

Transposed

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R 349

AUDIBERT-ARNU DILATOMETER TEST FOR COAL

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BRIEF HISTORY

The ISO Recommendation R 349, *Audibert-Arnu Dilatometer Test for Coal*, was drawn up by Technical Committee ISO/TC 27, *Solid Mineral Fuels*, the Secretariat of which is held by the British Standards Institution (B.S.I.).

Work on this question by the Technical Committee began in 1951 and led, in 1957, to the adoption of a Draft ISO Recommendation.

In May 1958, this Draft ISO Recommendation (No. 238) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Austria	Greece	Portugal
Belgium	India	Romania
Burma	Italy	Turkey
Canada	Japan	United Kingdom
Czechoslovakia	Mexico	U.S.S.R.
Denmark	New Zealand	Yugoslavia
Germany	Poland	

One Member Body opposed the approval of the Draft: Spain.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in December 1963, to accept it as an ISO RECOMMENDATION.

Amendment 1 – November 1971 – to ISO Recommendation R 349-1963

Amendment 1

to ISO Recommendation R 349-1963

AUDIBERT-ARNU DILATOMETER TEST FOR COAL

Section 4, PROCEDURE, clause 4.3

Replace the 1st paragraph by “The coal is heated at a rate of 3 °C per minute”.

In the 6th paragraph, 2nd line, replace “chosen” by “stated”.

BRIEF HISTORY

Amendment 1 to ISO Recommendation R 349-1963, *Audibert-Arnu dilatometer test for coal*, was drawn up by Technical Committee ISO/TC 27, *Solid mineral fuels*, the Secretariat of which is held by the British Standards Institution (BSI).

Draft ISO Recommendation No. 1932 was drawn up on this subject, and was circulated to all the ISO Member Bodies for enquiry in April 1970. It was approved by the following Member Bodies :

Australia	India	Sweden
Belgium	Iran	Switzerland
Canada	Italy	Thailand
Chile	Korea, Rep. of	Turkey
Czechoslovakia	Netherlands	U.A.R.
Denmark	New Zealand	United Kingdom
France	Poland	U.S.A.
Germany	Portugal	U.S.S.R.
Greece	South Africa, Rep. of	Yugoslavia

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as AMENDMENT 1 to ISO Recommendation R 349-1963.

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AUDIBERT-ARNU DILATOMETER TEST FOR COAL

1. OBJECT AND SCOPE

The Audibert-Arnu test is one of the parameters adopted for the International Classification of Hard Coals by Type of the United Nations Economic Commission for Europe. The object of the test is to determine the coking properties of hard coal or hard coal blends on the laboratory scale.

In principle, the test is not designed, nor can it be used, to indicate the pressures exerted by hard coals on the walls of industrial carbonization ovens.

2. PRINCIPLE AND TERMINOLOGY

A pencil made of powdered coal formed under pressure is inserted into a precisely calibrated narrow tube and topped by a calibrated steel rod (piston) which slides in the bore of the tube.

The whole is heated at a constant and definite rate.

By making regular readings of the displacement of the piston as a function of the temperature and expressing the displacements observed as percentages of the original length of the pencil, a curve of the type shown in Figure 1 below can be plotted.

The following items are characteristic (see note below):

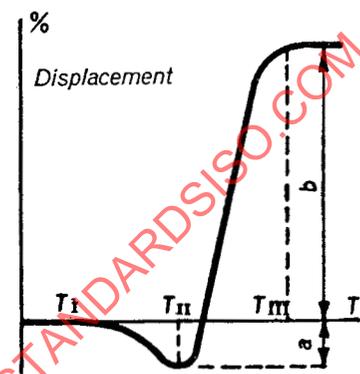


FIG. 1

T_I temperature at which the piston has moved down 0.5 mm *: *softening temperature*.

T_{II} temperature at which the piston reaches its lowest point: *temperature of maximum contraction*.

T_{III} temperature at which the piston reaches its highest point: *temperature of maximum dilatation*.

a maximum contraction, of the length of the pencil per cent.

b maximum dilatation, of the length of the pencil per cent.

If, after contraction has taken place, the piston does not return to its original level, the dilatation, equal, in absolute value, to the difference between the final level of the piston and the original zero level, is reported as negative.

