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Method of specifying the technical quality of coke for use in blast furnaces

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Committees responsible for this British Standard

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British Steel Industry
Coke Oven Managers' Association
National Coal Board

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Contents

Foreword

This revision of this British Standard has been prepared under the direction of the Solid Mineral Fuels Standards Committee. It supersedes the 1968 edition which is withdrawn.

The previous edition described a scheme which, if applied at producers' and consumers' works, enabled comparisons to be made by means of a "points value", based upon approved systems of sampling analysis and testing of coke.

The use of such a "points value" concept is no longer current in the industry and this revision provides a reference standard for the technical assessment of coke when it is subjected to internal transfer and also in international trade.

Attention is drawn to the provisions of the Health and Safety at Work etc. Act 1974 and the need to ensure that appropriate precautions are taken to ensure the safety of personnel carrying out the methods of test referred to in this standard.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This British Standard describes a method of specifying the technical quality of coke by listing the characteristics which are most widely used in the technical assessment of blast furnace coke.

In the case of each characteristic specified, reference is made wherever possible to an authoritative source giving detailed procedures for the experimental techniques and evaluation of the appropriate characteristic.

Information is given on typical values for each characteristic and the precision of the test methods.

It also provides guidance which may assist in the selection or choice of those properties which will be of most use in the assessment of coke for particular purposes (see Appendix A).

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Sampling and sample preparation

Sampling and sample preparation of coke for all purposes shall be carried out in accordance with the principles and particular requirements of BS 1017-2. In some instances it is necessary to prepare coke having a restricted range of particle size for a particular test. In such cases techniques of particle size reduction shall be such as to give the required yield of the specified size range.

3 Characteristics

The technical quality of a consignment or lot of coke shall be specified by quoting the values obtained from a sample (see clause **2**) by the method stated for the following characteristics:

a) total moisture content when tested in accordance with BS 1016-2;

b) ash and volatile matter content when tested in accordance with BS 1016-4;

c) sulphur content when tested in accordance with BS 1016-7;

d) phosphorus content when tested in accordance with BS 1016-9;

e) size of the coke when determined in accordance with BS 1016-18.

In addition the values for one or more of the following characteristics shall be quoted:

1) mechanical strength when assessed (see Table 1) either:

i) in accordance with BS 1016-13 (Micum test[1)] and Irsid drum test); or,

ii) in accordance with JIS K 2151:1977[2)] (JIS drum test); or,

iii) in accordance with ASTM 3402-8[3)] (ASTM tumbler test);

2) shatter indices of coke of a specified size when assessed in accordance with BS 1016-13;

3) bulk density, true relative density and apparent relative density when determined in accordance with BS 1016-13;

4) reactivity when assessed in accordance with the method described in Appendix C.

[1)] Appendix B gives details of the extended Micum test.

[2)] Obtainable from Japanese Industrial Standards Committee, c/o Standards Department, Agency of Industrial Science and Technology, Ministry of International Trade and Industry 1-3-1 Kasumigaseki, Chiyodaku, Tokyo 100, Japan.

[3)] Obtainable from ASTM, 1916 Race Street, Philadelphia, Pa 19103, USA.

Table 1 — Details of drum tests

		Half-micum drum test	JIS drum test	ASTM tumbler test
Drum dimensions				
Internal diameter	mm	1 000	1 500	914
Internal length	mm	500	1 500	457
Thickness of metal	mm	10	9	6
Lifting flights				
Number		4	6	2
Depth of face	mm	100	150	51
Rotating speed	r/min	25 ± 1	15 ± 0.5	24 ± 1
Test sample				
Mass	kg	25	10	10
Lump size	mm	> 20	⩾ 50 ⩽ 75	⩾ 50 ⩽ 75
Test sieve type		round hole	square hole	square hole
Moisture	%	< 3.0	air dried	⩽ 1.0
		Micum test		
Number of revolutions		100	30	1 400
Strength indices		M_{40} = % > 40 mm	DI^{30}_{15} = % > 15 mm	Stability factor T_{25} = % > 25 mm
		M_{10} = % < 10 mm		Hardness factor $T_{6.3}$ = % > 6.3 mm
		Irsid test		
Number of revolutions		500	150	
Strength indices		I_{20} = % > 20 mm I_{10} = % < 10 mm	DI^{150}_{15} = % > 15 mm	

4 Typical values and precision of the methods

An indication is given in Table 2 of the order of values to be expected in the case of each characteristic noted.

