INTERNATIONAL STANDARD 349

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Hard coal — Audibert-Arnu dilatometer test

Houille — Essai au dilatomètre Audibert-Arnu

First edition — 1975-01-15

UDC 662.66 : 536.416

Ref. No. ISO 349-1975 (E)

Descriptors : coal, tests, physical tests, dilatometry, coking.

Price based on 11 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 27 has reviewed ISO Recommendation R 349 and found it technically suitable for transformation. International Standard ISO 349 therefore replaces ISO Recommendation R 349-1963 to which it is technically identical.

ISO Recommendation R 349 was approved by the Member Bodies of the following countries :

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

The Member Body of the following country expressed disapproval of the Recommendation on technical grounds :

Spain

No Member Body disapproved the transformation of ISO/R 349 into an International Standard.

Printed in Switzerland

Hard coal — Audibert-Arnu dilatometer test

0 INTRODUCTION

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.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the swelling properties of hard coal when heated under standard conditions in a dilatometer.

2 PRINCIPLE AND TERMINOLOGY

A pencil made of powered 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 can be plotted.

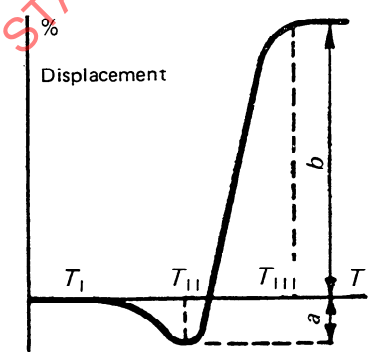


FIGURE 1

The following items are characteristic (see note below) :

T_I temperature at which the piston has moved down 0,5 mm[1] : *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 length of pencil, per cent.

b maximum dilatation of length of 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;

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; see figures 2 and 2A.

3.1.2 Gauge, see figure 2.

3.1.3 Ram, of which figure 3 shows an example.

3.1.4 Press, of which figure 4 shows an example.

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

3.2 Dilatometer and accessories

3.2.1 Dilatometer tubes and pistons, see figure 5.

3.3 Other apparatus

3.3.1 Electric furnace, of which figure 6 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 °C per minute over a temperature range of 300 to 550 °C.

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

To verify this, heat the furnace at a rate of 5 °C per minute. When the temperature reaches about 450 °C, make an exploration 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 shall be less than

± 2 °C in the lower 120 mm,
± 5 °C from 120 to 180 mm

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 shall be equipped with an adjustable scale for each hole. The scale shall be preferably engraved on a mirror in front of which the indicator pointer of the piston moves. It shall be 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 5.1).

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, a thermocouple or a resistance thermometer, accurate to within 1,0 % of the temperature in degrees Celsius and capable of being read, if necessary by estimation, to 1 °C.

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 shall slightly exceed 8 mm.

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

3.4 Calibration of apparatus

Calibrate the apparatus by comparing the temperature in a dilatometer tube in each hole with the temperature indicated by the temperature-measuring device in its normal position. Carry out the calibration 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 30 mm above the bottom. Correct the temperatures read during the test 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, compare the results of the next four determinations using that tube with those obtained in a new tube. This comparison will thus be made successively on four coals.

Divide the difference in percentage dilatation between the two tubes 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.

Average the figures so obtained from the four coals. If the average is greater than 3,5, irrespective of sign, discard the old tube (see annex). If the tube is still satisfactory, repeat this comparison after every subsequent 25 tests.

3.5.2 *Mould*

Check the wear of the mould periodically 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 shall be reamed out;

2) one line can be seen, the mould is satisfactory;

3) no line can be seen, the mould is worn and shall be replaced.

4 PREPARATION 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, store the test sample after reduction in an oxygen-free nitrogen atmosphere or in freshly boiled water. In the latter case, make a paste of the coal with water and put the paste into a flask which is then filled to the top with freshly boiled water.

Care shall be taken to ensure that the test sample taken is truly representative.