NOTE. — The principal factors capable of distorting the results of this empirical test are the following:

(a) Deterioration of the coal, consequent on bad storage or faulty drying;

* Or 1 division, if the scale is calibrated in percentage of the standard length of pencil.

- (b) Deviation from the tolerances of
- (1) the internal dimensions of the dilatometer tube,
 - (2) the clearance between tube and piston,
 - (3) the mass of the piston,
 - (4) the dimensions of the mould;
- (c) Deviation from the specified mean rate and regularity of heating;
- (d) Deviation from the specification for the preparation of the sample in respect of maximum particle size, or for the pencil in respect of its length after tamping.

3. APPARATUS

3.1 Apparatus for preparing the coal pencil

- 3.1.1 *Mould*, polished internally, with accessories, as shown in Figures 2 and 2 A, pages 10 and 11.
- 3.1.2 *Gauge*, as shown in Figure 2, page 10.
- 3.1.3 *Ram*, of which Figure 3, page 12, shows an example.
- 3.1.4 *Press*, of which Figure 4, page 13, shows an example.

3.2 Dilatometer and accessories

- 3.2.1 *Dilatometer tubes and pistons*, as shown in Figure 5, page 14.

3.3 Other apparatus

- 3.3.1 *Electric furnace*, of which Figure 6, page 15, shows an example, consisting of a block of metal resistant to oxidation and of sufficiently high melting point. A suggested material is aluminium bronze. The metal block is pierced by at least two holes of 15 mm diameter by 350 mm deep to take the dilatometer tubes and one hole 320 mm deep to take a temperature-measuring device. The block is heated by a metallic winding, suitably insulated. Control gear permits the use of any selected rate of heating up to 5 deg per minute over a temperature range of 300 to 550 °C.

The furnace should be constructed so that the temperature conditions are uniform in the dilatometer tubes placed in their normal position in each hole.

To verify this, the furnace is heated at a rate of 5 deg per minute. When the temperature reaches about 450 °C, an exploration is made of the lower 180 mm of a dilatometer tube placed in the furnace, by comparing the readings on the normal temperature measuring device and a thermometric probe placed in the tube. The difference between the probe temperatures and the mean temperature shown by the normal temperature measuring device should be less than

$$\begin{aligned} &\pm 2 \text{ deg in the lower 120 mm,} \\ &\pm 5 \text{ deg from 120 to 180 mm.} \end{aligned}$$

This verification is not to be confused with the later calibration of the temperature measuring device; it is intended to measure only the temperature variation along the tube.

The furnace is equipped with an adjustable scale for each hole. The scale is preferably engraved on a mirror in front of which the indicator pointer of the piston moves. It is at least 180 mm in length and calibrated in millimetres or in percentages of the standard length of the pencil (60 ± 0.25 mm, see clause 4.2).

If desired, the apparatus can be equipped with an automatic heating regulator and a device for the automatic registration of the curve.

3.3.2 *Temperature-measuring device*, consisting of a mercury thermometer, or a thermocouple, or a resistance thermometer, accurate to within ± 1.0 per cent of the temperature in degrees centigrade (Celsius) and capable of being read, if necessary by estimation, to 1 deg.

3.3.3 *Cleaning implements*, consisting of the following:

3.3.3.1 Auger, diameter approximately 7.8 mm;

3.3.3.2 Reamer, consisting of a steel bar of semi-circular section of diameter 7.95 mm;

3.3.3.3 Brass wire brush, the diameter of which should slightly exceed 8 mm.

The total length of each of the cleaning implements should be 400 mm.

3.4 Calibration of apparatus

The apparatus is calibrated by comparing the temperature in a dilatometer tube in each hole with the temperature indicated by the temperature-measuring device in its normal position. The calibration is carried out at the desired rate of heating by using a thermocouple with wires of diameter approximately 0.6 mm, the thermojunction touching the wall of the tube 3 cm above the bottom. The temperatures read during the test are corrected by the differences found during this calibration.

3.5 Inspection

3.5.1 *Dilatometer*. In order to inspect the wear of the tube and piston after a hundred determinations have been carried out in one tube, the results of the next four determinations using that tube should be compared with those obtained in a new tube. This comparison will thus be made successively on four coals.

