insoluble material in the blank test, with the unit of gram (g);

m is the mass of the test portion, with the unit of gram (g).

Take the arithmetic mean of duplicate tests as the final result; the final result should be rounded to two decimal places.

4.6.7 Tolerance

The absolute difference value of two parallel tests should be ≤0,30 %, when the mass fraction of water insoluble material is ≤2,0 %.

The absolute difference value of two parallel tests should be ≤0,40 %, when the mass fraction of water insoluble material is >2,0 %.

4.7 Determination of the pH value

Determine the pH value in accordance with ISO 6353-1:1982, 5.31.1.

4.8 Determination of arsenic, cadmium, lead, chromium and mercury contents

Determine the arsenic, cadmium, lead, chromium, and mercury contents in accordance with ISO 17318.

4.9 Determination of biuret content

Determine the biuret content in accordance with ISO 18643.

4.10 Determination of mass fraction of chloride

Determine the mass fraction of chloride in accordance with EN 16195 and EC Regulation No 2003/2003, Annex III Method 6.

5 Sampling and preparation of test sample

5.1 Sampling method

5.1.1 Products in bags

Carry out sampling operation by following the procedure described in ISO 8633.

5.1.2 Products in bulk

Carry out sampling operation by following the procedure described in ISO 8633.

5.2 Reduction of samples

Mix all the increments (collected as described in 5.1) uniformly and promptly to form a single aggregate sample using a device or by hand. The aggregate sample is reduced to about 1 kg by the riffle sample divider method (see ISO 7742:1988, Annex A.2) or the quartering method (see ISO 7742:1988, Annex A.3). Next, divide into two parts for final laboratory samples. The two laboratory samples are put into two clean and dry glass or plastic containers or any other inert material of adequate resistance capable of maintaining the sample in its original condition. The containers shall be fitted with airtight closures. Carry out all the operations described above as rapidly as possible to avoid loss or gain of moisture. Each container shall be secured and sealed following the instruction given in ISO 7410. Each laboratory sample shall be labelled following the instructions given in ISO 7410. The label shall, at minimum, carry the following information:

a) name of manufacturer;

b) name of product and type;

c) manufacturer's reference and batch number or production date (if available);

d) lot size;

e) date of sampling;

f) place of sampling;

g) signature of the sampler;

h) signature and name of the person or his representative on whose premises the sample was taken.

One of the containers is used for further quality analysis, while the other is kept for additional analysis in six months.

5.3 Test sample preparation

Select one of the laboratory samples from the two containers obtained in 5.2; mix the content within the container, according to the procedure given in ISO 7742, reduce the sample to 100 g as one of the test samples used for determination of all the content indexes required in Table 1. Grind this test sample in the grinder until all the sample has passed through the test sieve of aperture size 0,50 mm. All test samples are put into a clean dry bottle to be used for further analysis.

NOTE Some water soluble fertilizers may convert to a liquid phase if ground with a mechanical grinder. Either pre-test a subsample of the fertilizer first to ensure it will maintain its solid phase, or alternatively, the sample can be ground by hand with a mortar and pestle or tested in its unground state.

6 Marking and labelling

6.1 For marking and labelling, the relevant national (or regional) legislation shall be followed. Also for classification for health, safety, environment, storage, and transport, the appropriate legislation shall be followed. The following (6.2 to 6.7) are the guidelines.

6.2 The total nutrient content and single nutrient contents should be labelled on the container. All the forms of nitrogen nutrient content should be listed respectively. The declared total nitrogen nutrient content should be equal to the sum of all the single nitrogen nutrient contents.

The label should conform to applicable regulations/legislation.

6.3 The amount of water insoluble matter and pH value should be labelled on the container (such details are written regularly in data sheets, not on product label on bags). The pH should be presented as a single numerical value and reduced to the nearest integer.

6.4 Proper use instructions of product should be included on the container, which including, but not limited to the following content: applicable region, soil, crops, and growth stage (or instead, inapplicable region, soil, crops, and growth stages); usage and dosage; compatibility with other materials; incompatible materials; special requirement on the quality of irrigation water. Furthermore, the product should contain the proper classification and warnings.

Other information should be available in technical brochures as per local sales responsibilities. Labelling requirements are an integral part of the legislation.

6.5 If the product contains secondary and micro nutrients, the contents of each secondary and micro nutrient (secondary nutrients can be represented in elementary substance or oxides, micronutrients should be represented in elementary substance) shall be labelled, respectively, following applicable regulations. If the micro elements are present as chelated compounds, the chelating agent should be declared or labelled as per regulations.

6.6 Products containing more than 2 % of chloride ion (in mass fraction) should be labelled with "contains chloride" and the value of chloride mass fraction on the container and should not be labelled with "Chlorine" or "Cl". Containers of those products which are labelled with "contains chloride" should not have any picture of chlorine intolerance crops on it, or any label with the word "sulphate based", which will mislead the consumer that those products do not contain chloride content.

NOTE For the limitation of mass fraction of chloride, applicable local/regional legislations/laws/rules need to be followed.

6.7 All other information required by national and regional regulations and ISO 7409.

7 Package, transport and storage

7.1 The net mass of each package shall be determined by negotiation. The net mass should be on each package.

7.2 During transportation, the packaged products should be handled with care to avoid moisture, sunlight, and damage.

7.3 The products should be stored in a dry, cool place away from sunlight and moisture.