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

Maximum particle size	Minimum mass of test sample
mm	g
5	1 000
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 the filter paper at a temperature not exceeding 40 °C until the coal appears to be dry, but in any case for not more than 2 h.

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 shall be controlled so as to produce the minimum of fines (see note). 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 shall be carried out without interruption.

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

through 0,2 mm	100 %
through 0,1 mm	85 to 70 %
through 0,06 mm	70 to 55 %

5 PROCEDURE

5.1 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. Repeat this 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).

Then adjust the length of the pencil 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.

5.2 Determination of dilatation

Heat the coal at a rate of 3 °C per minute.

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 holes 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_1 is less than 350 °C, charging of the furnace shall take place when the temperature is 20 °C below T_1.

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 min.

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

After 340 °C has been reached, the rise in temperature shall be very steady, minute by minute, and equal to the rate stated, with a tolerance of ± 3 % of the specified temperature rise in a 5 min period (see note).

During each 5 min period, the operator shall 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 °C. In the region of the critical points, sufficient points shall be plotted to determine the exact shape of the curve.

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

Carry out the duplicate determination in a separate run.

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

5.3 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 :

5.3.1 *Tube*

Crush the semi-coke and remove as much of it as possible with the auger. Then fill the tube with crude benzene 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.

5.3.2 *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.

6 EXPRESSION OF RESULTS

Calculate the observed changes in length as percentages of the initial length of the pencil.

7 PRECISION OF DETERMINATION

	Maximum acceptable difference between results	
	Same laboratory (Repeatability)	Different laboratories (Reproducibility)
Dilatation	$7\left(1+\frac{b}{100}\right)$ where b is the maximum dilatation per cent	(see 7.2)

7.1 Repeatability

The results of duplicate determinations, carried out at different times in the same laboratory by the same operator with the same apparatus on the same analysis sample, should not differ by more than the above value.

7.2 Reproducibility

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

8 TEST REPORT

The test report shall include the following particulars :

a) whether the sample submitted to the test fulfils the conditions specified in clause 5;

b) the rate of heating employed;

c) a curve of the percentage changes in length as a function of temperature on a standard relative scale in which 10 °C on the horizontal scale is equal to 5 % on the vertical scale;

d) the corrected temperatures T_I, T_{II} and T_{III} rounded off to the nearest 5 °C;

e) the percentage contraction (a) rounded to the nearest whole number;

f) the percentage dilatation (b) rounded :

to the nearest whole number for negative dilatation;

to the nearest 5 % for positive dilatation up to 100 %;

to the nearest 10 % for positive dilatation over 100 %.

ANNEX

INSPECTION OF DILATOMETER TUBES

After 100 determinations have been carried out in one tube, the next four determinations made shall be duplicated in a new tube. The results shall then be examined as follows :

If b_o is the dilatation with the old tube, and

b_n is the 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 shall 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 shall therefore be rejected.