The difference in percentage dilatation between the two tubes should be divided by the "relative length" of the dilated pencil obtained with the new tube; the "relative length" is here expressed as the ratio of the length of the dilated pencil to its original length.

The figures so obtained from the four coals should be averaged. If the average is greater than 3.5, irrespective of sign, the old tube should be discarded (see Appendix). If the tube is still satisfactory, this comparison should be repeated after every subsequent 25 tests.

3.5.2 *Mould*. The wear of the mould is periodically checked with the gauge, which can also be used to check new moulds.

If, when the gauge is inserted in the larger orifice of the mould,

- (1) two lines can be seen on the gauge, the mould is too small and should be reamed out;
- (2) one line can be seen, the mould is satisfactory;
- (3) no line can be seen, the mould is worn and should be replaced.

4. PROCEDURE

4.1 Sampling and treatment of sample

As certain types of hard coal are very susceptible to oxidation, it is necessary to minimize the contact with air after reducing the gross sample.

As a special precaution, therefore, the part sample after reduction is stored in an oxygen-free nitrogen atmosphere or in freshly boiled water. In the latter case, a paste is made of the coal with water, the paste being put into a flask which is then filled to the top with freshly boiled water.

Care should be taken to ensure that the part sample taken for this test is truly representative.

A suitable relationship between the mass of the part sample and the maximum particle size is shown in the following table:

Maximum particle size	Minimum mass of part sample
millimetres	grammes
5	1000
4	500
3	250
2	100
1.5	50

If the coal has been stored under water, filter on a suction filter. Expose the coal on filter paper at a temperature not exceeding 40 °C until the coal appears to be dry, but in any case for not more than two hours.

Reduce the maximum particle size to 1.5 mm. Mix and take a part sample of 50 to 100 g. Crush to pass a 0.16 mm mesh sieve. Both crushing operations should be controlled so as to produce the minimum of fines (see note below). Mix again and carry out the determination on an average sample of about 10 g. Moisten this sample with 1 ml of water and mix rapidly. Too intensive mixing is liable to cause difficulty when the pencil is removed from the mould. For the same reason, it is essential that the preparation of the pencil should be carried out without interruption.

NOTE. — Too fine grinding of the coal affects the result of the determination. The sample should be crushed to obtain the following size analysis:

through 0.2 mm	100 per cent
through 0.1 mm	85-70 per cent
through 0.06 mm	70-55 per cent

4.2 Preparation of the coal pencil

Place the mould on its support with the larger orifice upwards and set the funnel on the mould. Place the coal in the funnel and lightly tamp into the mould without moving the funnel, by means of a tamping pin. Place the mould assembly under the ram in order to tamp the sample by dropping the plunger three or four times until the mass of coal ceases to yield. This should be repeated three or four times until the mould is filled.

In order to remove the coal pencil from the mould, remove the support and the funnel. Place the ejector guide at the end of the mould corresponding to the smaller diameter of the pencil. Place the guide tube at the other end of the mould and the receptacle in the guide tube. Then insert the ejector piston in the guide and push the coal pencil onto the receptacle by means of the press (see note below).

The length of the pencil is then adjusted to 60 ± 0.25 mm by cutting away as much as necessary of the thick end with a fine blade.

NOTE. — Particularly when dealing with coals which are difficult to remove from the mould, it is recommended that the ejector piston be removed from time to time and cleaned, the inner surface of the mould being cleaned at the same time.

4.3 Determination of dilatation

The coal may be heated at any desired rate up to 5 deg per minute, usually a rate of 1, 2 or 3 deg per minute is chosen. The rate of 3 deg per minute is used if the result is to be employed for international classification of hard coal.

Carefully insert the pencil, thick end first, into the dilatometer tube and push it very gently into position with the piston.

Place the tube and contents in one of the tubes of the metal block, when the temperature of the furnace is 330 °C. Place in any hole in the block which is not being used an empty tube complete with its piston. Where, in exceptional cases, T_r is less than 350 °C, charging of the furnace should take place when the temperature is 20 deg below T_r .

