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FUEL RESEARCH INSTITUTE

OF SOUTH AFRICA

TEGNIESE MEMORANDUM
TECHNICAL

NO. 39 OF 1972

RUHR DILATOMETER TEST FOR COAL
LABORATORY PROCEDURE

OUTEUR: E.F.E. Müller
AUTHOR:

Cosmi 7043 — B 56

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INTRODUCTORY COMMENTS

The following Laboratory Procedure for the dilatometer test for coal has been translated from the German Laboratoriumvorschrift (abbr. L.V.) of the Steinkohlenbergbauverein Chemical Committee, reference number 2 Kokerei LV 20/32/01, edition May 1966.

Basically there is no difference between the Ruhr dilatometer test and the Audibert-Arnu dilatometer test, except in the preparation of the coal pencil. It is therefore considered essential to consult also the Iso Recommendation R349 (Ref. No. ISO/R 349-1963 (E)) for the Audibert-Arnu dilatometer test for coal when studying laboratory procedure applicable to the Ruhr dilatometer. (In LV 20/32/01 no instructions are given as to standardization, inspection, and cleaning of apparatus (except for inspection of mould). The instructions of ISO/R 349 can be applied.

In the evaluation of dilatometer results LV 20/32/01 of May 1966 has an interesting addition in that where only contraction occurs and the curve does not become horizontal but maintains a downward sloping trend, it is conventionally accepted that the contraction at 500°C is the value reported. For South African coals this is an important point as this type of curve is encountered fairly often. It has been difficult in the past to report such results.

For a general discussion on the dilatometer test see section 3.2 of F.R.I. Report No. 16 of 1971 "A Comparison of Coking Properties of South African Coals, as Determined in the Laboratory" by E.F.E. Müller. Reference is made in the report to a publication by W. Schreiber

/"Die .....

"Die Brauchbarkeit des Dilatometer-Testes" Glückauf, 100 (1964), pp. 1324-1334, in which he compares results obtained with different dilatometers. Schreiber emphasizes that more rigorous international standardization of the dilatometer test is necessary and recommends the adoption of the German laboratory procedure which approaches the ideal requirements for the coal pencil, with regard to mass, volume, apparent density, and degree of compaction. Only these dilatometer pencils give the coking ability correctly and reproducibly.

E.F.E. Müller
PRINCIPAL RESEARCH OFFICER

PRETORIA.
2nd August, 1972
EFEM/EMc

RUHR DILATOMETER TEST

LABORATORY PROCEDURE

Translated from L.V. 20/32/01 (Ed. May 1966) of the Steinkohlenbergbauverein Chemical Committee

1. GENERAL

1.1 Purpose

The Dilatometer test is used for determining the coking power of coking coals and coking coal components. It also serves for the classification of coals in the International Classification System[1].

1.2 Theory

The dilatation course is that curve which indicates the change in length of a compressed coal pencil with heat, applied at a constant and definite rate. It is determined under conventional and fixed conditions. The coking power of a coking coal and coking coal component in the dilatometer is characterized by contraction, dilatation, softening temperature, resolidification temperature, and plastic range.

Contraction is the reduction in length which the coal pencil undergoes during heating. Dilatation is the change in length of the coal pencil, after the contraction phase up to resolidification, with respect to the original length of the pencil. The temperature at which contraction commences is conventionally the softening temperature. The temperature at which the dilatation phase ends is designated the resolidification temperature (or temp. of maximum dilatation). In the event of no dilatation occurring then the temperature of maximum contraction is also the resolidification temperature. The softening and resolidification temperatures are the limits of the plastic range.

/1.3 Application .....

1.3 Application

The procedure is applicable to any coking coal and its components.

2. PROCEDURE

2.1 Theory

To determine the dilatation, use is made of a Dilatometer (Fig. 3). A coal pencil is heated under load of a piston at the rate of 3°C per minute. In the event of the coal softening the space around the pencil becomes filled. The resultant lowering of the piston characterizes the contraction. If the softened pencil, after contraction, increases its volume by swelling, the piston is raised the amount of which is termed the dilatation. As soon as the recorder shows a horizontal straight line the dilatation is complete.

2.2 Apparatus and Chemicals

Mould of steel, polished inside, with tamping pin (see Fig. 1).

Gauge with two guide lines for the mould (see Fig. 1).

The mould is in order, if the mark at the narrower end of the gauge is just visible. When either both marks or none are visible, the mould is unsuitable and should not be used.