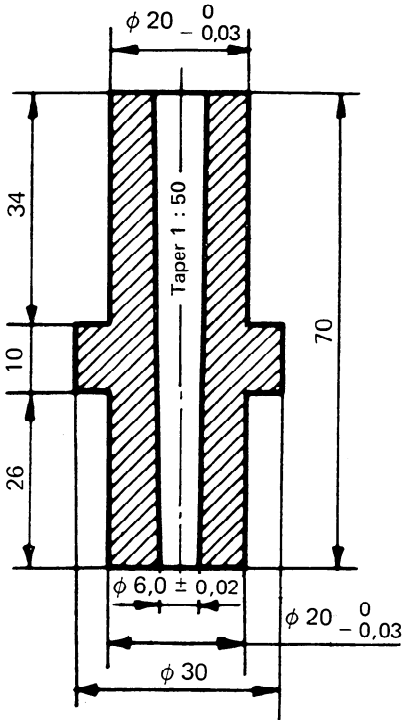
Example 2

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

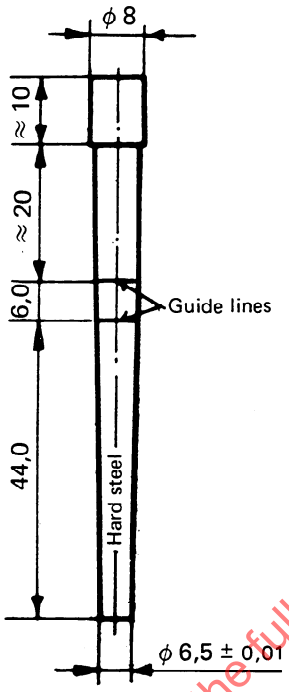
The average value of $x = -1,8$

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

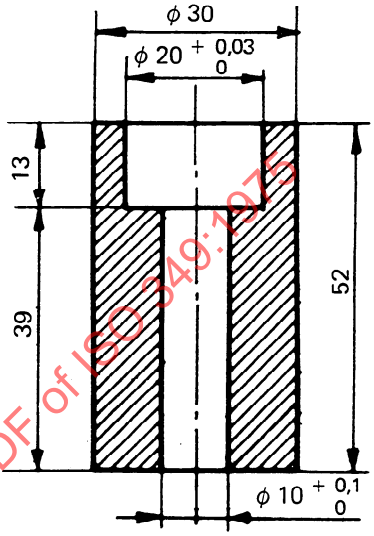
Dimensions in millimetres



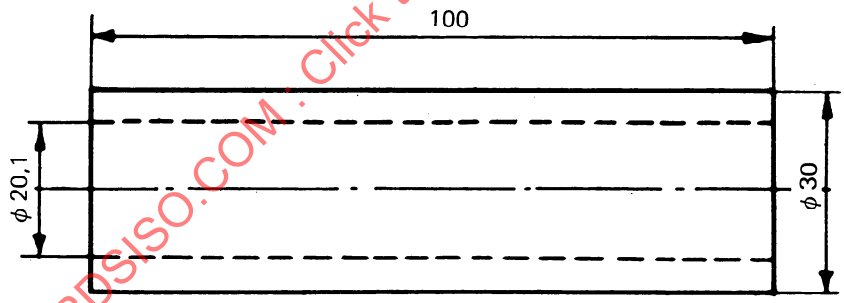