After the charged dilatometer tube has been inserted in the furnace, wait for the indicator pointer of the piston to reach a position of equilibrium before adjusting the zero of the scale. This position should be reached after about 5 minutes.

Immediately the dilatometer is placed in the furnace, the temperature begins to drop, and heating should be regulated so as to regain the temperature of 330 °C at the end of 7 to 10 minutes.

After 340 °C has been reached, the rise in temperature should be very steady, minute by minute, and equal to the rate chosen, with a tolerance of ± 3 per cent of the specified temperature rise in a 5-minute period (see note below).

During each 5-minute period, the operator should adjust the heating rate to correct any deviation observed in the preceding period, in order to avoid the accumulation of errors.

If the curve is not automatically recorded, note the time, the position of the piston and the temperature at intervals of not greater than 5 deg. In the region of the critical points, sufficient points should be plotted to determine the exact shape of the curve.

Continue heating for 5 minutes after the maximum dilatation is attained. Then stop heating and immediately remove the piston, in order to prevent its getting jammed.

The duplicate determination is carried out in a separate run.

NOTE. — The tolerance stated (± 3 per cent of the specified temperature rise in a 5-minute period) may not be attainable if the temperature-measuring device in use is such as to require a change of 1 deg to be estimated rather than read directly. In such cases, a tolerance of ± 1 deg per 5 minutes is recommended.

4.4 Cleaning of the tube and piston

It is essential that the test be carried out with the piston and the dilatometer tube scrupulously clean. The following method of cleaning is recommended:

- 4.4.1 *Tube.* Crush the semi-coke and remove as much of it as possible with the auger. Then fill the tube with crude benzol or other appropriate solvent and allow to soak for several hours. Complete the cleaning with the reamer, ensuring that no solid remains at the bottom or on the wall. Immediately before the test, clean finally with the brass wire brush.
- 4.4.2 *Piston.* Clean the piston, including the base, with very fine emery paper, taking care not to round the edges, and check that the piston slides freely in the tube.

5. CALCULATION AND EXPRESSION OF RESULTS

The observed changes in length are calculated as percentages of the initial length of the pencil.

When reporting the results, the following items should be recorded:

- (1) Whether the sample submitted to the test fulfils the conditions specified in clause 4.1.
- (2) The rate of heating employed.
- (3) A curve of the percentage changes in length as a function of temperature on a standard relative scale in which 10 deg on the horizontal scale is equal to 5 per cent on the vertical scale.
- (4) The corrected temperatures T_I , T_{II} and T_{III} rounded off to the nearest 5 deg.
- (5) The percentage contraction (a) rounded to the nearest whole number.
- (6) The percentage dilatation (b) rounded
 - to the nearest whole number for negative dilatation;
 - to the nearest 5 per cent for positive dilatation up to 100 per cent;
 - to the nearest 10 per cent for positive dilatation over 100 per cent.

6. ACCURACY OF DETERMINATION

Dilatation	Maximum acceptable difference between results obtained	
	in the same laboratory	in different laboratories
	$7 \left(1 + \frac{b}{100} \right)$ where b = maximum dilatation per cent	(see clause 6.2)

6.1 In the same laboratory

The results of duplicate determinations carried out at different times in the same laboratory, by the same operator, with the same apparatus, on representative portions taken from the same sample after the last stage of the reduction process, should not differ by more than the above value.

6.2 In different laboratories

No tolerance can be quoted for determinations carried out in different laboratories since insufficient evidence is available on which to base such a tolerance.

APPENDIX

INSPECTION OF DILATOMETER TUBES

After one hundred determinations have been carried out in one tube, the next four determinations should be made and duplicated in a new tube. The results should then be examined as follows:

If b_o = dilatation with the old tube,
 b_n = dilatation with the new tube.

Calculate the ratio:

$$x = \frac{b_o - b_n}{1 + \frac{b_n}{100}}$$

Then, if the average value of x for the four coals, taking into account the sign, is greater than 3.5 irrespective of sign, the old tube should be rejected.

Example 1

Coal	b_o	b_n	$b_o - b_n$	x
A	100	113	-13	-6.1
B	13	17	-4	-3.4
C	61	59	2	1.3
D	45	55	-10	-6.4

The average value of $x = -3.6$

The old tube should therefore be rejected.

