International Standard ISO 5069/2

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Brown coals and lignites — Principles of sampling — Part 2 : Sample preparation for determination of moisture content and for general analysis

Charbons bruns et lignites — Principes d'échantillonnage — Partie 2 : Préparation des échantillons pour la détermination de l'humidité et pour l'analyse générale

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5069/2 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels*, and was circulated to the member bodies in March 1982.

It has been approved by the member bodies of the following countries :

Australia	Egypt, Arab Rep. of	Poland
Austria	Germany, F. R.	Romania
Belgium	Hungary	Spain
Canada	India	Turkey
China	Korea, Rep. of	USSR

The member bodies of the following countries expressed disapproval of the document on technical grounds :

Czechoslovakia
Japan

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Brown coals and lignites — Principles of sampling — Part 2 : Sample preparation for determination of moisture content and for general analysis

1 Scope and field of application

This part of ISO 5069 specifies methods of preparation of laboratory and analysis samples of brown coals and lignites for the determination of moisture content and for general analysis.

This document should be read in conjunction with ISO 5069/1.

2 References

ISO 1213/2, *Solid mineral fuels — Vocabulary — Part 2 : Terms relating to coal sampling and analysis.*[1]

ISO 1988, *Hard coal — Sampling.*

ISO 5069/1, *Brown coals and lignites — Principles of sampling — Part 1 : Sampling for determination of moisture content and for general analysis.*

3 Sample

Usually a common sample (see ISO 1213/2) for both determination of moisture content and general analysis shall be taken. The moisture content sample shall be extracted from the common sample and the remainder used for general analysis. In particular cases, a special moisture content sample shall be taken. The types of samples are shown in figure 1.

4 General principles

The process of sample preparation consists of several operations three of which are fundamental :

a) particle size reduction by crushing or grinding and milling;

b) mixing;

c) decrease of sample mass by dividing the sample (sample division). In most cases, the drying process of the sample is also necessary. Sample preparation may be carried out as a one-stage or a two-stage procedure. Sample preparation for total moisture content determination may require that both of these procedures be employed, the one-stage procedure being considered preferable. Sample preparation for general analysis requires that the two-stage procedure be used. Methods and theory of checking sample preparation errors are detailed in ISO 1988.

5 Location

The area designated for sample preparation shall be enclosed, roofed, free from draughts, direct sunlight, and other atmospheric influences. A heating of the area is admissible only during the winter period to a temperature not exceeding 15 °C and the heating devices shall be isolated.

Samples shall be treated immediately after delivery, and stored in such a manner as to prevent contamination and loss of moisture.

6 Equipment

6.1 General principles

Sample preparation equipment (crushers, mills, cutting dividers and dividers), shall ensure

a) grinding the gross sample to produce a laboratory sample of 10 mm and 3,15 mm grain size as required, and dividing these samples into the quantities required;

b) milling the laboratory samples down to the analysis sample with a grain size of 1 mm and 0,212 mm and dividing into the quantities required.

6.2 Crushers, mills

These shall be of the high-speed type.

6.3 Dividers

Two types are used :

a) Riffle dividers. The slope of the device shall be inclined at not less than 30° to the vertical.

1) At present at the stage of draft. (Revision of ISO/R 1213/2.)

b) Mechanical sample-dividers, of the rotary or other types for which the slot-type receivers are placed on the turntable so as to intercept a falling stream of coal once or twice in each revolution or to collect a continuous stream of coal falling from the hopper on to the apex of a cone.

The aperture of all divider types shall be 2,5 times wider than the upper particle size.

6.4 Technological requirements

6.4.1 The divider shall separate at least one representative portion from the gross sample.

6.4.2 The minimum mass of the extracted portion shall be different for the moisture content sample and the sample for general analysis, and depends on the particle size of the coal (figures 2 and 3).

6.4.3 The ratio of division shall be close to 0,1, for each portion extracted.

6.4.4 The quality of sample shall not change during division, for example by loss of moisture.

6.4.5 The divider shall not cause bias in the determined parameters when operating with non-homogeneous materials.

6.4.6 When dividing moist coal, care shall be taken to avoid the sticking of coal to the sides of the divider.

6.5 Machinery requirements

6.5.1 The dividers may be of the one- or two-stage type (for high ratio of division).

6.5.2 The dimensions of the functional parts of the divider shall be such as to permit the passage of the entire sample without the elimination of any portion or clogging.