Further information regarding the reporting of results, change of base, rounding and precision may be obtained by reference to BS 1016-16.

Table 2 — Typical values of characteristics of coke and precision of the test methods

Property		Typical values	Rounding	Precision	
				Repeatability	Reproducibility
Total moisture	%	3 to 10	0.1	0.5 absolute	0.7 absolute
Ash: dry basis	%	6 to 12	0.1	< 10 : 0.15 absolute ⩾ 10 : 0.20 absolute	0.3 absolute 0.4 absolute
Volatile matter: d.a.f. basis	%	< 1.0	0.1	0.20 absolute	—
Sulphur: dry basis	%	0.5 to 1.5	0.01	0.05 absolute	0.10 absolute
Phosphorus: dry basis	%	< 0.02	0.01	< 0.02 : 0.002 absolute ⩾ 0.02 : 10 of the amount	0.005 absolute 25 of the amount
Size: upper limit	mm	100	—	—	—
lower limit	mm	20	—	—	—
Arithmetic mean size	mm	50 to 60	0.1	—	—
Micum indices					
60 mm sample	M_{40}	> 75	0.1	5 units	—
	M_{10}	< 10	0.1	1.5 units	—
Extended micum slope		1	0.01	—	—
Irsid indices	I_{20}	> 75	0.1	7 units	—
	I_{10}	< 20	0.1	2 units	—
JIS indices	DI^{30}_{15}	80 to 98	0.1	< 90 : 4.0 units ⩾ 90 : 1.5 units	— —
	DI^{150}_{15}	70 to 90	0.1	< 80 : 2.5 units ⩽ 80 : 1.5 units	— —
ASTM indices					
Stability factor	T_{25}	> 50	0.1	2.0 units	—
Hardness factor	$T_{6.3}$	> 60	0.1	2.0 units	—
Shatter index	S_{40}	> 80	0.1	6 units for isolated consignments 5 units for regular consignments	— —

NOTE The Micum, Irsid, JIS, ASTM and shatter indices are dependent, to different extents, on the size distribution of the bulk sample. As it is not possible to circulate a bulk sample to a number of laboratories without risk of breakage, and, therefore, alteration of the size distribution, no reproducibility tolerances have been given for these tests.

Appendix A Guidance on the selection of tests for coke quality for blast furnaces

The trend towards coke as opposed to coking coal becoming an internationally marketable product is growing and with it the requirement to meet critical quality specifications.

Sampling of coke for proximate and ultimate analyses presents no major difficulties; however, sampling and the selection of tests for assessing the physical quality of coke does present difficulties particularly where the coke has been or is to be subjected to considerable mechanical handling during transport and stocking. As a general rule the more rigorous the test, the lower the extent to which the test result is influenced. Micum test indices are extremely sensitive to the amount of handling to which the coke has been subjected, so much so that M_{40} indices may increase by 6 to 8 points between the coke ovens and the blast furnace. The ASTM test being the most rigorous is barely influenced whilst the extended Micum slope is independent of any pretreatment.

Physical quality specifications based upon drum tests should always specify in addition to the value of the index of quality the point in the handling system where the sample is taken, e.g. point of loading or receipt.

Metallurgical coke for use in the modern blast furnace also requires a narrow size range, e.g. 30 mm to 70 mm, consequently tests such as a Micum test based upon size greater than 60 mm round hole coke and to a lesser extent the ASTM test based upon size greater than 50 mm square hole coke are becoming less popular for assessing the quality of furnace coke since they are testing a less than representative sample of the coke. Where used, these tests are utilized for assessing coke oven operations rather than coke quality for ironmaking. For this reason the basic Micum test and the Irsid test use a sample of size greater than 20 mm. Other sizes of coke may be used for the Micum test when required but it is essential that the size used for the test should be reported with the result.

In assessing the quality of coke for the blast furnace there is a growing tendency to move towards more rigorous tests based upon the generation of the fines fraction as an indicator of quality. These parameters are more indicative of the resistance to abrasion rather than the physical strength of the coke which has tended to be associated with the larger size indices and the coarse breakage.

Many equations have been published relating the various drum indices (see Table 3). Where a specific drum index is quoted this should wherever possible be measured and not calculated from another index. Extreme caution is advised in using these equations and their use should be restricted to the abrasion indices and to coke made from conventional wet charges.

