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**Carbonaceous materials used in the  
production of aluminium — Pitch for  
electrodes — Sampling**

*Produits carbonés utilisés pour la production de l'aluminium — Brais pour  
électrodes — Échantillonnage*

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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 6257 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*.

This second edition cancels and replaces the first edition (ISO 6257:1980), which has been technically revised.

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## Introduction

Sampling is a vital step in analysis and testing. Its importance is recognized in this International Standard which specifies comprehensive methods of sampling the grades of pitch used for the electrolytic production of aluminium.

Such samples should be as representative as possible of the materials sampled (whether from the whole or part of a batch or consignment) and in a form that facilitates the determination of the distribution of values of properties.

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# Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Sampling

**WARNING** — This International Standard may involve the use of hazardous materials, operations and equipment. This International Standard cannot address all the safety implications associated with its use. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and assess the applicability of regulatory limitations prior to use.

## 1 Scope

This International Standard specifies methods for sampling and preparing samples prior to testing of binder pitch used in the manufacture of electrodes for the electrolytic production of aluminium.

These methods are applicable to grades of pitch in liquid or solid form, the latter having softening points higher than 30 °C (determined according to the method specified in ISO 5940), in bulk, or in a number of containers making up one batch at sites of manufacture, storage, or delivery. Sampling methods and sampling plans for large consignments, or lots, of pitch in liquid form during the loading and unloading of ships are included.

## 2 Normative references

The following normative documents contain 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 editions of the normative documents 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 3165, *Sampling of chemical products for industrial use — Safety in sampling*

ISO 5940, *Carbonaceous materials for the production of aluminium — Pitch for electrodes — Determination of softening point by the ring-and-ball method*

## 3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

### 3.1

#### sampling unit

defined quantity of material having a boundary which may be physical, for example a container, or hypothetical, for example a particular time or time interval in the case of a stream of material

NOTE 1 A number of sampling units may be gathered together, for example in a package or box.

NOTE 2 In French, the term “individu” is sometimes used, a synonym of “unité d'échantillonnage”. In English, the terms “individual”, “unit” and “item” are sometimes used in practice as synonyms of “sampling unit”.

[ISO 6206]

### 3.2

#### **sample**

one or more sampling units taken from a larger number of sampling units, or one or more increments taken from a sampling unit

[ISO 6206]

### 3.3

#### **representative sample**

sample assumed to have the same composition as the material sampled when the latter is considered as homogeneous whole

[ISO 6206]

### 3.4

#### **sampling plan**

planned procedure of selection, withdrawal and preparation of a sample or samples from a lot (see 3.6) to yield the required knowledge of the characteristic(s) from the final sample (see 3.10) so that a decision can be made regarding the lot

NOTE Considerations of cost, effort and delay usually determine an acceptable sampling error.

[ISO 6206]

### 3.5

#### **consignment**

quantity of material covered by a particular consignment note or shipping document

[ISO 6206]

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### 3.6

#### **lot**

total quantity of material to be sampled using a particular sampling plan

NOTE A lot may consist of consignments, batches or items.

[ISO 6206]

### 3.7

#### **batch**

definite quantity of material that may be one item or a number of items which belong together because of their manufacture or production under conditions which are presumed to be uniform

[ISO 6206]

### 3.8

#### **bulk sample**

collected set of samples which do not maintain their individual identity

[ISO 6206]

### 3.9

#### **incremental sample**

one of a series of samples taken during the sampling process

NOTE Incremental samples may or may not retain their individual identity depending on the sampling instructions.



**3.10****final sample**

sample obtained or prepared under the sampling plan for possible subdivision into identical portions for testing reference or storage

[ISO 6206]

**3.11****laboratory sample**

sample as prepared for sending to the laboratory and intended for inspection or testing

[ISO 6206]

**3.12****reference sample**

sample prepared at the same time as, and identical with, the laboratory sample, which is acceptable to the parties concerned and retained for use as a laboratory sample if a disagreement occurs

[ISO 6206]

**3.13****spot sample**

sample of specified number or size taken from a specified place in the material or at a specified place and time in a stream of material and representative of its own immediate or local environment

NOTE In English, the term "snap sample" is sometimes used as a synonym for "spot sample".

[ISO 6206]

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**4 General procedures and precautions**

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**4.1 Sampling methods**

Manual and automated sample-taking are equally acceptable. Details of the methods employed shall be described in the sampling report (see clause 10).

**4.2 Contamination of sample or of pitch being sampled**

The sampling procedure shall not cause contamination of the sample or of the pitch being sampled.

Sample containers, sampling apparatus and any ancillary gear shall be dry.

Sample containers, sampling apparatus, any ancillary gear, the hands and gloves and the protective clothing of the sampler shall be clean.

Pitch undergoes slow surface oxidation in the presence of air. Finely divided products, because of their large surface area per unit mass, may show a significant rise in softening point even if stored for only a short time. It is recommended therefore that finely divided reference samples be prepared for storage by melting and resolidifying as follows.