6.5.3 The speed of the movable parts of the divider shall be constant within 1 m/s.

6.5.4 The minimum dimension (width) of the through-flow cross-section shall be at least 2,5 D, and in any case not less than 50 mm.

6.5.5 For the sample dividers of the rotary type, the maximum aperture dimension shall be 100 mm.

7 Sample preparation for moisture determination

7.1 General principles

Care shall be taken to avoid loss of moisture due to evaporation during handling and transport. All moisture samples shall be kept in closed containers in a cool place before and after preparation of the sample, as well as during any interval between particular stages of sample preparation.

To avoid loss of moisture during sample reduction and division, these operations shall be carried out as quickly as possible.

7.2 Moisture sample preparation from common sample

The moisture sample may be extracted before or after reduction as convenient, according to the scheme shown in figure 2.

7.2.1 Extraction of moisture sample before reduction

Without previous mixing, tip out the common sample onto a plate to form a cone, taking care to minimize segregation. The cone shall then be flattened to form a circular layer, the thickness of which shall be such that the increments to be taken cover the full thickness. The moisture sample shall then be extracted by taking nine increments for the positions illustrated in figure 4. The total mass X of the extracted sample depends on the maximum particle size and is indicated in figure 2.

The sample preparation shall then be carried out according to the procedure shown in figure 2 depending on the kind of mill available :

a) When a closed mill is available, i.e. a mill completely enclosed to avoid loss of moisture or dust, and the moisture content allows it to be used, the sample shall be crushed directly to pass a 3,15 mm aperture square mesh sieve and divided to 500 g, this being the sample for the total moisture determination [figure 2 a)].

b) If a closed mill is not available, and the maximum particle size exceeds 20 mm, the sample shall be reduced mechanically so that it just passes 20 mm. If coal is smaller than 20 mm, this reduction is unnecessary. The sample shall then be divided to 2 kg. This sample shall be brought to approximate moisture equilibrium with the atmosphere according to 9.2, method A, the first stage of a two-stage method total moisture content determination. The sample shall then be crushed to pass a 3,15 mm aperture square mesh sieve and divided to 500 g. This is the sample for residual moisture content determination [figure 2 b)].

7.2.2 Extraction of moisture sample after reduction

The common sample may be, if desired, reduced directly to pass a 3,15 mm aperture square mesh sieve, provided a closed mill is available. The reduced sample shall be placed in dry containers and sealed.

7.3 Moisture sample preparation from special moisture content sample

The procedure shall be the same as for the preparation of the moisture content sample from part X of the common sample described in 7.2 [figures 2 a) and 2 b)]. The special moisture content sample cannot be used for the determination of other properties of coal.

8 Sample preparation for general analysis

The sample Z left after extraction of the moisture content sample from the common sample in accordance with 7.2

should be used for the preparation of the sample for general analysis. The procedure for sample preparation is illustrated in figure 3.

a) When a suitable mill is available and if the moisture content permits, the sample shall be reduced directly to particle size of 3,15 mm. The reduced sample shall be divided to 4 kg and brought into approximate equilibrium with the atmosphere, after which it shall again be divided to about 2 kg (laboratory sample).

b) If a suitable mill for milling coal to 3,15 mm is not available, the sample Z shall be crushed below 20 mm and divided to Y kg, depending on the maximum particle size and the expected ash content (figure 3). This amount of coal shall be brought into approximate equilibrium with the atmosphere and then reduced to below 3,15 mm particle size and divided to about 2 kg (laboratory sample).

From the laboratory sample prepared according to a) or b), three samples shall be extracted :

1) Sample for the determination of the tar yield and benzene-soluble extract[1] (mass approximately 500 g);

2) Sample for general analysis (mass approximately 500 g);

3) Sample for checking purposes (mass approximately 1 000 g).

The sample for the determination of tar yield and benzene-soluble extract shall be crushed to below 1 mm, with no further division. This sample shall also be used for the residual moisture determination for calculation of these yields on a dry basis.

The sample for general analysis shall be milled to below 0,212 mm and divided to 250 g as analysis sample. This sample shall also be used for the residual moisture content determination. If the direct gravimetric method is used for moisture content determination, the mass of the sample may be 150 g instead of 250 g as described.

All three laboratory samples shall be hermetically sealed in suitable containers.