Mould



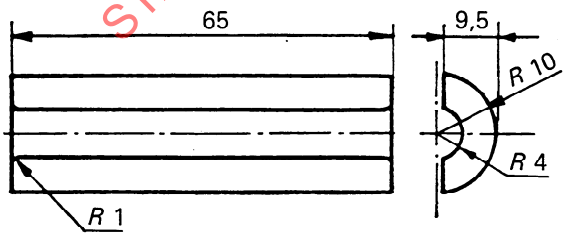
Gauge



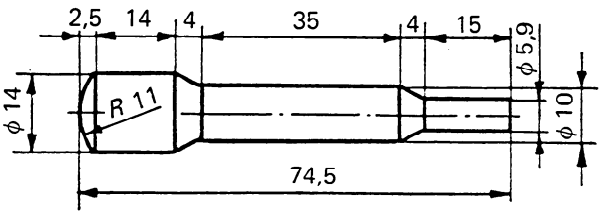
Ejector guide



Guide tube



Receptacle

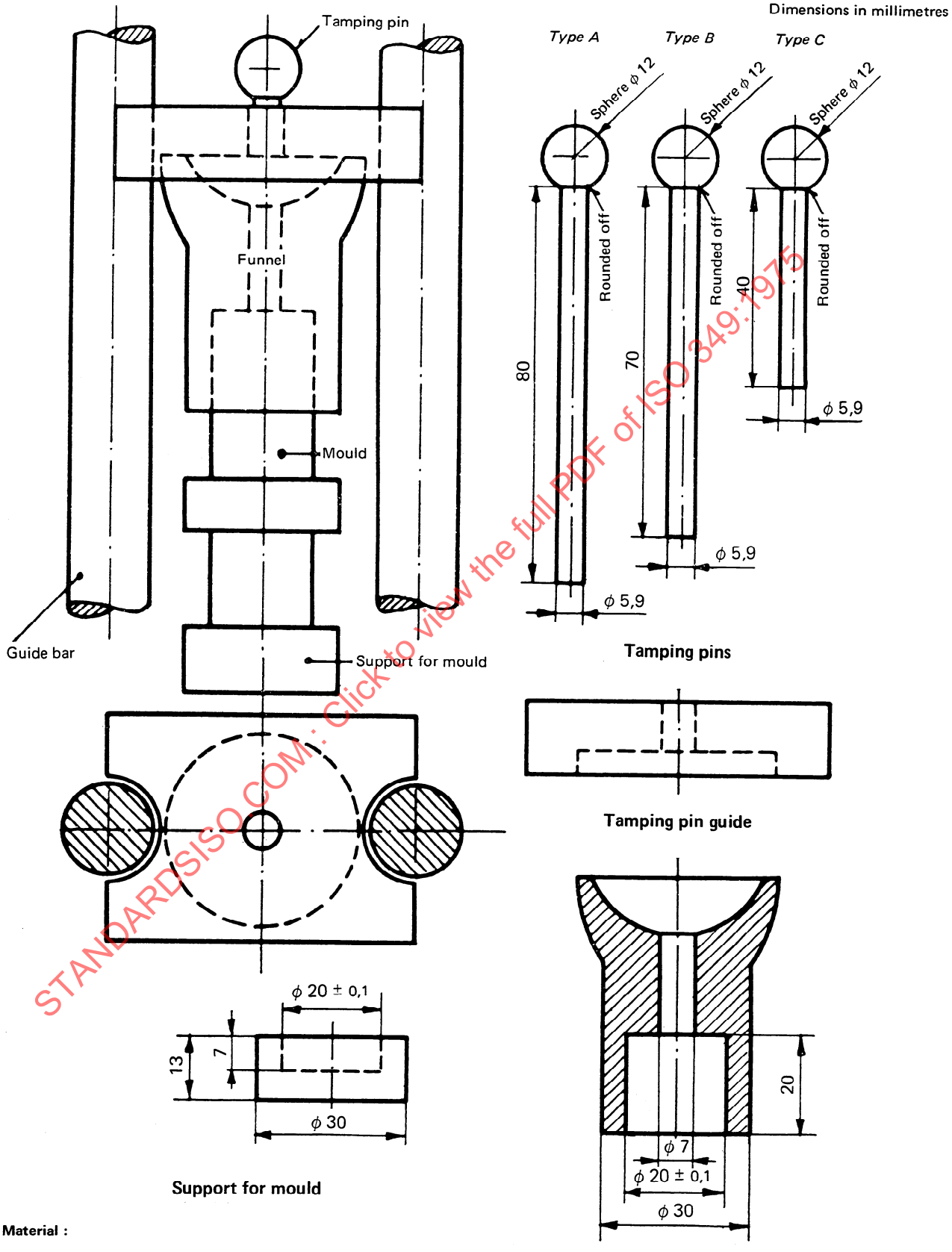


Ejector piston

Material :

Mould, ejector guide, guide tube, receptacle — hard bronze.
Gauge, ejector piston — vanadium steel.

