
**Carbonaceous materials used in
the production of aluminium — Green coke
and calcined coke for electrodes —
Analysis using an X-ray fluorescence
method**

iTeh STANDARD PREVIEW
*Produits carbonés utilisés pour la production de l'aluminium — Coke
calciné et coke cru pour électrodes — Analyse par fluorescence aux
rayons X*
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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 12980 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium oxide, cryolite, aluminium fluoride, sodium fluoride, carbonaceous products for the aluminium industry*.

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Introduction

The determination of the elemental impurities is important for reasons of metal quality and anode consumption. This International Standard refers only to petroleum coke although the principles described are valid for pitch, anthracite, graphite or electrode materials. As the sample preparation and/or the mass fraction ranges for these other materials are quite different it is intended that specific standard methods will be published for each material.

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Carbonaceous materials used in the production of aluminium — Green coke and calcined coke for electrodes — Analysis using an X-ray fluorescence method

1 Scope

This International Standard specifies an X-ray fluorescence method for the determination of elemental impurities in green and calcined petroleum cokes used for the manufacture of anodes. These anodes are used in the production of aluminium.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 6375, *Carbonaceous materials for the production of aluminium — Coke for electrodes — Sampling*.
<https://standards.iteh.ai/catalog/standards/sist/9c5641ed-b636-4dd3-853a-a0a4671e7cab/iso-12980-2000>

3 Principle

A pressed tablet of pulverized coke and organic binder is irradiated by X-rays from a target thin window X-ray tube. The X-ray tubes can be chromium, rhodium or scandium depending on which elements are to be determined.

Irradiation of the test specimen causes ejection and rearrangement of orbital electrons resulting in the emission of secondary radiation with a characteristic wavelength for each element.

This secondary radiation is reflected, onto the detector system, by a crystal, which is set at a specific angle to the secondary radiation. Only specific radiation which obeys Bragg's law reaches the detector system.

$$n\lambda = 2d \cdot \sin \theta \quad (1)$$

where

n is the order of the diffraction;

λ is the X-ray wavelength;

d is the interplanar distance in the crystal;

$\sin \theta$ is the angle setting of the crystal.

The intensities of the specific secondary radiation are calculated into mass fractions according to given calibration curves.

4 Apparatus and materials

4.1 X-ray fluorescence spectrometer, equipped with the following crystals: LiF₂₀₀ (lithium fluoride, reflecting plane 200), PE (pentaerythiol), PX1 (synthetic W-Si), Ge, TIAP (thallium acid phthalate) and LSM (layered synthetic microstructure). For instruments equipped with a side window X-ray tube, a scandium tube is advantageous for light elements. For instruments equipped with an end window X-ray tube, a rhodium tube offers optimum sensitivity.

4.2 Swinging-disc mill, with tungsten-carbide grinding devices.

4.3 Organic binder, suitable for pressed-tablet preparation. The binder shall not contain any of the elements to be analysed. Suitable binders are 'Hoechst wachs G' or stearic acid.

4.4 Tablet press, capable of providing a 20-tonne load.

4.5 Appropriate sample holders, for example aluminium dishes or brass rings with the dimensions as shown in Figure 1.

4.6 Detector gas, consisting of 90 % argon and 10 % methane.

4.7 Calibration standards, commercially available and certified covering the range of mass fraction given in clause 6.5.

4.8 Reference samples (RS), commercially available and certified with appropriate mass fractions for all elements. (See 6.4.2)

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Dimensions in millimetres

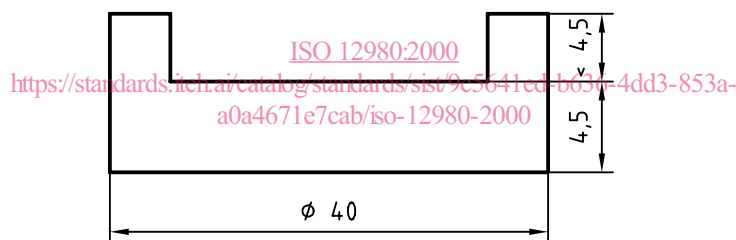


Figure 1 — Dimensions of sample holder

5 Sampling and test specimen preparation

5.1 Sampling

Sampling shall be in accordance with the requirements of ISO 6375.

5.2 Test specimen preparation

Crush the sample into sufficiently small particles so as to pass it through a 4 mm sieve. Dry to constant mass at 120 °C. Mill the sample of crushed material into sufficiently small particles so as to pass it through a 63 μ m sieve. Mix approximately 30 g of this sieved material with approximately 5 g of binder and remill for 20 s. Crush any remaining lumps with a spatula.

If too many lumps are formed in this mixture, repeat the above procedure with a smaller quantity of binder, for example 4 g. When a smaller quantity of binder is used, test it to see if the calibration sample and other samples give pressed tablets strong enough to withstand the procedure. Repeat the above procedure until a suitable quantity of binder has been added to provide an acceptable mixture for pressing a tablet.

Place the material into the selected sample holder.

NOTE Aluminium dishes are preferred, but brass rings may also be used.

Place the filled sample holder in the press and press at 20 t for 20 s.

For the best results, the test specimen should be at least 4 mm thick.

6 Procedure

6.1 Measurement conditions

The measurement conditions are apparatus-dependent but the indications in Table 1 can serve as guidelines. Normally no line overlapping will be experienced if these conditions are used for the measurement of petroleum coke.

Table 1 — Typical measurement conditions for the different elements

Element line	Na K _α	Al K _α	Si K _α	S K _α	Ca K _α	V K _α	Fe K _α	Ni K _α
Crystal	LSM ^a or PX1	PE or PX1	PE or PX1	PE or Ge	LiF200	LiF200	LiF200	LiF200
Counter	F ^b	F	F	F	F	F	F	F
Exposure, total time, s	100	20	20	10	10	20	30	20
Collimator	coarse	coarse	coarse	medium	fine	fine	fine	fine
kV	40	40	40	40	40	40	60	60
mA	60	60	60	60	60	60	40	40
Angle	28°	PE: 145,1° PX1: 19,5°	PE: 109,2° PX1: 16,6°	PE: 75,8° PX1: 110,9°	113,1°	76,9°	57,5°	48,7°
Offset angle ^c	±2°	—	—	—	—	+1°	+1°	-1°

F = flow counter

^a Layered synthetic microstructure with 2d value around 5 nm.
^b A 1 μm or 2 μm flow counter window is required.
^c Background measurement angles (± offset angles).

6.2 Intensities and background corrections

The net peak intensity I , expressed as the number of counts per second, of the element of interest is calculated as the difference between the measured peak intensity of the element and the background intensity at the peak angle in accordance with equations (4) and (5). See Figure 2.

$$I = I_p - I_b \quad (2)$$

where

I_p is the count rate, expressed as the number of counts per second, at the element position (2θ);

I_b is the background count rate, expressed as the number of counts per second, at the element position (2θ);