9 Determination of loss of moisture on air-drying

9.1 General principles

Air-drying shall be carried out at room temperature or higher but not exceeding 40 °C, permitting free circulation of air above the samples but excluding dust. The procedure used for air-drying depends on the form in which coal is received.

9.2 Method A

If air-drying is to be carried out by the procedure specified in 7.2.1, weigh a dry tray and place the coal to be air-dried directly in the tray. Spread the coal evenly to a depth not exceeding 20 mm (except for lumps greater than this size). Weigh the tray with the coal. Carry out the weighing to an accuracy of 0,05 % of the original mass of coal. Allow the coal to air-dry until the loss of mass of the sample over a period of 2 h is less than 0,3 % of its original mass. Note the final mass of the tray with the air-dried coal and calculate the loss of moisture on air-drying W_{ex} as a percentage from the following equation :

$$W_{ex} = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the dry tray;

m_2 is the mass, in grams, of the tray with coal before air-drying;

m_3 is the mass, in grams, of the tray with coal after air-drying.

9.3 Method B

If the sample is delivered in a sealed tin and air-drying is required, weigh the container and the coal as received, before opening the tin, to an accuracy of 0,5 % of the combined mass. After weighing, transfer the coal to a dry tray and spread it evenly to a depth not exceeding 20 mm (except for lumps greater than this size). Weigh the tray with the coal. Allow the container, the lid and the coal to air-dry. Brush any adhering dried coal from the container and lid into the tray and weigh the dry empty container and its lid. Carry out the air-drying until the loss of mass of the coal over a period of 2 h is less than 0,3 % of its original mass. Return the coal from the tray to the container, replace the lid and reweigh the whole. Calculate the loss of moisture on air-drying W_{ex} as a percentage from the following equation :

$$W_{ex} = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 is the mass, in grams, of the dry empty container with lid;

m_2 is the mass, in grams, of the closed container with coal before air-drying;

m_3 is the mass, in grams, of the closed container with coal after air-drying.

NOTE — W_{ex} is the symbol used in ISO 5068 and is used for consistency.

1) It is proposed to use toluene in place of benzene in the method of ISO 975.

