

Calibration of rubber and plastics test equipment

Part 3. Calibration schedules

Section 3.1 Thermal properties

ICS 83.060; 83.080

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Committees responsible for this British Standard

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- British Rubber Manufacturers' Association Ltd.
- Department of Trade and Industry (National Physical Laboratory)
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Foreword

This Section of BS 7825 has been prepared by Technical Committee PRI/20.

In commercial practice it is necessary for the parties concerned in the manufacture or supply of materials or products to operate or agree upon specified performance requirements. Those performance requirements are verified by standardized methods of test. Test result variability is influenced by both test variability and product variability. As it is product variability which needs to be identified, test variability has to be kept to a minimum. An important element in the achievement of this reduced variability is the calibration of test instrumentation, to traceable standards, at acceptable intervals.

The use of this British Standard will encourage proper calibration and make it possible for all users and calibration laboratories to work to common procedures.

BS 7825 will comprise the following three Parts:

Part 1	<i>Principles and general requirements</i>
Part 2	<i>Calibration procedures</i>
Part 3	<i>Calibration schedules</i>

Part 3 will comprise 13 Sections, each of which will provide calibration schedules for specific areas of rubber or plastics testing, as follows:

Section 3.1	<i>Thermal properties</i>
Section 3.2	<i>Electrical properties</i>
Section 3.3	<i>Mechanical properties of plastics</i>
Section 3.4	<i>Mechanical properties of rubber</i>
Section 3.5	<i>Optical and weathering properties</i>
Section 3.6	<i>Dimensional properties</i>
Section 3.7	<i>Rheological properties</i>
Section 3.8	<i>Degradation and environmental properties</i>
Section 3.9	<i>Flammability characteristics</i>
Section 3.10	<i>Conditioning, sample and test piece preparation</i>
Section 3.11	<i>Rubber polymer identification and chemical properties</i>
Section 3.12	<i>Chemical properties of plastics</i>
Section 3.13	<i>Chemical properties of resins</i>

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 22, an inside back cover and a back cover.

1 Scope

This Section of BS 7825 provides schedules for the calibration of a range of methods of test used for the evaluation of the thermal properties of plastics materials.

2 References

2.1 Normative references

This Section of BS 7825 incorporates, by dated or undated reference, provisions from other publications. These normative references are made at the appropriate places in the text and the cited publications are listed on page 21. For dated references, only the edition cited applies; any subsequent amendments to or revisions of the publications apply to this Section of BS 7825 only when incorporated in the reference by amendment or revision. For undated references, the latest edition of the cited publication applies, together with any amendments.

2.2 Informative references

This Section of BS 7825 refers to other publications that provide information or guidance. Editions of these publications current at the time of issue of this standard are listed on the inside back cover, but reference should be made to the latest editions.

3 Definitions

For the purposes of this Section of BS 7825 the definitions, where relevant, given in BS EN 30012-1, BS 5233, PD 6461 : Part 1, BS 4778 and ISO Guide 30 apply.

4 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded on any calibration report or certificate. It shall be reported whether calibration is made in the 'as-received' condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any requirements specified as approximate and hence not formally calibrated. If such parameters could change, then the need for periodic checks shall be written into the detailed calibration procedures.

5 Schedules

The calibration schedules given in this Section of BS 7825 have been compiled by listing all of the parameters specified in the relevant test method cited, together with the specified requirement. A parameter and requirement may relate to the main test apparatus, part of that apparatus or to an

ancillary apparatus necessary for the test. In some cases tolerances not given in the test method standard have been added.

For each parameter a calibration procedure is indicated by reference to Part 2 of BS 7825, to another publication or to a procedure particular to that test method which is detailed.

The verification frequency for each parameter is given by a code letter. The code letters used in the schedules are:

Procedure:	P	Particular procedure
	R	Use of certified reference material
	C	Requirement to be confirmed but no measurement
Requirement:	*	Tolerance not specified in the test method standard
Verification:	N	Initial verification only
	S	Standard interval as given in Part 1 of BS 7825
	P	Particular interval given in the standard
	U	In use

In use checks additional to the schedule are listed separately.

A list is given of reference materials either required by the calibration schedule or otherwise required by the test method.

Reagents, including cleaning materials, are listed. In some cases a reagent may be considered as a particular type of reference material.

In addition to the items listed, some standards assume the use of 'normal' laboratory apparatus, e.g. timers, beakers etc. These may need calibrating in accordance with the appropriate procedure given in Part 2 of this British Standard. Also, there may be items specified in the relevant cited standard which have a dimension, for example a 250 ml flask. Where the size has no particular significance, other than convenience of use, these items will not require to be calibrated.