FIGURE 2 — **Mould and accessories**



Material :

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

FIGURE 2 A — **Mould and accessories** (*concluded*)

Dimensions in millimetres

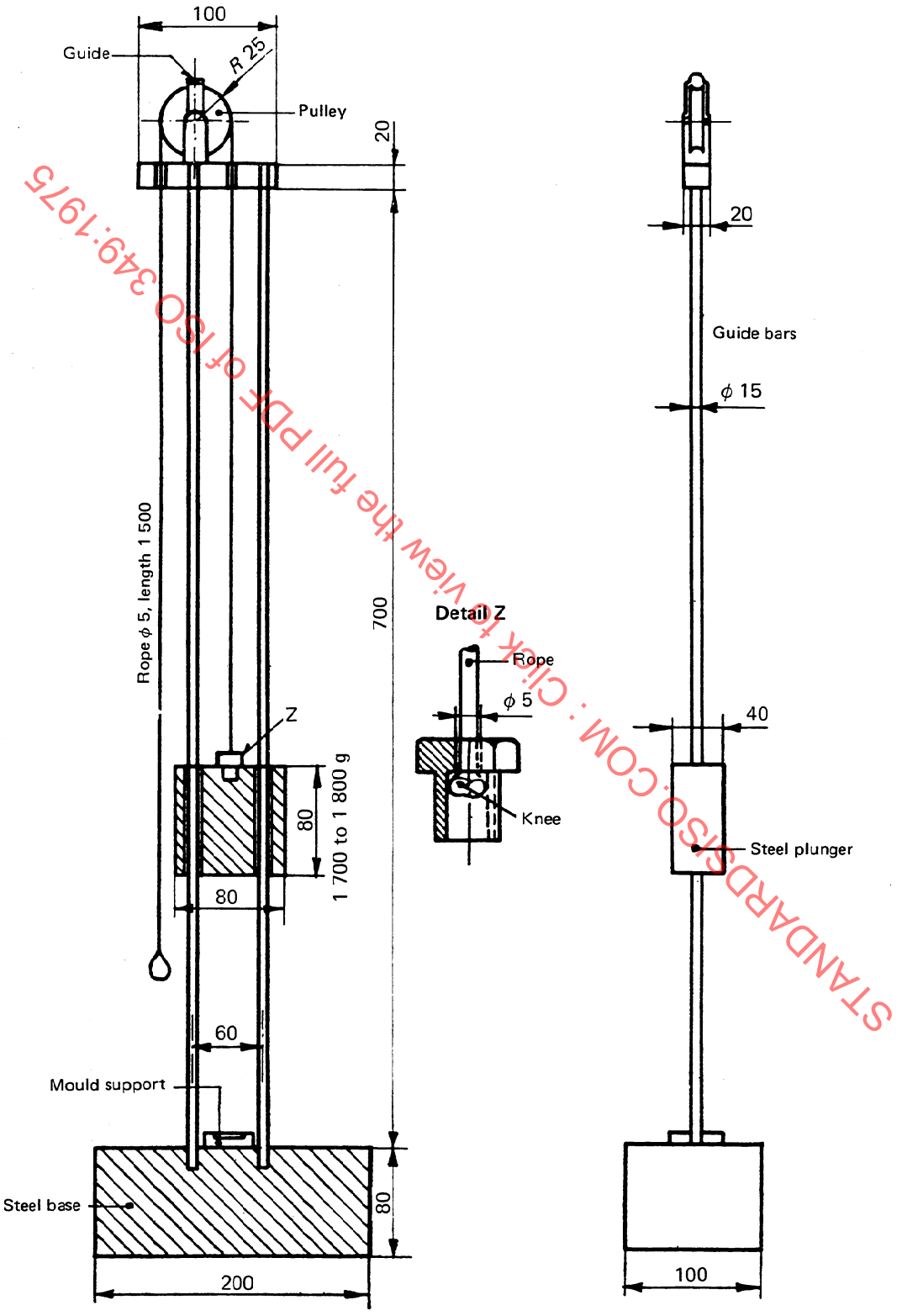
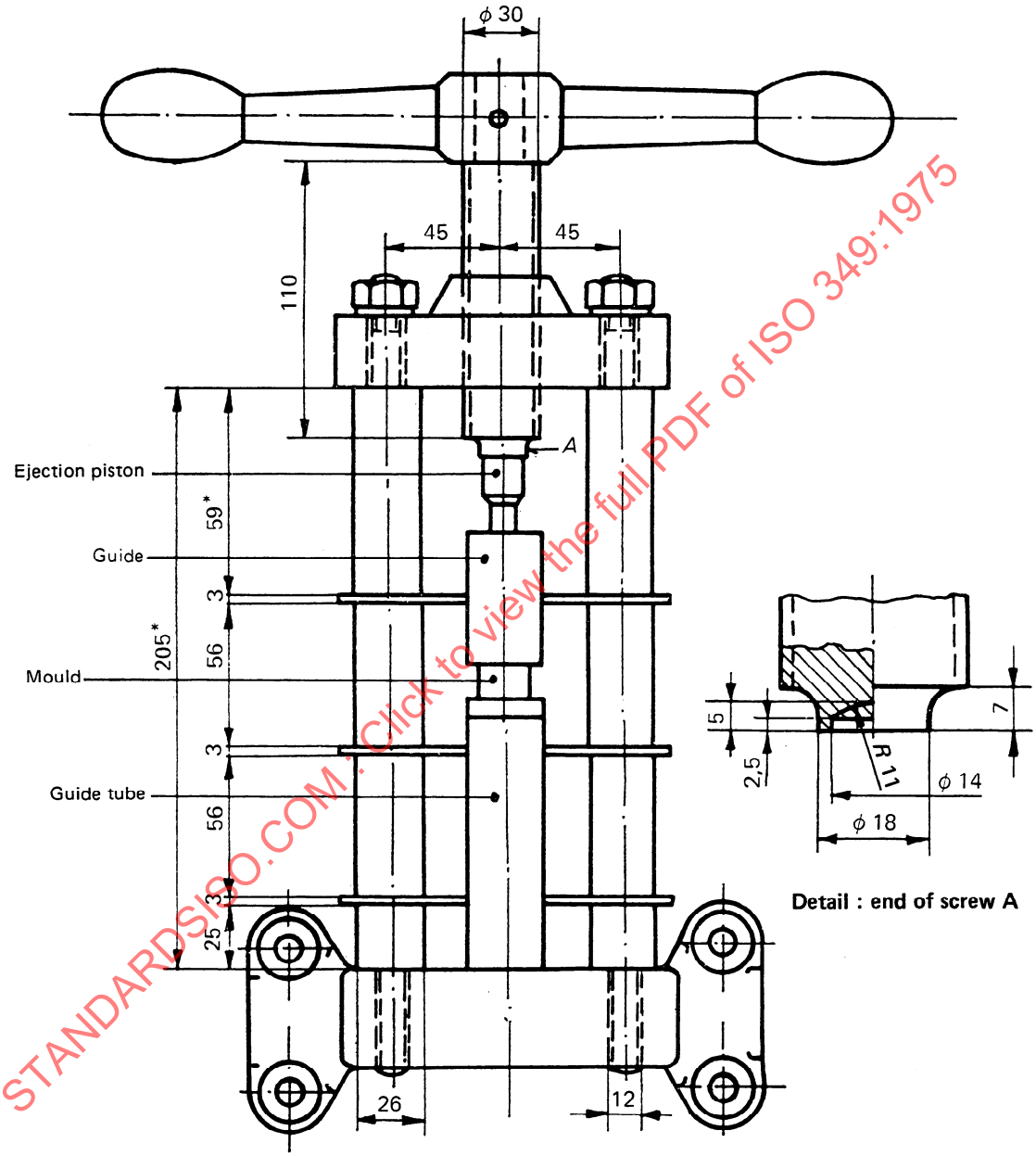