In addition to assessing the quality of coke at ambient conditions there is a tendency to include high temperature test properties such as reactivity to carbon dioxide and the assessment of physical quality following reaction with carbon dioxide.

Table 3 — Relations between drum indices

Reporter	
Joh et al[a]	**Miyazu et al**[b]
$DI_{15}^{30} = (17.14M_{40})^{0.39}$	$M_{40} = -96.08 + 1.85DI_{15}^{30}$
$T_{25} = -2.62M_{10} + 73.9$	$M_{40} = 55.18 + 0.24DI_{15}^{150}$
$DI_{15}^{30} = -1.54M_{10} + 10.3$	$M_{40} = 57.73 + 0.31\ T_{25}$
$T_{25} = M_{40} - 20.1$	$M_{10} = 62.97 - 0.58DI_{15}^{30}$
$T_6 = 0.28M_{10} + 68.6$	$M_{10} = 14.56 - 0.07DI_{15}^{150}$
$DI_{15}^{30} = (42.0T_{25})^{0.198}$	$T_{25} = 53.78 + 0.13DI_{15}^{150}$

[a] Joh, H and G da, S. Yawata Technical Report. 1970 (233) 116 – 124.
[b] Miyazu T *et al* — Nippon Kokan Technical Report (6 overseas) — Dec 1970, pp 1 – 13.

Appendix B Extended Micum test

B.1 General

A Micum test, known as the extended Micum test, can be carried out by repeating the test procedure and determining the size analysis of the sample after each test cycle, e.g. 100, 200, 300, 500, 700, 900 drum revolutions.

B.2 Apparatus

As described in **4.3.2** of BS 1016-13:1980.

B.3 Test sample

As described in **4.3.3** of BS 1016-13:1980.

B.4 Procedure

Carry out this variant of the Micum test by using the procedure described in **4.3.4.2** of BS 1016-13:1980. After the coke sample is sized following 100 drum revolutions, carefully return the whole mass of the sample to the drum and rotate for a further test cycle and again remove and resize. Repeat this procedure until the predetermined test cycles are completed.

NOTE It is impossible to carry out an extended Micum test without losing some of the sample which is reduced to a particle size of less than 1 min. It is also impossible to reject the test if the loss at any stage exceeds the limit laid down in **4.3.4.3** of BS 1016-13:1980. Great care therefore is necessary to ensure that loss in mass at each stage of the extended test is kept to a minimum. All losses are added to the mass of the smallest fraction.

B.5 Calculation of results

Convert the cumulative mass of each fraction, in kilograms and weighed to the nearest 25 g, in each test cycle to a percentage by multiplying by four, and tabulate the results cumulatively. Calculate the arithmetic mean size of the sample, after each test cycle using the formula given in **4.2.5** of BS 1016-13:1980. Inspect the data by plotting the reciprocal of the square of the arithmetic mean size (multiplied by 10^4 for convenience) against the number of drum revolutions.

An example of the results of a typical extended Micum test, using a sample containing all sizes greater than 20 mm, is given in Table 4 and shown graphically in Figure 1.

Table 4 — Typical extended Micum test data

Drum revolutions	0	100	200	300	500	700	900
Size analysis: % cumulative							
> 100 mm	1.4	—	—	—	—	—	—
> 80 mm	14.0	6.4	5.4	3.7	2.6	2.0	1.1
> 60 mm	43.6	21.1	20.2	17.8	13.0	8.8	6.8
> 40 mm	84.1	66.6	60.0	55.0	47.5	42.1	37.8
> 30 mm	94.8	83.0	78.3	74.6	67.6	61.6	57.5
> 20 mm	100.0	90.9	87.4	84.1	78.6	73.7	69.2
> 10 mm	100.0	93.6	90.0	87.4	82.1	77.8	74.1
< 10 mm	0.0	6.4	10.0	12.6	17.9	22.2	25.9
Arithmetic mean size S_m (mm)	58.90	47.84	44.69	42.16	38.08	34.78	32.33
$\frac{10^4}{S_m^2}$	2.882	4.369	5.007	5.625	6.896	8.266	9.567

