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Solid fertilizers — Determination of mineral-acid-soluble sulfate content — Gravimetric method

Matières fertilisantes solides — Détermination de la teneur en sulfates solubles dans les acides minéraux — Méthode gravimétrique

Reference number
ISO 10084:1992(E)

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.

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.

International Standard ISO 10084 was prepared by Technical Committee ISO/TC 134, *Fertilizers and soil conditioners*, Sub-Committee SC 4, *Chemical analysis*.

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Solid fertilizers — Determination of mineral-acid-soluble sulfate content — Gravimetric method

1 Scope

This International Standard specifies a method for the gravimetric determination of the mineral-acid-soluble sulfate content of solid fertilizers. The method is applicable to fertilizers with sulfate contents, expressed as SO_3, from 3 % (*m/m*) to 50 % (*m/m*).

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 8358:1991, *Solid fertilizers — Preparation of samples for chemical and physical analysis.*

3 Principles

Dissolution of soluble sulfates in hydrochloric acid solution. Precipitation of sulfate ions in a hydrochloric acid medium with barium chloride. Filtration, washing, drying, ignition and weighing of the precipitate.

4 Reagents

All reagents shall be of recognized analytical grade. The water used shall be distilled water or water of equivalent purity.

4.1 Hydrochloric acid solution, ρ_{20} = 1,19 g/ml.

4.2 Barium chloride dihydrate solution, $c(BaCl_2 \cdot 2H_2O)$ = 122 g/l.

4.3 Silver nitrate solution, $c(AgNO_3)$ = 5 g/l.

5 Apparatus

Ordinary laboratory apparatus and the following.

5.1 Electric hot-plate

5.2 Filter crucibles with **porcelain disc**, porosity grade P10, pore size index 4 µm to 10 µm.

5.3 Oven, capable of being maintained at a temperature of 120 °C ± 5 °C.

5.4 Oven, capable of being maintained at a temperature of 800 °C ± 50 °C.

5.5 Desiccator, filled with a suitable desiccant.

6 Preparation of the test sample

Prepare the laboratory sample according to ISO 8358 and transfer at least 50 g to the laboratory for analysis.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,1 mg, the mass of the test sample (clause 6) which, according to table 1, corresponds with the expected acid-soluble sulfate content.

NOTE 1 The amount of SO_3 present in the test portion should be 200 mg to 600 mg.

Table 1 — Mass of test portion in relation to the expected acid-soluble sulfate content

Expected acid-soluble sulfate content, as SO_3 % (*m*/*m*)		Approximate mass of test portion g
from	to	
3	5	10
5	10	5
10	30	2
30	50	1

7.2 Dissolution of the test portion

Transfer the test portion (7.1) into a 400 ml beaker. Add 170 ml to 180 ml of water and 15 ml of hydrochloric acid (4.1). Bring to the boil and continue boiling for 10 min. Allow to cool.

Transfer the mixture quantitatively to a one-mark 250 ml volumetric flask, with the aid of a funnel, and then rinse the beaker and the funnel into the same flask. Make up to the mark with water, mix and filter through a dry, folded filter paper, collecting the filtrate in a dry bottle.

7.3 Determination

Transfer 100 ml of the filtrate (7.2) to a 800 ml beaker. Make up with water to 300 ml and add 20 ml of hydrochloric acid (4.1). Bring to the boil.

While mixing thoroughly, add drop by drop 20 ml of the barium chloride solution (4.2). Continue boiling for a few minutes.

Place the beaker on the hot-plate (5.1) set at 60 °C and cover it with a watch-glass. Allow to stand at 60 °C for 3 h.

Dry a filter crucible (5.2) in the oven (5.4) set at 800 °C. Allow it to cool in the desiccator (5.5) then weigh to the nearest 0,1 mg.

Decant the clear supernatant liquid from the beaker through the crucible. Wash the precipitate in the beaker several times by decanting with hot water. Transfer the precipitate to the dried crucible by means of a hot water jet. Wash the precipitate with hot water in order to remove chloride ions. Continue washing until the absence of chloride ions has been established by means of the silver nitrate solution (4.3). Dry the crucible with the precipitate for 1 h in the oven (5.3) set at 120 °C. Place the crucible in the oven (5.4) set at 800 °C and heat the dried precipitate for 0,5 h.

Allow the crucible to cool in the desiccator and weigh to the nearest 0,1 mg.

8 Calculation and expression of results

Calculate the mineral-acid-soluble sulfate content from the following equation:

$$w_S = 85{,}75 \times \frac{m_1}{m_0}$$

where

w_S is the mineral-acid-soluble sulfate content expressed as SO_3, as a percentage by mass;

m_1 is the mass of the dried and heated precipitate, in grams;

m_0 is the mass of the test portion, in grams.

Round the result obtained to the nearest 0,01 % (*m*/*m*).

9 Precision

The precision of this method has been established by an international inter-laboratory test carried out in accordance with ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

In this test, six samples of different origin and acid-soluble sulfate contents were investigated by sixteen laboratories in seven countries.

For the values obtained for repeatability limit and reproducibility limit, a probability level of 95 % holds.

9.1 Repeatability

The absolute difference between two single tests results, obtained under repeatability conditions, shall not exceed the value of r given by the formula

$$r = 0{,}042\ \% + 0{,}009\ 1\ \overline{w}_S$$

where $\overline{w}_S$ is the arithmetic mean of the results expressed as SO_3, as a percentage by mass.

Reject both results if the difference exceeds the calculated value of r and carry out two new single determinations.

If the repeatability requirement is met, calculate the arithmetic mean of the two results to the nearest 0,01 % (*m*/*m*).

NOTE 2 The following definitions are taken from ISO 5725.

single test result: The result obtained by carrying out a specified test method one time according to the prescribed procedure.

repeatability conditions: Conditions where mutually independent test results are obtained with the same

method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

9.2 Reproducibility

The absolute difference between two single test results, obtained under reproducibility conditions, shall not exceed the value R given by the formula

$$R = 0{,}098\ \% + 0{,}028\ 4\ \overline{w}_S$$

where $\overline{w}_S$ is the arithmetic mean of the two results expressed as SO_3, as a percentage by mass.

NOTE 3 The following definition is taken from ISO 5725.

reproducibility conditions: Conditions where test results are obtained with the same method on identical test material in different laboratories with different operators using different equipment.

10 Test report

The test report shall include the following information:

a) all information necessary for the complete identification of the sample;

b) a reference to this International Standard;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not specified in this International Standard or regarded as optional.

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