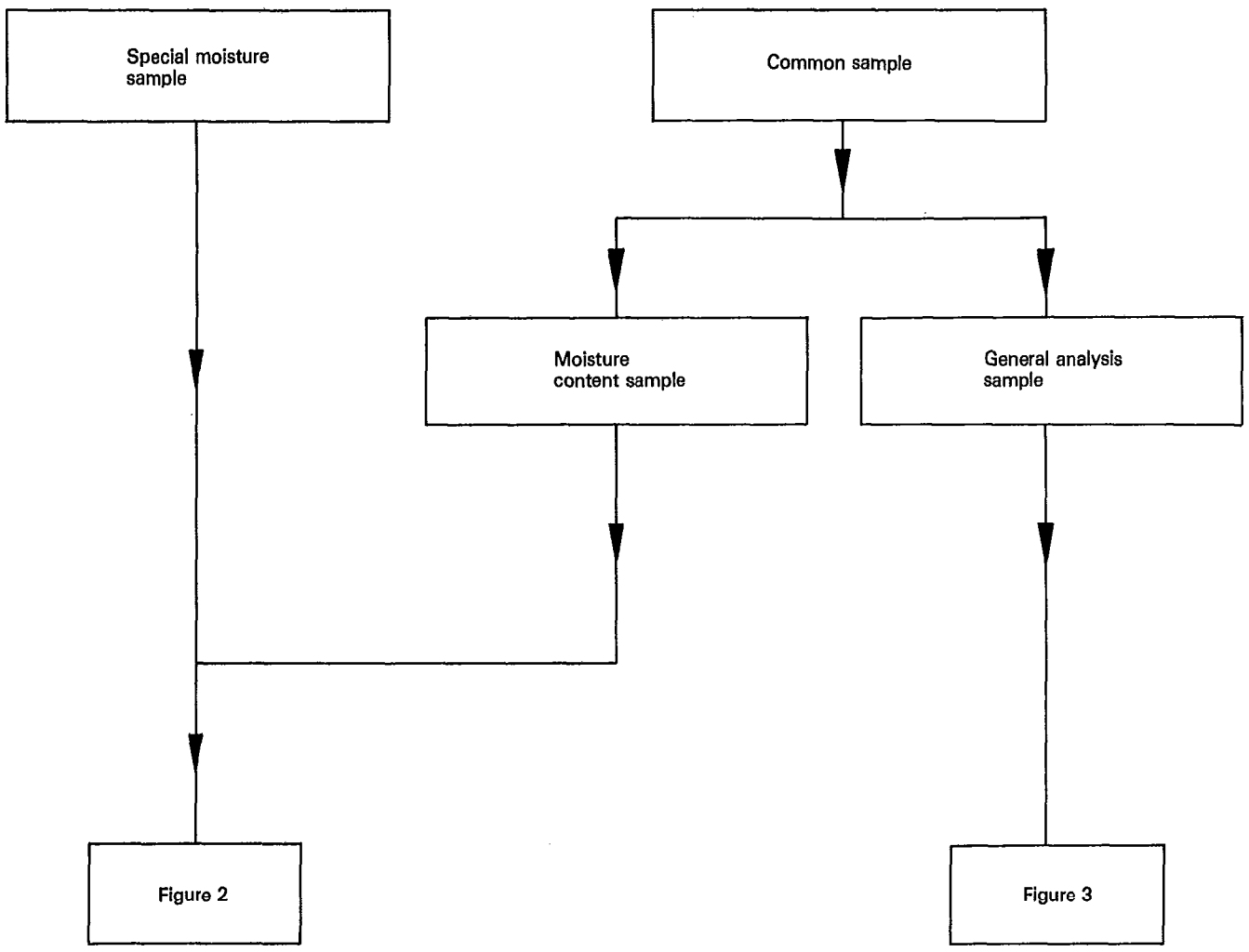
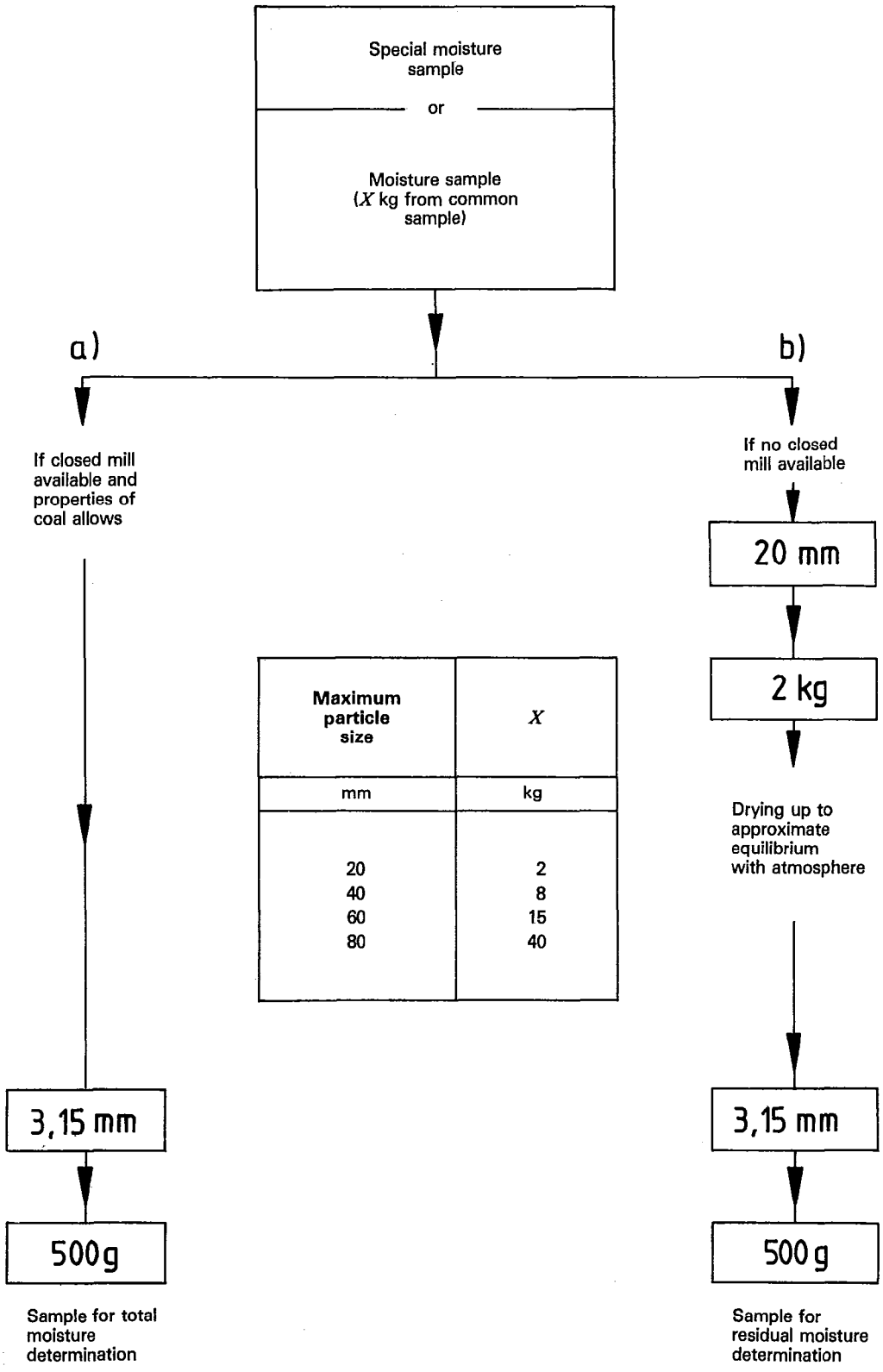