FIGURE 3 – Ram

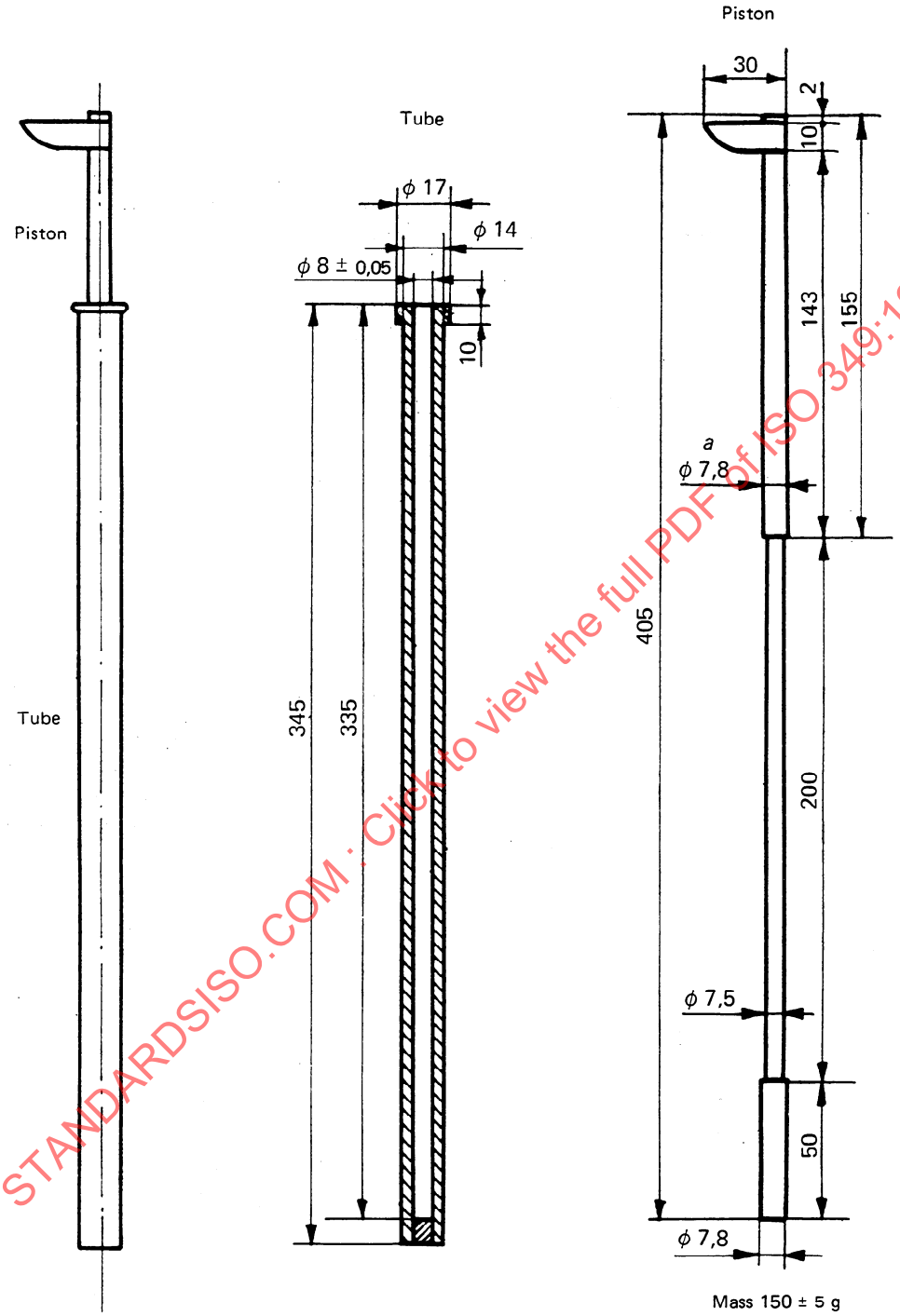
Dimensions in millimetres



* These dimensions may be increased to 230 and 84 mm respectively.

FIGURE 4 — **Press**

Dimensions in millimetres



Assembled dilatometer

Material : chrome-nickel steel.

FIGURE 5 — **Dilatometer tube and piston**