Hammer with a mass of about 200 g.

Press and pressure gauge with indicator, range 0 to 100 kp/mm^2. (This range corresponds with commercial gauges with marking 0 to 3 t.) (see Fig. 2.)

/Gauges .....

Gauges for measuring length of pencils to lengths (60±0,5)mm, (45±0,5)mm, and (30±0,5) mm. These can be made of glass-tubing with internal diameter of 8 mm. (Note: The FRI uses a metal block with a bore 60 mm long.)

Dilatometer (see Fig. 3).

Temperature control unit with programmer coupled to NiCr-Ni thermocouples giving a heating rate of 3°C per minute with the dilatometer, with a range to 550°C.

Water with the addition of 0,5 g of wetting agent.

2.3 Method

Before using for the first time, after longer use, and after longer periods of disuse, the dilatometer and accessories should be inspected. (See ref. 2 and 3.)

The laboratory sample prepared according to L.V. 20/00, i.e. according to standard procedures, is dried at room temperature. The dried sample is crushed according to 2 DIN 4188 and then divided in a fine coal divider until two subsamples of 100 g each remain. One of these subsamples is ground so that its sieve analysis complies with the following size distribution:

Sieve (DIN 4188)	Percentage
through 0,2 mm	100
" 0,1 mm	70-85
" 0,063 mm	55-70

(Note: For sample prep. see also ISO/R 349[3])

/To .....

To about 10 g of this analytical sample 1 ml of water containing wetting agent is added and thoroughly mixed. A portion of this mixture is placed in the mould and compacted with hammer and tamping pin. Further portions are added and compacted; the number of portions should consist of at least five. After the last portion has been added and compacted, the complete mould is placed on the pressure gauge in the press (support swung out of the way) and the coal compressed by turning the spindle to an indicator reading of 70 kp/mm^2 which corresponds to a marking of 2 t on commercial instruments. The plug and mould base are removed and the mould hung on the support which is swung into position and the pencil pushed out by turning the spindle. The pencil is placed in the appropriate length gauge (normally 60 mm) so that the narrow end is in line with the end of the gauge. The protruding wider end is then cut off.

The Dilatometer is preheated to a temperature dependent on the type of coal. The particular temperature should be ca 30°C below the softening temperature of the test coal; this is in order that the dilatometer curve starts with a horizontal line. The following table serves as a guide to starting temperatures:

Coal type	°C
Long flame coal, gas coal	320
Coking coal, steam coal	340
Non-coking coal	400
Anthracite	450

(Note: These temperatures do not necessarily apply to S.A. coals.)

/As .....

As soon as the desired starting temperature has been reached a pencil of coal, narrow end up, is pushed into each of the two dilatometer tubes. The pistons are also pushed into the tubes and the tubes placed in the dilatometer furnace. If only one coal pencil is to be tested the second dilatometer tube with piston must nevertheless also be placed in the furnace so as not to change the heat capacity of the dilatometer. The third dilatometer tube in the furnace contains the thermocouple.

After the introduction of the dilatometer tubes the temperature will fall. It will then rise once more, regaining the starting temperature in about 5-7 minutes. During this period the recording pens and supports are fitted to the pistons and the pens adjusted to the corresponding zero points on the recording chart. As soon as the starting temperature has been regained the programmer and recorder are switched on, and if required the pens readjusted to the zeros. The recording of the dilatometer curve is thus initiated. The test is stopped when the curve, after dilatation, has remained horizontal for about five minutes, or 500°C if no dilatation has occurred.

In the event of a dilatation in excess of 300% the test must be carried out with a pencil of half the standard length. The 60 mm pencil prepared as described is shortened at both ends, 15 mm each end, with the use of the 45 mm and 30 mm length guides. The remaining middle piece is then used to determine the dilatation. In this case the recorded contractions and dilatations are to be doubled.

/3. EVALUATION .....

3. EVALUATION

The dilatometer curve is evaluated depending on its course. See Figs. 4 and 5.

The distance of the lowest point on the dilatometer curve from the zero line is the contraction.

If the dilatometer curve rises after contraction (see Fig. 4) then the distance between the horizontal line and the zero line is termed the dilatation; this is positive when the horizontal line is above the zero line (see example 1) and negative when the horizontal line is below the zero line (example 2). The dilatation is zero when the horizontal line after contraction returns to the zero line.