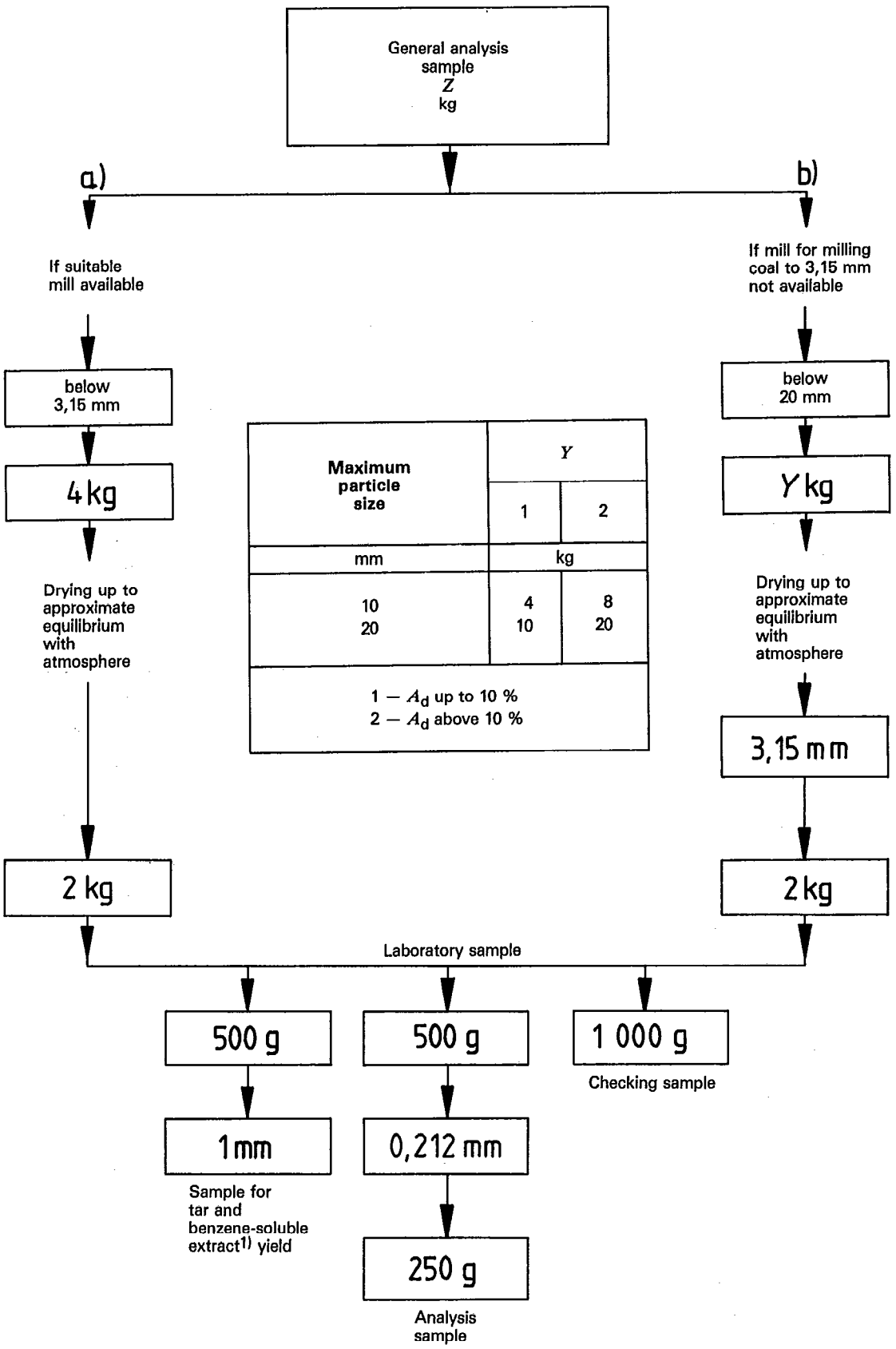


