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Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of particle size distribution

*Produits carbonés utilisés pour la production de l'aluminium — Coke
calciné — Détermination de la granulométrie*



Reference number
ISO 12984:2000(E)

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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 12984 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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Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of particle size distribution

1 Scope

This International Standard specifies a method for the determination of the particle size distribution of a sample of coke having a typical size distribution as normally produced by calcination of delayed petroleum coke. The same procedure can be applied to consignments of calcined anthracite. This International Standard cannot be applied to fractions of carbon material prepared by sieving and crushing or to filter fines.

This method is applicable to the determination of particle sizes ranging from 0,25 mm to 16 mm, as the sum of the percentages of the size distribution above and below this range is typically less than 10 %. This International Standard is not applicable to determining particle sizes below 0,25 mm where a specific test for dust should be used.

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 1953, *Hard coal — Size analysis by sieving*.

ISO 2325, *Coke — Size analysis (Nominal top size 20 mm or less)*.

ISO 2591-1, *Test sieving — Part 1: Methods using test sieves of woven wire cloth and perforated metal plate*.

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*.

ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*.

ISO 6375:1980, *Carbonaceous materials for the production of aluminium — Coke for electrodes — Sampling*.

3 Principle

A sample of coke is subjected to a process of particle size analysis by dry sieve testing. The results are expressed in terms of the measured percentage by mass of the coke remaining on sieves of different sized openings between 16 mm and 0,25 mm, or alternatively in terms of calculated cumulative percentages for the different sized openings.

4 Apparatus

4.1 Wire cloth test sieves, of the following opening sizes: 16 mm, 8 mm, 4 mm, 2 mm, 1 mm, 0,5 mm and 0,25 mm.

The diameter of the sieves shall not be less than 200 mm and preferably 400 mm. The height shall be a minimum of 30 mm. The sieves shall comply with ISO 565, ISO 3310-1 and ISO 3310-2.

All specified sieves shall be used to prevent any one sieve overloading.

4.2 Batch-type sieve shaker, which oscillates the particles in short time periods with minimal particle breakage. Shaking by hand is not appropriate.

4.3 Balances, capable of weighing the mass of one sieve plus the mass of the sample, such that the sample weighing error is less than 0,1 %.

4.4 Drying oven, for temperatures up to at least 120 °C, and **drying dishes**, made from stainless steel or aluminium or another suitable material.

5 Sampling and preparation of test portion

5.1 Take a representative sample of the coke load in accordance with ISO 6375.

NOTE 1 The sample is normally split between the supplier and the customer and 4 kg of the material is used for the size analysis in each laboratory (see 7.2 in ISO 6375:1980).

NOTE 2 Care should be taken to ensure that the particle sizes are not altered during sampling and sample preparation including drying.

5.2 Take a test portion from the representative sample in accordance with the quantities in Table 1.

NOTE The test portion size is a function of sieve size to avoid overloading the sieves.

Table 1 — Relationship between sieve size and test portion size

Sieve diameter mm	Test portion g
200	500 ± 100
300	1 000 ± 200
400	2 000 ± 400

Take the amount shown in Table 1 from the representative sample (see 5.1) using one of the methods of sample division described in ISO 6375.

6 Procedure

Dry the test portion at a temperature of 110 °C ± 5 °C for 2 h or until consecutive weighings at 5 min intervals show less than 1 % loss in mass. Determine the mass by weighing the test portion to an accuracy of 0,1 %. Inspect the sieves and if blinding of the apertures is observed clean them with a stiff bristle or soft metal sieve brush. Assemble the sieves in a nest in descending order of size and fit to the receiver. Transfer the test portion to the top sieve, fit the lid and shake the nest of sieves for 10 min. Take care to retain the particles within the sieve. Weigh each sieve with its oversize material and repeat the sieving process for a further 5 min and weigh each sieve with its oversize material again. If the mass of each sieve with its oversize material differs by less than 1 % from the first weighing the sieving is complete. Otherwise repeat the operation until this is the case. Record the mass of sample retained on each size starting with the sieve of largest aperture. Check for overloading the sieves according to Table 2. If the maximum allowable mass on one sieve is exceeded repeat the procedure using screens with larger diameter or repeat the sieving with a smaller test portion.

Table 2 — Maximum allowable fractional mass on screens

Nominal width of opening of screen mm	Maximum allowable mass at end of sieving	
	200 mm	400 mm
16	100	400
8	100	400
4	250	1 000
2	250	1 000
1	150	600
0,5	120	500
0,25	100	400

7 Expression of results

Calculate and record the apparent loss in mass of the test portion, i.e. the difference between the total mass of the test portion before and after sieving. If the loss in mass is less than 1 % of the original mass of the test portion add it to the mass of the smallest size fraction. If the loss is greater than 1 % of the original mass of the test portion, reject the results of the size analysis test.

NOTE Loss of mass means loss of test portion, which should not occur.

Convert the mass retained on each sieve to a percentage of the total mass of the test portion. Calculate the cumulative percentage as shown in Table 3, rounding the results to the nearest 0,1 % mass fraction.

Table 3 — Example of the calculation of the cumulative percentage

Fraction size mm	Distribution percentage %	Size mm	Cumulative percentage %
> 16	0,0	> 16	0,0
16 to 8	9,5	> 8	9,5
8 to 4	22,4	> 4	31,9
4 to 2	17,8	> 2	49,7
2 to 1	16,3	> 1	66,0
1 to 0,5	15,3	> 0,5	81,3
0,5 to 0,25	10,9	> 0,25	92,2
< 0,25	8,0	< 0,25	100,2

8 Precision

8.1 Repeatability

The repeatability of the results obtained for the different individual fraction size is 10 % relative. For the cumulative percentage between 20 % to 80 %, the repeatability is 3 % absolute and, outside this range, 2 %.

8.2 Reproducibility

No reproducibility is quoted for determinations carried out in different laboratories because the transport of a coke sample involves the risk of breakage and thus alteration of the size distribution.

Specifications for “as dispatched” and “as received” materials, respectively measured by the producer and by the user, shall be mutually agreed.

9 Test report

The test report shall include the following information:

- a) all details necessary for identification of the product tested;
- b) a reference to this International Standard, i.e. ISO 12984:2000;
- c) the results and the units in which they are expressed;
- d) any operation not included in this International Standard or in any of the International Standards to which reference is made, as well as any operations regarded as optional;
- e) the date on which the test was carried out.

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