International Standard 5809

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Starches and derived products — Determination of sulphated ash

Amidons, fécules et produits dérivés — Détermination des cendres sulfatées

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the member bodies of the following countries:

Austria	Netherlands	South Africa, Rep. of
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France	Portugal	USSR
Germany, F.R.	Romania	

No member body expressed disapproval of the document.

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Starches and derived products — Determination of sulphated ash

1 Scope and field of application

This International Standard specifies a method for the determination of sulphated ash in starches and derived products.

2 References

ISO 1666, *Starch — Determination of moisture content — Oven-drying methods.*[1)]

ISO 1741, *Dextrose — Determination of loss in mass on drying — Vacuum oven method.*

ISO 1742, *Glucose syrups — Determination of dry matter — Vacuum oven method.*

ISO 1743, *Glucose syrup — Determination of dry matter — Refractive index method.*

3 Definition

sulphated ash: The residue obtained after incineration of the product, according to the method specified in this International Standard.

It is expressed as a percentage by mass either of the product as-received or on the dry basis.

4 Principle

Incineration of a test portion, in the presence of sulphuric acid, at a temperature of 525 ± 25 °C.

The sulphuric acid facilitates the destruction of the organic matter and avoids losses by converting the volatile chlorides into non-volatile sulphates.

5 Reagents

During the analysis, use only reagents of recognized analytical quality and only distilled water or water of at least equivalent purity.

5.1 Sulphuric acid solution.

Add, carefully, 100 ml of concentrated sulphuric acid, ϱ_{20} 1,83 g/ml, to 300 ml of water and mix.

5.2 Hydrochloric acid solution.

Add, carefully, 100 ml of concentrated hydrochloric acid, ϱ_{20} 1,19 g/ml, to 500 ml of water and mix.

6 Apparatus

Ordinary laboratory apparatus, and in particular

6.1 Incineration dish, of platinum or any other material which does not deteriorate under the test conditions (for example a silica incineration dish), of capacity 100 to 200 ml and with a minimum useful surface of 15 cm^2.

6.2 Electric furnace with air circulation, capable of being controlled at 525 ± 25 °C.

6.3 Electric hot-plate or **gas burner** or **heating lamp.**

6.4 Desiccator, provided with an efficient desiccant.

6.5 Water bath, capable of being controlled at 60 to 70 °C.

6.6 Analytical balance.

7 Procedure

7.1 Preparation of the incineration dish

Clean the incineration dish (6.1), whether it is new or used, with boiling hydrochloric acid solution (5.2), then rinse generously with water.

Calcinate the incineration dish for 30 min in the furnace (6.2), controlled at 525 ± 25 °C. Allow to cool to ambient temperature in the desiccator (6.4) and weigh to the nearest 0,000 2 g (the incineration dish should be calcinated to constant mass).

1) Under revision.

7.2 Preparation of the test sample

Mix the sample carefully and quickly by stirring (for a powder) or by mixing with a spatula (for a liquid) in a sample container.[1)]

If the volume of the container is insufficient for this, quickly transfer the whole sample to another, previously dried container of a suitable size.

Take care to avoid any change in the moisture content of the sample. The taking of a representative sample of approximately 5 g can be difficult (for example glucose in lumps). In this case, use one of the procedures described below:

a) Weigh carefully, to the nearest 0,01 g, approximately 100 g of the sample into a dry container, provided with a lid, previously tared with the lid. Add approximately 100 ml of water at 90 °C, place the lid on the container and stir until the sample is completely dissolved. Allow to cool to ambient temperature and weigh to the nearest 0,01 g.

b) Melt the sample in solid form by immersing it, in a container, provided with a lid, in the water bath (6.5), controlled at 60 to 70 °C, and placing the lid on the container. Remove the container from the water bath and allow it to cool to ambient temperature, agitating frequently but without removing the lid, and then mix the condensed water with the sample.