If the dilatometer curve does not rise after contraction (see Fig. 5, example 1), it is termed "contraction only". If the final course of the curve is not horizontal, but sloping downwards (see example 2) it is conventionally taken that the contraction is the distance of the curve from the zero line at 500°C.

If no contraction occurs, i.e. the dilatometer curve lies on the zero line the term "no softening" is applied.

Contraction a and dilatation b, expressed as percentage displacements of the original length of pencil, are read off the dilatometer recordings and reported in rounded off figures.

In duplicate determinations of the same sample the difference in the figures for contraction shall not exceed 5. The difference in the figures for dilatation shall not exceed the following maximum values:

/Dilatation .....

	Dilatation	Difference
≤	50%	7
	50% to 100%	10
	100% to 200%	14
>	200%	20

If the results comply, the average figures for contraction and the average figures for dilatation, rounded off to the nearest 1%, are reported, otherwise the tests are rejected.

The softening temperature t_1, the temperature at the minimum of the curve t_2, and the resolidification temperature t_3, insofar as they occur, are read off the recording and reported rounded off to the nearest $3^{\circ}C$.

In duplicate determinations of the same sample the corresponding temperatures shall not differ by more than $12^{\circ}C$. If this is the case the averages of the corresponding temperatures, rounded off to the nearest $1^{\circ}C$, are reported, otherwise the tests are rejected.

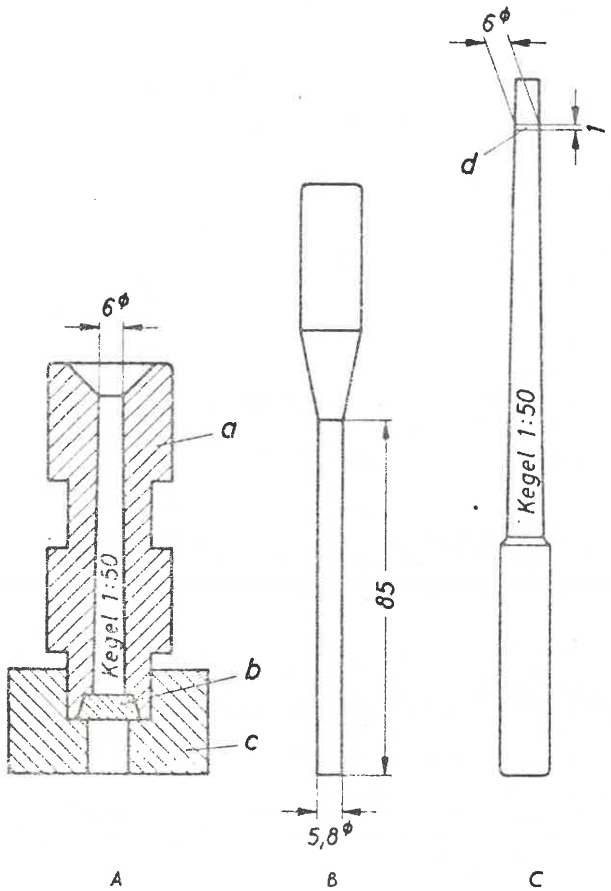
REFERENCES

1) DIN 23003: Internationales Klassifikations-System für Steinkohlen.

2) Lange, W., W. Radmacher, und H. Vierneisel: Brennst.-Chem. 42 (1961) pp. 312/19, 385/94; 43 (1962) pp. 39/45.

3) Iso Recommendation R349, Ref. No. ISO/R 349-1963 (E) Audibert-Arnu Dilatometer Test for Coal.

Note: Ref. 3 is not included in the original German version.