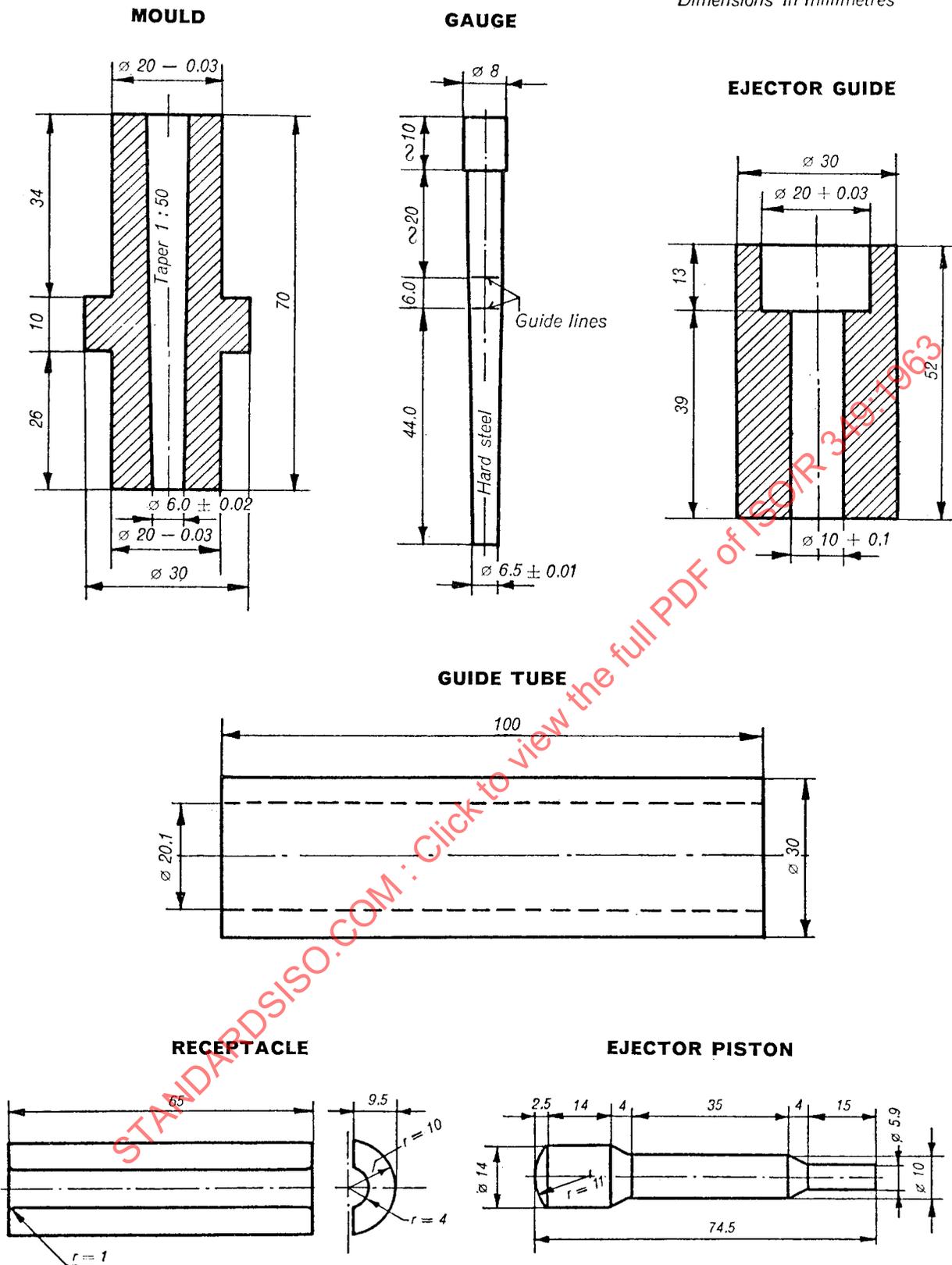
Example 2

Coal	b_o	b_n	$b_o - b_n$	x
E	54	56	-2	-1.3
F	81	80	1	0.6
G	109	117	-8	-3.7
H	40	44	-4	-2.8

The average value of $x = -1.8$

The old tube may therefore be used for a further 25 determinations, after which it should be re-inspected.