Most standards for the testing of materials require the test piece to be conditioned. This implies the use of a thermometer and possibly a hygrometer, both of which will require calibration. Conditioning and test temperatures and humidities cited in the relevant standards have not been repeated.

Test piece dimensions are usually specified. This implies the use of dimensional measuring instruments which will require calibration. Test piece dimensions cited in the relevant standard have not been repeated.

6 Calibration schedules for individual test methods

The calibration schedules relating to this Section of BS 7825 are listed below in the numerical order of the international standard to which they relate:

BS EN ISO 75 :	<i>Plastics. Determination of temperature of deflection under load</i>	ISO 305 : 1990	<i>Plastics – Determination of thermal stability of poly(vinyl chloride), related chlorine-containing homopolymers and copolymers and their compounds – Discoloration method</i>
BS EN ISO 75-1 : 1996	<i>General test method</i> (Identical with ISO 75-1 : 1993)	BS 2782 :	<i>Methods of testing plastics</i>
BS EN ISO 75-2 : 1996	<i>Plastics and ebonite</i> (Identical with ISO 75-2 : 1993)	BS 2782 : Part 1 :	<i>Thermal properties</i>
BS EN ISO 75-3 : 1996	<i>High-strength thermosetting laminates and long-fibre reinforced plastics</i> (Identical with ISO 75-3 : 1993)	BS 2782 : Methods 120A, 120B, 120D and 120E : 1990	<i>Determination of Vicat softening temperature of thermoplastics</i> (Identical with ISO 306 : 1987)
BS 2782 :	<i>Methods of testing plastics</i>	BS 2782 : Method 153A : 1991	<i>Determination of stiffness in torsion of flexible materials (general method)</i> (Identical with ISO 458/1 : 1985)
BS 2782 : Part 1 :	<i>Thermal properties</i>	BS 2782 : Method 153B : 1991	<i>Determination of stiffness in torsion of flexible materials (method for vinyl chloride compounds)</i> (Identical with ISO 458/2 : 1985)
BS 2782 : Method 130A : 1991	<i>Determination of the thermal stability of polyvinyl chloride by the Congo red method</i> (Identical with ISO 182-1 : 1990)	BS 903 :	<i>Physical testing of rubber</i>
BS 2782 : Method 130B : 1991	<i>Determination of the thermal stability of polyvinyl chloride by the pH method</i> (Identical with ISO 182-2 : 1990)	BS 903 : Part A25 : 1992	<i>Determination of low-temperature brittleness</i> (Identical with ISO 812 : 1991)
BS 2782 : Method 130C : 1993	<i>Determination of the thermal stability of polyvinyl chloride by the conductometric method</i> (Identical with ISO 182-3 : 1993)	ISO 974 : 1980	<i>Plastics – Determination of the brittleness temperature by impact</i>
BS 2782 : Method 130D : 1993	<i>Determination of the thermal stability of polyvinyl chloride by the potentiometric method</i> (Identical with ISO 182-4 : 1993)	ISO 1133 : 1991	<i>Plastics – Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics</i> (A British Standard related to ISO 1133 : 1991 is BS 2782 : Part 7 : Method 720A : 1979)
		ISO 1147 : 1988	<i>Plastics — Polymer dispersions — Freeze-thaw cycle stability test</i>
		ISO 1218 : 1975	<i>Plastics — Polyamides — Determination of ‘melting point’</i> (British Standards related to ISO 1218 : 1975 are BS 2782 : Part 1 : Method 123A : 1976 and Method 123B : 1976)

BS 903 :	<i>Physical testing of rubber</i>	BS 2782 : Methods	<i>Determination of melting</i>
BS 903 : Part A13 :	<i>Method for determination of</i>	125A to 125C : 1991	<i>behaviour (melting</i>
1990	<i>stiffness at low temperature</i>		<i>temperature or melting range)</i>
	<i>(Gehman test) (Identical with</i>		<i>of semi-crystalline polymers</i>
	<i>ISO 1432 : 1988)</i>		<i>(Identical with ISO 3146 : 1985)</i>
BS 903 : Part A29 :	<i>Determination of low</i>	ISO 8570 : 1991	<i>Plastics — Film and sheeting —</i>
1984	<i>temperature characteristics by</i>		<i>Determination of cold-crack</i>
	<i>temperature-retraction</i>		<i>temperature</i>
	<i>procedure (TR test)</i>		<i>(A British Standard related to</i>
	<i>(Identical with ISO 2921 : 1982)</i>		<i>ISO 8570 : 1991 is BS 2782 :</i>
BS 2782 :	<i>Methods of testing plastics</i>		<i>Part 1 : Method 150D : 1976)</i>
BS 2782 : Part 1 :	<i>Thermal properties</i>		

