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Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Determination of volatile matter content

Produits carbonés utilisés pour la production de l'aluminium — Brais pour électrodes — Détermination de la teneur en matière volatile

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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 12977 was prepared by Technical Committee ISO/TC 47, *Chemistry*, Subcommittee SC 7, *Aluminium oxide, cryolite, aluminium fluoride, sodium fluoride, carbonaceous materials for the aluminium industry*.

Annex A of this International Standard is for information only.

Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Determination of volatile matter content

1 Scope

This International Standard describes an empirical method for the determination of the volatile matter content of pitch by gas chromatographic analysis of its solution in toluene. Strict adherence to the procedure is essential because of the empirical nature of the method.

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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

ISO 6257, *Carbonaceous materials used in the production of aluminium — Pitch for electrodes — Sampling.*

3 Term, definition and symbols

3.1 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1.1
volatile matter content
total percentage by mass of those components present in the pitch which have gas-chromatographic retention times corresponding to atmospheric pressure boiling points up to 360 °C under the conditions used

3.2 Symbols, subscripts and superscripts

3.2.1 Symbols

A surface area of the chromatographic peak

a slope of the regression line

B equivalent boiling point at atmospheric pressure

b intercept of the regression line

F factor to convert tabulated peak areas to the corresponding percentages by mass in the original pitch sample

K_A ratio of areas of dibenzothiophene to phenanthrene

K_T ratio of retention times

m mass of calibrant compound

R reproducibility

R_f response factor for calibrant compounds (5.2)

r repeatability

T retention time of the chromatographic peak

w_d percentage by mass of dibenzothiophene present in sample

3.2.2 Subscripts and superscripts

A, B, C solutions A, B, C corresponding respectively to chromatograms A, B, C

d dibenzothiophene

i i-th peak

p phenanthrene

pitch pitch

T retention time

tot total surface area of calibrant compounds in the chromatogram

0 presence in the original pitch sample

′ contribution resulting from the addition of the dibenzothiophene internal standard

4 Principle

A calibration analysis of a multicomponent test mixture of pure compounds comprising selected major components of coal tar together with an internal standard component is performed by gas chromatography under defined conditions on a chromatographic column having a non-polar stationary phase.

The results of this analysis establish a substantially rectilinear relationship between the chromatographic retention times of the individual components of the mixture and their atmospheric pressure boiling points. In addition, the analysis provides a mean relative response factor which relates the ratio of the total mass to total chromatographic peak area for the selected calibrants to the equivalent ratio for the internal standard.

Because the selected internal standard compound, dibenzothiophene, is also a normal component of coal tar it is necessary to perform the ensuing chromatographic analysis of the pitch on two solutions, one with and one without addition of the internal standard, thus enabling the area to mass response of the chromatograph to the internal standard to be calculated.

From the latter figure and from the mean relative response factor for the components in the calibration test mixture the amounts of material represented by the peaks of the test sample analysis chromatogram are calculated. These data, together with the previously established relationship between the chromatographic retention time and the