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Aluminium fluoride for industrial use — Determination of trace elements — Wavelength dispersive X-ray fluorescence spectrometric method using pressed powder tablets

Fluorure d'aluminium à usage industriel — Détermination d'éléments traces — Méthode par spectrométrie de fluorescence des rayons X à dispersion de longueur d'onde utilisant des pastilles de poudre pressée

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12926 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

Introduction

Aluminium fluoride is used as an electrolyte additive to the aluminium smelting bath to regulate the acidity, or excess aluminium fluoride, level of the electrolyte. This use is critical in the operation of all electrolysis cells used for the production of aluminium. Aluminium fluoride is also used in the cast house as an additive to metal in crucibles in the Treatment of Aluminium in a Crucible process (TAC).

Aluminium fluoride for industrial use — Determination of trace elements — Wavelength dispersive X-ray fluorescence spectrometric method using pressed powder tablets

1 Scope

This International Standard describes an X-ray fluorescence spectrometric (XRF) method for the determination of aluminium fluoride (AlF_3) from the content of fluorine and the content of trace elements in the test specimen. The method does not determine the oxygen content. The calibration reference materials are not specified in this method.

The method is applicable to industrial-grade aluminium fluoride where the concentration range for aluminium fluoride and each trace element is within the concentration range given in Table 1. The validity and precision of test results for concentrations outside these ranges has not been determined (see Note).

Table 1 — Concentration range for aluminium fluoride (from fluorine) and trace elements

Compound or element	Symbol	Concentration range mass %
Aluminium fluoride	AlF_3	86,5 to 95,75
Sodium	Na	0,05 to 0,25
Silicon	Si	0,001 to 0,14
Phosphorus	P	0,001 to 0,02
Sulfur	S	0,01 to 0,6
Calcium	Ca	0,001 to 0,10
Iron	Fe	0,005 to 0,05

NOTE The determination of fluorine has an uncertainty due to mineralogical variation among origins of aluminium fluoride. Batches from different origins with the same fluorine contents can give different intensities when determined by this method and pressed tablet preparation does not eliminate this problem. An AlF_3 sample of unusual mineralogical origin should be tested with an absolute method to verify that, when using this test method, the fluorine concentration range reported is valid.

2 Normative references

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

ISO 1619, *Cryolite, natural and artificial — Preparation and storage of test samples*

3 Principle

A representative sample of aluminium fluoride is milled. A test portion is packed and pressed on a powder tablet press to make the test tablets.

The test tablets are analysed on an X-ray fluorescence spectrometer instrument that has been calibrated using a series of aluminium fluoride reference materials covering the required concentration range of the elements to be determined.

4 Reagents and materials

4.1 Lithium tetraborate backing, analytical pure lithium tetraborate.

4.2 Acetone, analytical pure.

4.3 Aluminium tablet dish, of diameter suitable for the tablet press (5.3) and tapered to hold pressed pellets.

4.4 Mylar film, for keeping the test portion separate from press surfaces.

4.5 Binding agent, suitable for sample preparation. The following are some examples of materials that have been found satisfactory:

— polyethylene binder, PE-160, PE-190

— mill and press additive, HMPA40 - Herzog mill and press additive[1)]

— XRF Multi-Mix RXR-250, Premier Lab Supply[2)]

— Retsch - Licowax C micropowder binder (formerly known as Hoechst Wax or Hoechst LICO wax C Micropowder[3)]

— wax, Clairiant Licowax PE-190[4)]

— mixture, 9:1 of BASF styrene EMU 120 FD[5)] to Hoechst Ceridust 9615A Wax.

4.6 Cleaning agent, if required. Dupont Vertrel XF (MS-782)[6)] has been found suitable.

5 Apparatus

5.1 Wavelength dispersive X-ray fluorescence spectrometer, with vacuum path and equipped with crystals required as shown in Table 3.

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1) HMPA40 - Herzog mill and press additive is the trade name of a product supplied by Hertzog Automation Corporation www.herzogautomation.com This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

2) XRF Multi-Mix RXR-250 is the trade name of a product supplied by Premier Lab Supply. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

3) Licowax C micropowder (formerly Hoechst wax) is the trade name of a product supplied by Retsch, Socachim, Spectro. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

4) Clairiant Licowax PE-190 is the trade name of a product supplied by Clariant Ltd www.clariant.com (formerly Hoechst). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5) BASF styrene EMU 120 FD is the trade name of a product supplied by BASF www.basf.com. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

6) Dupont Vertrel XF (MS-782)is the trade name of a product supplied by Dupont www2dupont.com/vertrel. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.2 Vibratory disc mill, with tray, ring and puck made of non-contaminating material, tungsten-carbide and chrome steel have been found to be satisfactory.

5.3 Tablet press, capable of providing a 20 ton or up to 400 kN load for 10 s.

5.4 Mould with Mylar film, for forming tablets in press.

5.5 Desiccator, with a non-contaminating desiccant for storing calibration materials and monitor samples.

5.6 Balance, with precision ± 0,01 g.

5.7 Flat spatula.

6 Test procedure

6.1 Test specimen preparation

Two methods of sample preparation are described: the addition of binder method and the backing method. Either method may be used.

NOTE The addition of binder method is most used but the preparation of the precision statement showed that several different methods of sample preparation gave good and sufficient within-laboratory repeatability.

See ISO 1619 for guidance.

6.2 Addition of binder method

6.2.1 Dry the sample to constant mass at (110 to 120) °C.

6.2.2 Take a test portion and weigh it, taking care to always use the same mass as for the calibration reference material. A typical test portion is 10,0 g.

6.2.3 Mix the test portion with binding agent (see the list of examples in 4.5) before grinding in the disc mill (5.2), taking care to always use the same mass and proportion of sample and binding agent as for the calibration reference materials. Some examples of binders in use are given in Table 2.

NOTE The binding agent can decrease the level of segregation in the sample and make it easier to remove the tablet from the grinding vessel.

Table 2 — Some examples of test portion size and binding agents (4.5) in the addition method

Test portion g	**Binder addition**[a]
10,0	8 pills Hoechst PE-190 amounting to 1,0 g
5,0	+ 4,0 ml Dupont Vertrel XF Cleaning Agent (MS-782) + 0,5 g binder tablet Premier Lab Supply (XRF Multi-Mix RXR-250)
10,0	2,5 g of 9:1 BASF styrene EMU 120 FD: Hoechst Ceridust 9615A Wax
4,0	2,0 g binder (Lico wax C Micropowder)
[a] These materials can be replaced with products with similar properties	

6.2.4 Mill (5.2) the sample until all particles are below 63 micrometer fineness.

NOTE 1 Examples of milling times are 10 s to 10 min, typical is (1 to 4) min. Short milling times give larger spread in within-laboratory precision. The particle size should be determined by using a suitable sieving technique.