Figure 1 — Types of samples



Maximum particle size	X
mm	kg
20	2
40	8
60	15
80	40

Figure 2 — Sample preparation for moisture determination



Maximum particle size	Y	
	1	2
mm	kg	
10	4	8
20	10	20
1 — A_d up to 10 % 2 — A_d above 10 %		

Figure 3 — Sample preparation for general analysis

1) It is proposed to replace benzene by toluene in the method of ISO 975.

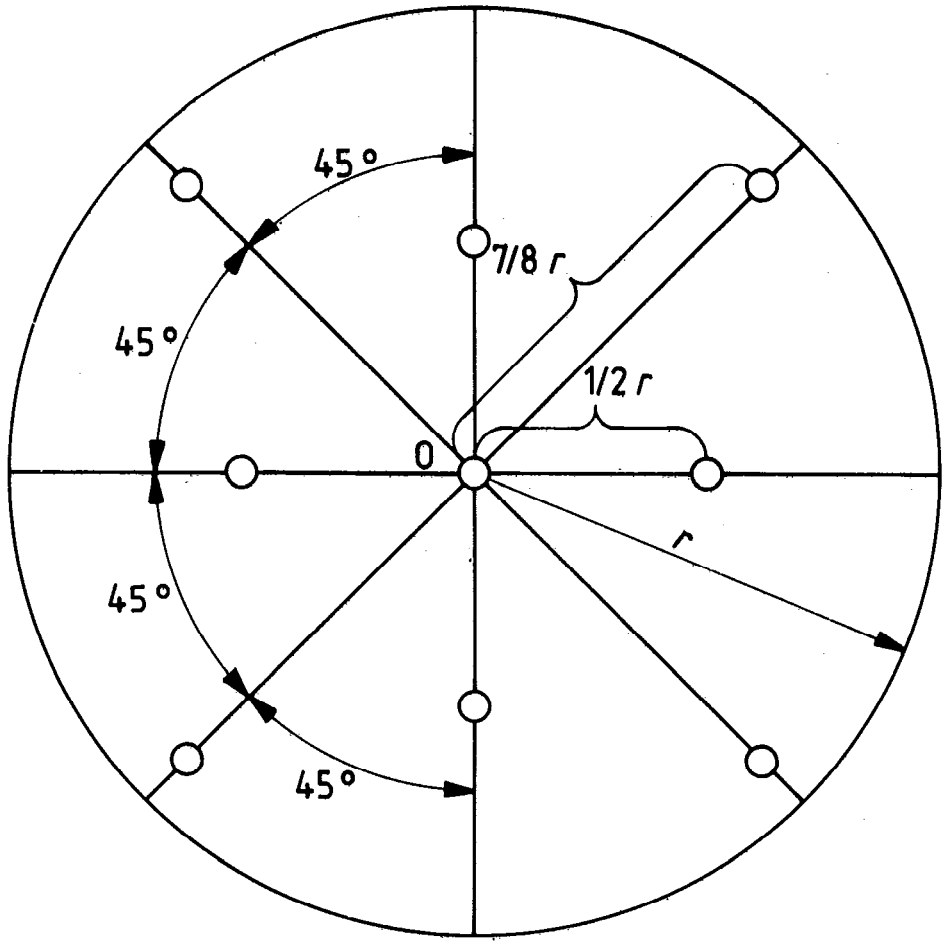


Figure 4 — Distribution of the nine increments to be taken from the flattened cone for moisture sample