Table 1. BS EN ISO 75-1 : 1996 (identical with ISO 75-1 : 1993)

Parameter	Requirement	Procedure	Verification frequency	Notes
Apparatus	As figure 1	–	–	Frame and rod to have same coefficient of expansion
Test specimen supports	Lines of contact in a horizontal plane	3.1	S	–
Vertical force	Midway	3.2	S	–
Contact edges	Parallel to loading edge	–	–	Longer than width of specimen
	Right angles to length	3.9	S	–
Radius	3.0 mm $\pm$ 0.2 mm	3.3	S	–
Rate of temperature rise	120 °C/h $\pm$ 10 °C/h	12.6	S	or 12 °C $\pm$ 1 °C in 6 mm
Temperature distribution	$\leq$ 1 °C	6	S	–
5.2.1 Heating bath with efficient stirrer	–	–	–	Specimen immersed to 50 mm
5.2.2 Oven with forced air or nitrogen circulation	$\geq$ 10 L/specimen holder about 60 times/min	3.8	S	–
	1.5 m/s to 2 m/s horizontal and perpendicular to specimen	4.3/12.4	S	–
Weights	1 g increments	11.2	S or U	–
Temperature measuring instrument	$\pm$ 0.5 °C or less	6	S	Calibrated at depth of immersion in 8.2
Deflection measuring instrument	$\pm$ 0.01 mm	3.1	S	–
Force is exerted by dial gauge spring	To be measured in the part of the range in which instrument is used	10.3	S	The mass of the rod must also be taken into account
Actual force applied	Calculated force $\pm$ 2.5 %	–	–	–
Micrometers and gauges for specimen dimensions	$\pm$ 0.01 mm	3.1	S	–
Thermometer position	$\leq$ 10 mm of specimen	3.2	U	But not in contact or by agreement if no error is introduced
Initial temperature	20 °C to 23 °C	6	S	–
Span	$\pm$ 0.5 mm	3.2	S	–
Stopclock	$\pm$ 1 s*	12.1	S	–
Reagents	Oil for heating batch	See NOTE 3 to 5.2.1	–	–

NOTE. A blank test with invar or borosilicate glass is to be run to determine the correction term for expansion. If equal to or greater than 0.010 mm it is to be noted.

Table 2. BS EN ISO 75-2 : 1996 (identical with ISO 75-2 : 1993)

Parameter	Requirement	Procedure	Verification frequency	Notes
Apparatus and conditions	As in ISO 75-1	—	—	—
Span	64 mm $\pm$ 1 mm or 100 mm $\pm$ 2 mm	3.2 3.2	S S	Flatwise Edgewise
Force applied F	A 1.800 MPa B 0.045 MPa C 8.000 MPa	10.3	U	—
Actual force applied	$F \pm 2.5\%$	—	—	—

Table 3. BS EN ISO 75-3 : 1996 (identical with ISO 75-3 : 1993)

Parameter	Requirement	Procedure	Verification frequency	Notes
Apparatus and conditions	As ISO 75-1	—	—	—
Span	$(30 \pm 2) \times$ thickness h of test specimen	3.2	U	i.e. 60 mm to 210 mm
Force applied to produce bending stress	1/10 flexural strength in relevant standard	10.3	U	Or 1/10 determined value as ISO 178

Table 4. BS 2782 : Method 130A : 1991 (identical with ISO 182-1 : 1990)

Parameter	Requirement	Procedure	Verification frequency	Notes
Test tubes: external diameter wall thickness length	Approximately 17 mm Approximately 0.4 mm ≥ 150 mm	C C C	N N N	— — —
Stoppers with hole	Interference fit for glass tubes	—	—	—
Glass tubes	2 mm to 3 mm internal diameter about 100 mm long ± 2 mm*	C	N	—
Oil batch capacity	≥ 10 L	3.8	N	—
Heat resistant shield	See figure 1 and 5.4	C	N	—
Thermometer	Scale divisions 0.1 °C	6	S	—
Sieve	2.0 mm ISO 565 R20/3 series	3.6	N+	—
Sieve	1.4 mm ISO 565	3.6	N+	—
Up to four stop clocks	Time in minutes ± 1 s*	12.1	S	—
Indicator paper: length width	30 mm ± 1 mm* 10 mm ± 0.5 mm*	— —	— —	— —
Height above sample	25 mm ± 0.5 mm*	—	U	—
Reagents	Congo-red indicator paper in 10 mm wide strips or Universal indicator paper pH range 1 to 10 Water, Grade 2 (see ISO 3696) Oil for oil bath (to withstand 210 °C)	— — —	— — —	— — —