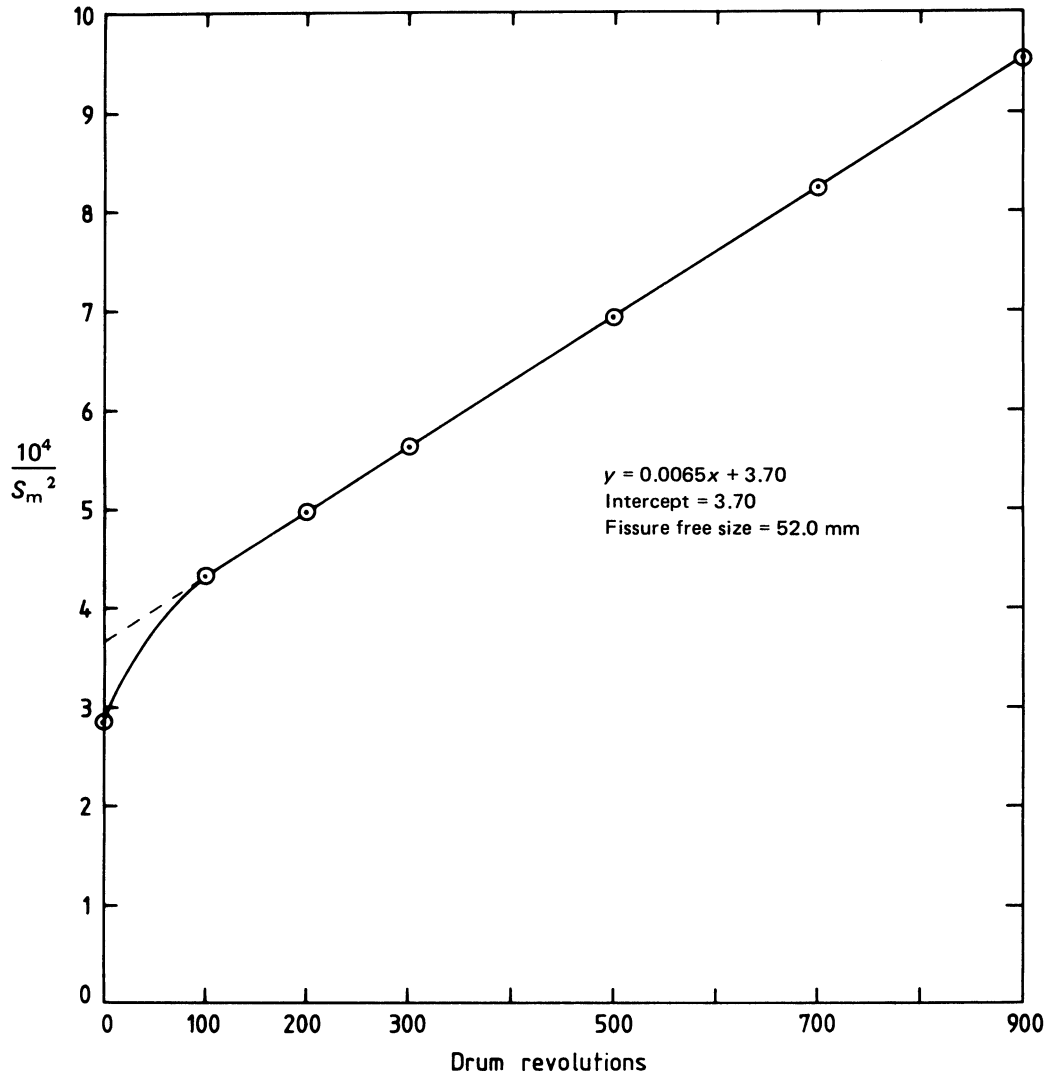


Figure 1 — Relationship between $10^4/S_m{}^2$ and drum revolutions

From Table 4:

a) Micum indices > 20 mm M_{40} = 66.6

> 20 mm M_{10} = 6.4

If required the M_{40} of a sample of greater than 60 mm coke can be estimated, with an accuracy of ± 5.0 units, by taking account of the proportion of the greater than 20 mm sample which is greater than 40 mm at zero revolutions of the drum.

Hence:

$$> 60 \text{ mm } M_{40} = \frac{> 20 \text{ mm } M_{40}}{\% > 40 \text{ mm in } > 20 \text{ mm sample}} \times 100$$

$$= \frac{66.6}{84.1} \times 100 = 79.2 \pm 5.0$$

b) Irsid indices I_{20} = 78.6

I_{10} = 17.9

From Figure 1 the equation for the best straight line relationship between the reciprocal of the square of the arithmetic mean size and the number of drum revolutions is derived by graphical or mathematical means.