A Mould
a Cylinder with tapered bore
b Plug
c Base

B Tamping pin

C Gauge
d Guide lines

FIG. 1 MOULD WITH ACCESSORIES

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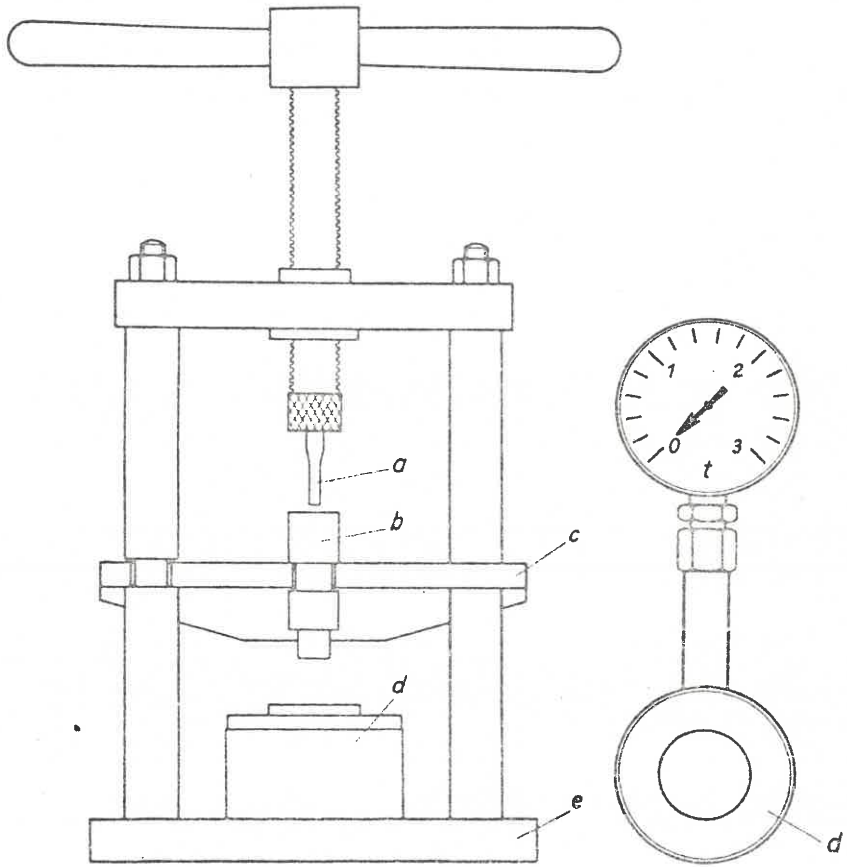