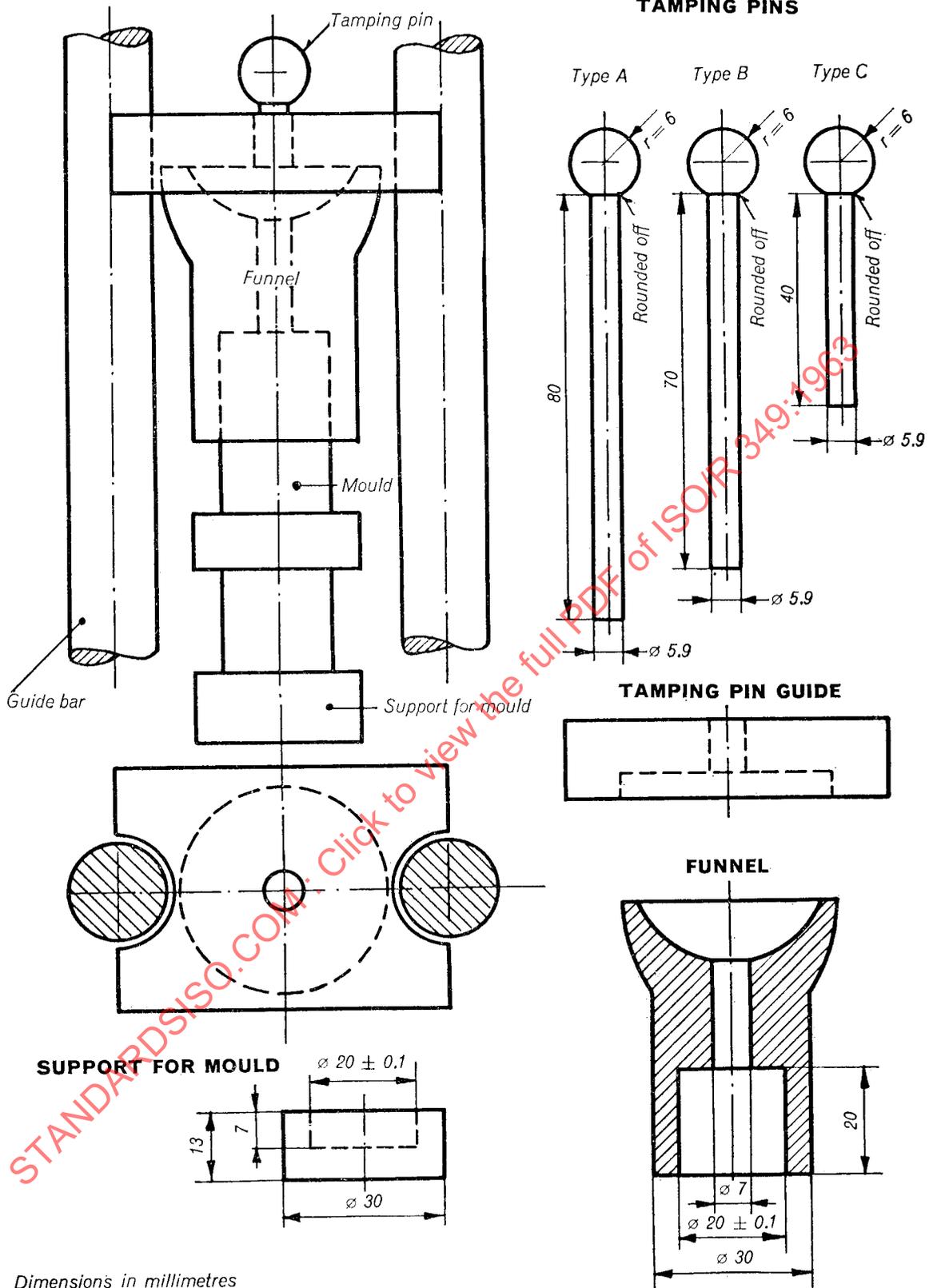
Dimensions in millimetres

**Material:**

Mould, ejector guide, guide tube, receptacle—hard bronze.