Hence:

$y = 0.0065x + 3.70$

then:

$$\text{Micum slope} = \frac{0.65 \times 10^{-6}}{(\text{mm}^2)\ \text{rev}}$$

intercept = 3.70

$$\text{fissure free size} = \left(\frac{10^4}{3.70}\right)^{0.5} = 52.0\ \text{mm}$$

From Table 4 and Figure 1:

$$\text{fissure index} = \frac{\text{arithmetic mean size of sample at zero revolutions}}{\text{fissure free size}}$$

$$= \frac{58.9}{52.0} = 1.13$$

The relationship between the extended Micum slope (M_s) and M_{30} after 900 revolutions (M_{30}^{900}) for 36 blast furnace cokes with extended Micum slopes in the range 0.43 to 0.93 is given by the equation:

$M_s = 1.85 - 0.02\ M_{30}^{900}$ (95 % confidence limits ± 0.10)

If this equation is used, the long and tedious procedure for carrying out the extended Micum test may be avoided.

Appendix C Reactivity test

C.1 Principle

A mass of coke of a specified size is heated in an atmosphere of carbon dioxide and the loss in mass determined. The residual material is then subjected to a drum strength test and the mass of particles greater than 10 mm expressed as a percentage.

C.2 Apparatus

NOTE 1 It is necessary to calibrate the thermocouples and flow meter every 3 months.
NOTE 2 A typical arrangement of reactivity apparatus is shown in Figure 2.

C.2.1 *Analytical balance,* with an accuracy of 0.1 g.

C.2.2 *Electric furnace,* of soaking zone depth at least 170 mm and capable of being controlled at 1 100 ± 5 °C.

NOTE A suitable type is a two part electric furnace with a three point control.

C.2.3 *Jaw crusher*

C.2.4 *Sieves,* of 21 mm nominal aperture and 3 mm max. bridge width and of 10 mm and 19 mm nominal aperture, square hole complying with BS 410.

C.2.5 *Oven,* capable of being controlled at 150 ± 2 °C.

C.2.6 *Reaction tube,* 78 mm internal diameter manufactured from type 310 Cr Ni stainless steel.

C.2.7 *Thermocouple,* for indicating test sample temperature.

NOTE Pt/Rh type is suitable.

C.2.8 *Gas flow meter,* calibrated at 20 °C giving flow rates of 5 ± 0.1 L/min and 10 ± 0.1 L/min.

C.2.9 *Gas preheater,* made of stainless steel with a perforated lid and filled with porcelain balls.

C.2.10 *External control thermocouple,* manually adjustable.

C.2.11 *Strength drum with no protrusions,* of 130 ± 2 mm internal diameter and 700 ± 2 mm long, capable of rotating on its transverse axis at 20 ± 0.5 r/min.

C.3 Reagents

C.3.1 *Nitrogen*

C.3.2 *Carbon dioxide*

C.4 Test sample preparation

Crush 10 kg of the sample of 25 mm minimum grain size until there is no over-size above 21 mm and a yield of at least 10 % of 19 mm to 21 mm size.

Heat the thoroughly mixed, 19 mm to 21 mm size sample for 2 h at 150 ± 2 °C and cool in a desiccator.

C.5 Procedure

C.5.1 Heat the furnace to 1 100 ± 5 ° C.

C.5.2 Accurately weigh approximately 200 g of the test sample. Record the value.

C.5.3 Pour the test sample around a rod inserted in the centre of the reaction tube; place the lid on the tube and shake twice to give some compaction to the test sample.

C.5.4 Adjust the flow rate of nitrogen to 10 ± 0.1 L/min and connect to the gas preheater.

C.5.5 Place the reaction tube in the gas preheater and then place the gas preheater in the electric furnace.

C.5.6 Allow the test sample to heat to 1 100 ± 5 °C with a flow of nitrogen for 10 min.

C.5.7 Change the gas flow to carbon dioxide at 5 ± 0.1 L/min and continue heating for 120 min.

C.5.8 Change the gas flow to nitrogen at 10 ± 0.1 L/min; remove the gas preheater from the furnace and allow to cool to room temperature.

C.5.9 Accurately weigh the residual test sample. Record the value.