a Press tamping pin, rotary

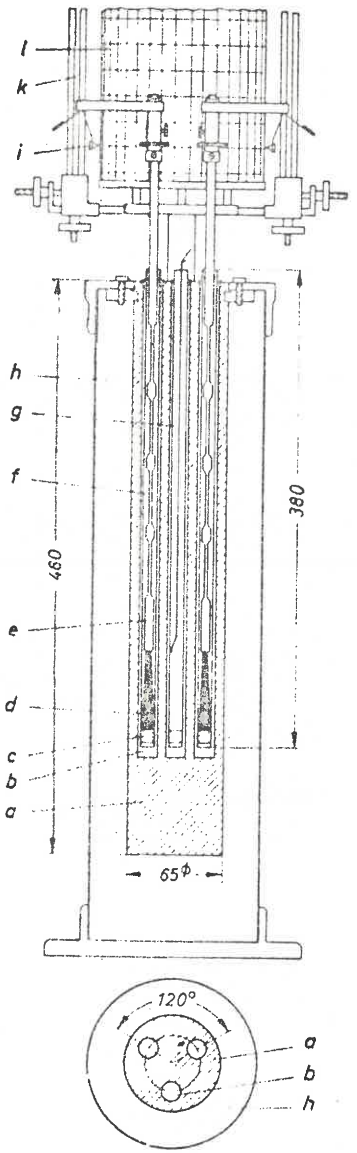
b Cylinder of mould

c Support for mould

d Pressure box with gauge

e Baseplate

FIG. 2 PRESS AND PRESSURE BOX

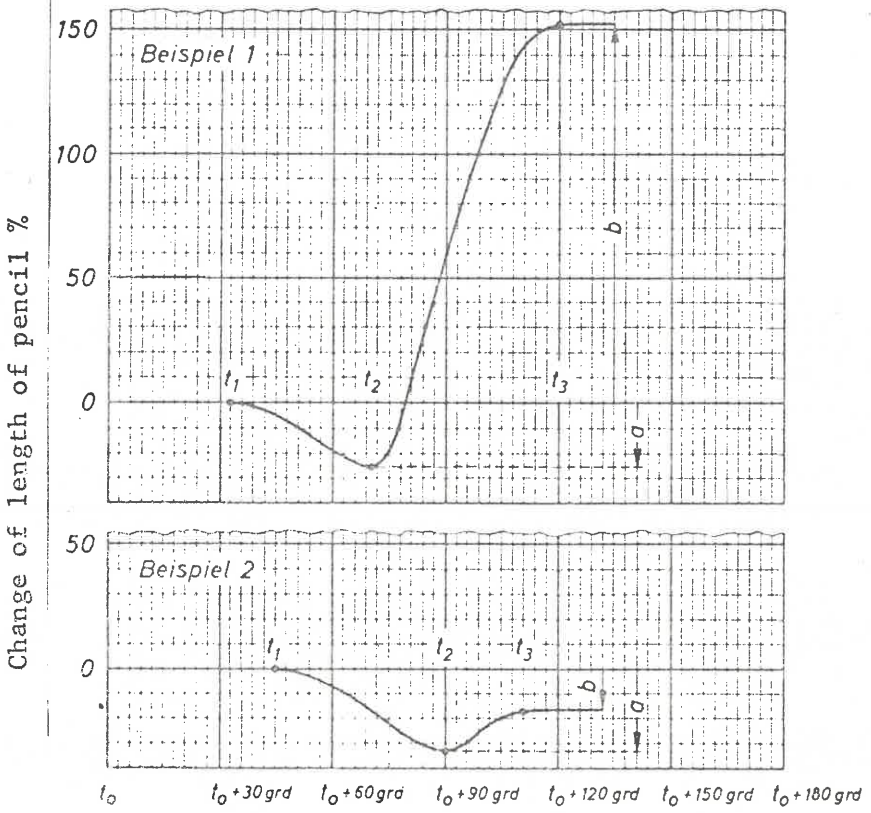


a Messingblock, elektrisch beheizbar
b Bohrung für Dilatometerrohr
c Verschlußschraube des Dilatometerrohres
d Kohlepreßling
e Belastungsstempel aus Stahl
f Dilatometerrohr aus Stahl
g Thermoelement, Nickel-Nickelchrom
h Isoliermasse
i Schreibvorrichtung
k Führung für Schreibvorrichtung
l Registriertrommel mit Registrierblatt

Abb. 3 Dilatometer

a Bronze block, electrically heated
b Bore for dilatometer tube
c Screw plug for dilatometer tube
d Coal pencil
e Piston, steel
f Dilatometer tube, steel
g Thermocouple, NiCv-Ni
h Insulation
i Pen assembly
k Pen guide
l Recording drum and chart

FIG. 3 DILATOMETER



		Example 1	Example 2
a	Contraction %	25	33
b	Dilatation %	152	-16
t_o	Initial temp. °C	350	320
t_1	Softening temp. °C	383	365
t_2	Temp. at min. of curve °C	420	410
t_3	Resolidification temp. °C	470	431

FIG. 4 EXAMPLES OF DILATOMETER CURVES

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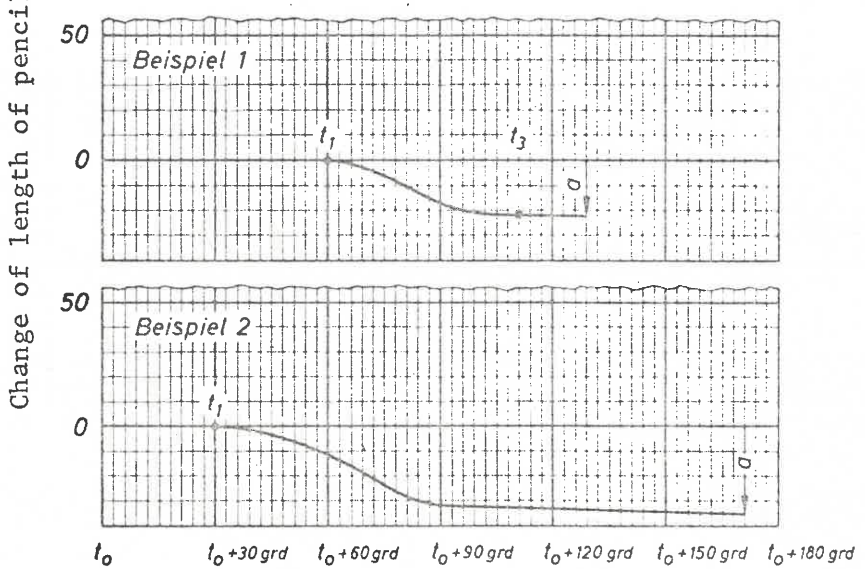

		Example 1	Example 2
a	Contraction %	22	35 at 500°C
b	Dilatation	none	none
t_o	Initial temp. °C	370	330
t_1	Softening temp. °C	430	360
t_2	Resolidification temp. °C	481	-

FIG. 5 EXAMPLES OF DILATOMETER CURVES