- a) Place a sufficient amount of the powdered sample in a suitable container with a loose-fitting lid in an oven controlled at approximately 50 °C above the expected softening point of the pitch. Leave in the oven for 2 h at this temperature. After heating, the surface of the melt should be smooth, shiny and free of skin.
- b) If the surface of the melt is covered with froth, suggesting the presence of water, discard it and prepare another melt using a further portion of the sample which has first been dried by allowing it to stand in an evacuated desiccator in the presence of a suitable desiccant for approximately 2 h.

- c) Pour the molten pitch without turbulence, so as to avoid entrapping air bubbles, into an air-tight metal container, allowing it to solidify and seal the container.
- d) Note any froth formation during melting in the sample report and, in such cases; retain a separate portion of the finely-divided sample, stored in a sealed air-tight container, for the determination of water content.

If a sample container is opened, it shall be securely closed again as soon as possible and any damaged sealing rings shall be replaced.

### 4.3 Sampling for the determination of water content of pitch in solid form

For bulk shipments of solid pitch, a series of spot samples shall be taken for determination of water content, either from the conveying system during charge or discharge, or from the hold or compartment, or lorry at the top, middle or bottom during charge or discharge. The individual samples shall be sealed immediately in air-tight containers and tested promptly to avoid loss of moisture.

### 4.4 Mass of laboratory and reference samples

The mass of the laboratory and reference samples shall be at least three times the amount required for testing and shall never be less than 1 kg unless otherwise agreed in accordance with an established quality procedure. The sample mass shall be included in the sampling report (see clause 10).

The minimum recommended sample mass is 2 kg.

### 4.5 Safety precautions

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Comprehensive safety instructions are given in ISO 3165. Attention is drawn in particular to the following.

- When sampling hot liquid pitch from large containers such as ships, road or rail tanks, the sampler shall closely follow the safety procedures laid down for that site or location. These procedures typically include protective equipment and guidelines on safe working practice.
- Avoid inhalation of pitch vapour and dust.
- Sampling from ships, road or rail vehicles shall be strongly discouraged when in motion or when motion is likely to begin.

### 4.6 Suspect consignment

A consignment shall be considered suspect if:

- a) a container is damaged or defective;
- b) there is any doubt as to the nature of the contents of a container, for example because of the presence of an old label or incorrect markings;
- c) there is evidence of an unexpected lack of uniformity;
- d) there are obvious and unusual variations observed in the consignment.

Such samples shall be fully reported and shall not be regarded as acceptable without mutual agreement between the parties concerned.

NOTE As pitch is a supercooled liquid, some advantage is gained during crushing the sample and cleaning of apparatus by pre-refrigerating the sample.

Any work applied to the sample (e.g. punning) results in heat generation, caking and consequent segregation of the sample.

Preparation of low softening point pitches is also possible under cryogenic conditions.

## 5 Sampling of solid pitch

### 5.1 General considerations

Table 1 indicates the minimum amount of representative sample that shall be withdrawn initially from a container or bulk as a series of incremental samples (see 4.4) unless the quantity to be sampled is very large (see 5.2).

**Table 1 — Minimum representative sample size**

Material mass tonnes	Representative sample mass kg
1 to 10	10
10 to 50	15
50 to 100	20

Each incremental sample taken shall have a minimum mass of approximately 1 kg and a preferred mass of 2 kg unless otherwise agreed in accordance with an established quality procedure. Large sampling errors may arise if the material being sampled is inhomogeneous and under such circumstances, it will be necessary to increase the sample size.

Special considerations will also apply if the quantity to be sampled is small (approximately 1 t or less) or very large (approximately 1 000 t or more).

If the quantity of material to be sampled is more than approximately 1 t but less than approximately 100 t, then no fewer than 10 samples shall be taken to allow for the heterogeneity of the material. Refer to Table 1. The mass of each sample taken from quantities of 1 t to 100 t approximately shall be between 0,5 kg and 1 kg, but shall be at least 20 times the mass of the largest particle.

In the case of large quantities not in containers, several 10 kg samples shall be taken. The procedure specified in 5.2.2 shall be used to determine the number of incremental samples that shall be taken to form the representative sample.

### 5.2 Sampling plan for large quantities of solid pitch

#### 5.2.1 Large quantities of solid pitch in containers

The minimum number of items to be sampled is given in Table 2.

#### 5.2.2 Consignment of solid pitch not in containers

Use Figure 1 to determine the number of 10 kg incremental samples required to represent the lot or consignment.

### 5.3 Practical procedures for taking samples of solid pitch

#### 5.3.1 Coarse pitch (including pencil, rod, plate and similar-formed pitch)

##### 5.3.1.1 General considerations

This type of material is likely to show the greatest variation of composition within its container. Particular care shall therefore be taken in obtaining a representative sample. Larger samples are necessary for materials of large particle size or size range. A suitably sized scoop may be used for sampling, preferably of width at least six times the diameter of the largest particles. An open-ended or closed-end sampling spear (see Figures 2 and 3) may be used as alternative sampling tools.