C.5.10 Transfer the residual test sample to the strength drum and rotate at 20 ± 0.1 r/min for 30 min.

C.5.11 Sieve the residual test sample from the drum through a 10 mm nominal aperture square hole sieve and accurately weigh the material retained by the sieve. Record the value.

C.6 Calculation

Calculate the following parameters:

$$\text{reactivity} = \frac{\text{loss of mass during reaction}}{\text{mass of test sample}} \times 100$$

$$= \frac{A - B}{A} \times 100$$

$$\text{strength after reaction} = \frac{\text{mass of test sample} > 10 \text{ mm after strength test}}{\text{mass of test sample after reactivity test}} \times 100$$

$$= \frac{C}{B} \times 100$$

where

A is the mass of the test sample in **C.5.2**;

B is the mass of the residual test sample in **C.5.9**;

C is the mass of the material retained by the sieve in **C.5.11**.

C.7 Report

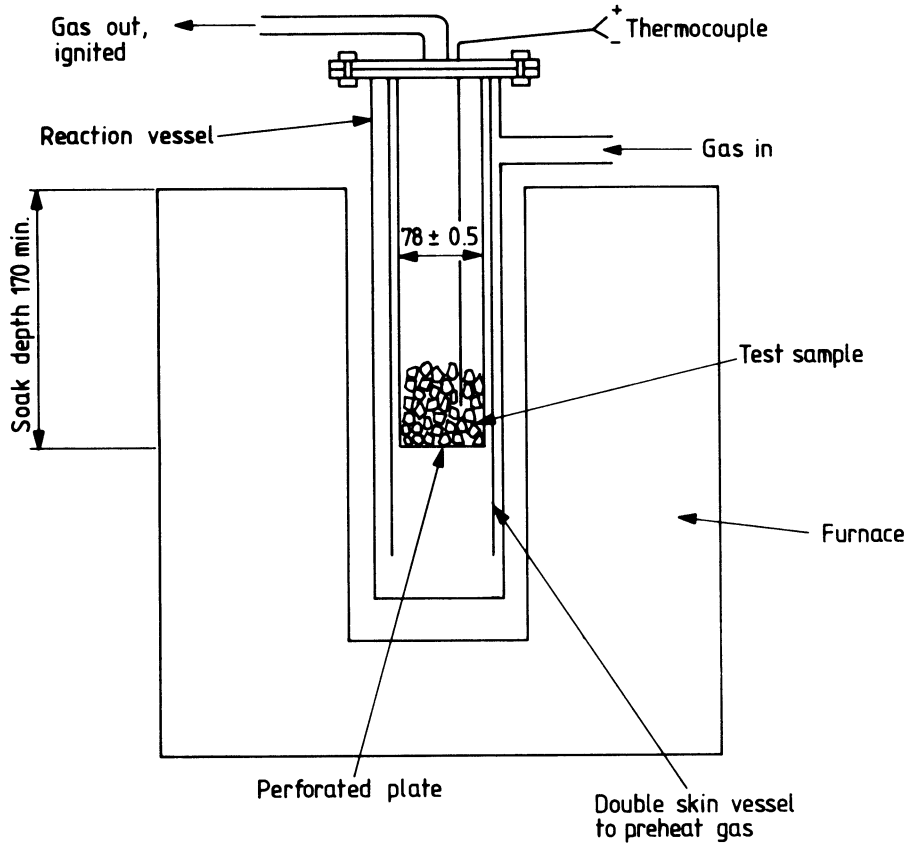
The following shall be included in the test report:

a) the identification of the sample;

b) the reactivity percentage;

c) the strength after reaction percentage;

d) the date of the test.



All dimensions are in millimetres.

Figure 2 — Typical arrangement of reactivity apparatus

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Publications referred to

BS 1016, *Methods for the analysis and testing of coal and coke.*
BS 1016-2, *Total moisture of coke.*
BS 1016-4, *Moisture, volatile matter and ash in the analysis sample of coke.*
BS 1016-7, *Ultimate analysis of coke.*
BS 1016-9, *Phosphorus in coal and coke.*
BS 1016-13, *Tests special to coke.*
BS 1016-16, *Methods for reporting results.*
BS 1016-18, *Size analysis of coke.*
BS 1017, *Methods for sampling of coal and coke.*
BS 1017-2, *Sampling of coke.*

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