Gauge, ejector piston—vanadium steel.

FIG. 2. — Mould and accessories



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Dimensions in millimetres

Material:

Support, funnel—hard bronze.
Tamping pins—vanadium steel.

FIG. 2 A .— Mould and accessories (concluded)

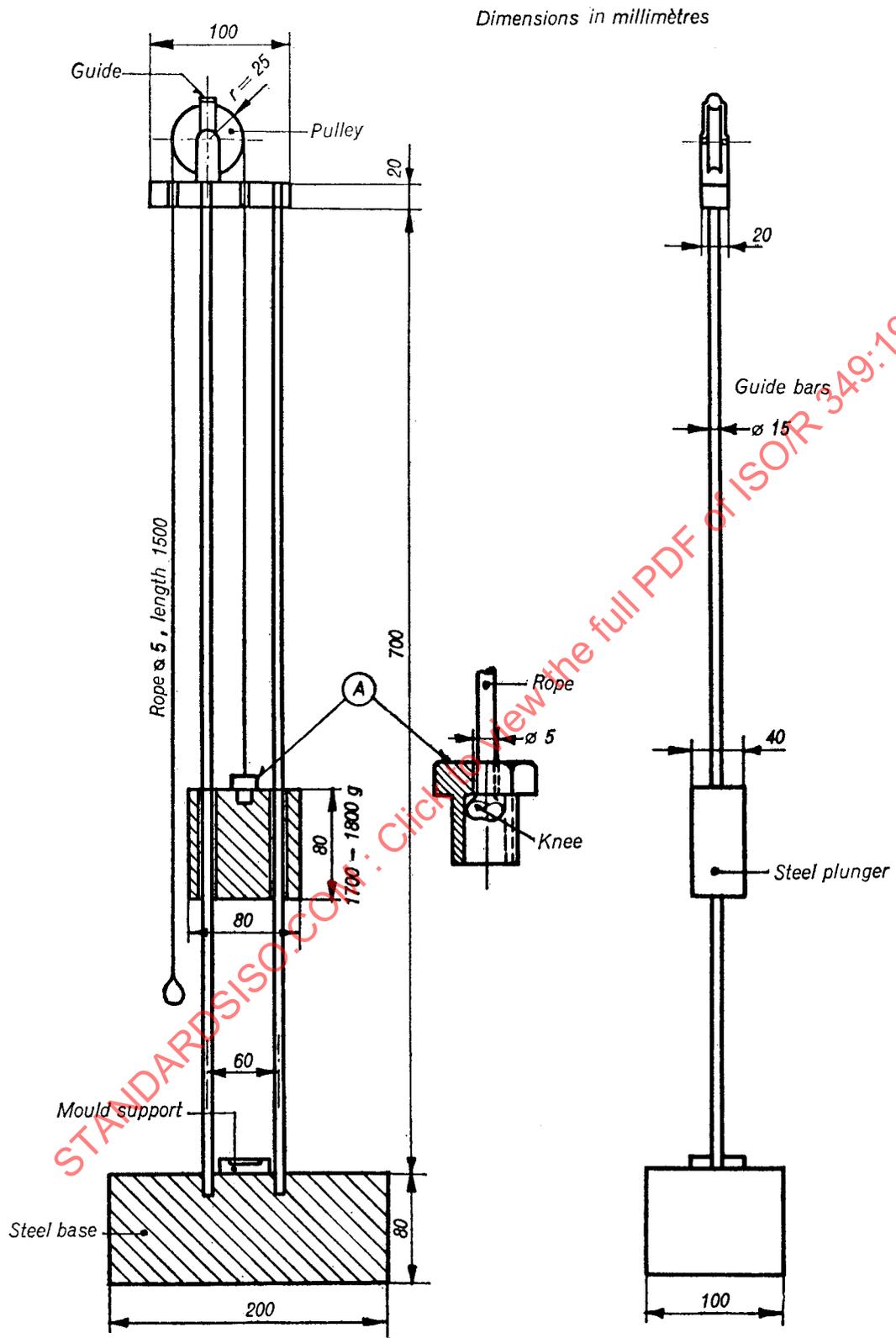


FIG. 3. — Ram