7.3 Test portion

If a dilution has been carried out, take an aliquot portion of the solution obtained (see 7.2), so as to obtain a mass of sample corresponding to a mass of test portion as shown in the table.

In all other cases, weigh, to the nearest 0,001 g, in the incineration dish (6.1), previously weighed to the nearest 0,000 2 g, a mass of test sample (7.2) in accordance with the following table.

Sulphated ash	Mass of test portion
% (*m*/*m*)	g
⩽ 5	10
> 5 ⩽ 10	5
> 10	2

7.4 Preincineration

Add 5 ml of the sulphuric acid solution (5.1) to the test portion or the aliquot portion (7.3). Mix with a glass stirring rod and rinse with a little water, collecting the rinsings in the incineration dish. Heat the incineration dish slowly and carefully, over the electric hot-plate or gas burner or using the heating lamp (6.3), until completely carbonized (it is recommended that this be carried out under an extraction hood).

7.5 Incineration

Place the incineration dish in the oven (6.2), controlled at 525 ± 25 °C, and maintain this temperature until the carbon residue has disappeared. A period of 2 h is usually sufficient.

Allow to cool. Take up the residue with several drops of the sulphuric acid solution (5.1), evaporate on the edge of the oven (6.2) and incinerate again for 0,5 h. Place the incineration dish in the desiccator (6.4) and allow it to cool to ambient temperature. Weigh the dish and contents to the nearest 0,000 2 g. The incineration should be continued until constant mass is attained.

Do not put more than four incineration dishes in the desiccator at any one time.

7.6 Number of determination

Carry out two determinations on test portions taken from the same test sample (7.2).

8 Expression of results

8.1 Method of calculation and formulae

The sulphated ash, expressed as a percentage by mass of product as-received, is given by the formula

$$(m_2 - m_1) \times \frac{100}{m_0}$$

The sulphated ash, expressed as a percentage in mass on the dry basis, is given by the formula

$$(m_2 - m_1) \times \frac{100}{m_0} \times \frac{100}{100 - H}$$

where

m_0 is the mass, in grams, of the test portion, taking into account any dilution (7.3);

m_1 is the mass, in grams, of the incineration dish before incineration (7.1);

m_2 is the mass, in grams, of the incineration residue and incineration dish after incineration (7.5);

H is the moisture content of the product, determined concurrently in accordance with ISO 1666, ISO 1741, ISO 1742 or ISO 1743.

1) For glucose syrup, remove the surface layer (approximately 0,5 cm) before mixing.

Take as the result the arithmetic mean of the values obtained in the two determinations, provided that the conditions of repeatability (see 8.2) are satisfied. If not, repeat the test.

NOTE — In the hydrolysis products of starch containing sodium chloride as the preponderant mineral matter, the ash may be evaluated as sodium chloride by conventionally multiplying the sulphated ash by the coefficient 0,823 which is the ratio

$$\frac{\text{sodium chloride}}{\text{sodium sulphate}} = \frac{2\,(\text{Na Cl})}{(\text{Na}_2\text{SO}_4)}$$

Express the result rounded to two decimal places.

8.2 Repeatability

The absolute difference between the values obtained in two determinations carried out simultaneously or in rapid succession by the same analyst on the same test sample shall not be greater than

— 4 % of their arithmetic mean in the case of sulphated ash greater than 2 % (m/m);

— 0,08 g per 100 g in the case of sulphated ash less than 2 % (m/m).

8.3 Reproducibility

The absolute difference between the values obtained in two determinations carried out on the same sample in two different laboratories shall not exceed

— 0,1 g per 100 g for sulphated ash less than 0,5 % (m/m);

— 1 % of their arithmetic mean for sulphated ash greater than 5 % (m/m);

— 20 % of their arithmetic mean for sulphated ash between 0,5 and 5 % (m/m).

9 Test report

The test report shall indicate the method used, together with the result obtained and the method of expression used. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have affected the results.

The test report shall include all details required for the complete identification of the sample.

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