

Calibration of rubber and plastics test equipment

Part 3. Calibration schedules

Section 3.7 Rheological properties

ICS 83.200

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Committees responsible for this British Standard

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- British Rubber Manufacturers' Association Ltd.
- National Physical Laboratory
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Contents

	Page
Committees responsible	Inside front cover
Foreword	ii
1 Scope	1
2 Normative references	1
3 Definitions	1
4 Inspection	1
5 Schedules	1
6 Calibration schedules for individual test methods	2
Tables	
1 ISO 289-1 : 1994 and ISO 289-2 : 1994	3
2 BS 2782 : Method 733A : 1996	4
3 BS 2782 : Method 733C : 1991	5
4 ISO 1599 : 1990	6
5 BS 2782 : Method 732A : 1991	7
6 BS 2782 : Method 732B : 1991	8
7 BS 2782 : Method 732C : 1991	9
8 BS 2782 : Method 732D : 1991	10
9 BS 2782 : Method 732E : 1991	11
10 BS 2782 : Method 732F : 1991	12
11 BS EN ISO 3219 : 1995	13
12 BS 2782 : Method 730C : 1992	13
13 BS 903 : Section A60.2 : 1992	14
14 ISO 4575 : 1985	15
15 ISO 6186 : 1980	16
16 ISO 6502 : 1991	16
List of references	Inside back cover

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 comprises the following three separate Parts:

Part 1	<i>Specification for 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>

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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 18, an inside back cover and a back cover.

1 Scope

This Section of BS 7825 provides schedules for the calibration of equipment and apparatus used for the measurement of the rheological properties of plastics and rubber materials.

2 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 the inside back cover. 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.

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 BS 7825. 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. Standard conditioning and test temperatures and humidities cited in the relevant standard 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:

ISO 289-1 : 1994	<i>Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity</i>	BS 2782 : Method 732E : 1991	<i>Determination of viscosity number of poly(alkylene terephthalates)</i> (Identical with ISO 1628-5 : 1986)
ISO 289-2 : 1994	<i>Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 2: Determination of pre-vulcanization characteristics</i>	BS 2782 : Method 732F : 1991	<i>Determination of viscosity number of methyl methacrylate polymers</i> (Identical with ISO 1628-6 : 1990)
BS 2782 : BS 2782 : Part 7 : BS 2782 : Method 733A : 1996	<i>Methods of testing plastics</i> <i>Rheological properties</i> <i>Determination of viscosity number of polyamides</i> (Identical with ISO 307 : 1994)	BS EN ISO 3219 : 1995 (Also numbered BS 2782 : Part 7 : Method 730B : 1994)	<i>Plastics. Polymers/resins in the liquid state or as emulsions or dispersions. Determination of viscosity using a rotational viscometer with defined shear rate</i> (Identical with ISO 3219 : 1993)
BS 2782 : Method 733C : 1991	<i>Determination of viscosity number and viscosity ratio of cellulose acetate</i> (Identical with ISO 1157 : 1990)	BS 2782 : BS 2782 : Part 7 : BS 2782 : Method 730C : 1992	<i>Methods of testing plastics</i> <i>Rheological properties</i> <i>Determination of apparent viscosity of resins by the Brookfield Test method</i> (Identical with ISO 2555 : 1989)
ISO 1599 : 1990	<i>Plastics — Cellulose acetate — Determination of viscosity loss on moulding</i>	BS 903 : BS 903 : Part A60 : BS 903 : Section A60.2 : 1992	<i>Physical testing of rubber</i> <i>Curemetering</i> <i>Method for the determination of vulcanization characteristics using an oscillating disc curemeter</i> (Identical with ISO 3417 : 1991)
BS 2782 : BS 2782 : Part 7: BS 2782 : Method 732A : 1991	<i>Methods of testing plastics</i> <i>Rheological properties</i> <i>Determination of viscosity number: general conditions</i> (Identical with ISO 1628-1 : 1984)	ISO 4575 : 1985	<i>Plastics — Polyvinyl chloride pastes — Determination of apparent viscosity using the Severs rheometer</i>
BS 2782 : Method 732B : 1991	<i>Determination of viscosity number of poly(vinyl chloride) resins</i> (Identical with ISO 1628-2 : 1988)	ISO 6186 : 1980	<i>Plastics — Determination of pourability</i>
BS 2782 : Method 732C : 1991	<i>Determination of viscosity number of polyethylenes and polypropylenes</i> (Identical with ISO 1628-3 : 1991)	ISO 6502 : 1991	<i>Rubber — Measurement of vulcanization characteristics with rotorless curemeters</i>
BS 2782 : Method 732D : 1991	<i>Determination of viscosity number of polycarbonate moulding and extrusion materials</i> (Identical with ISO 1628-4 : 1986)		

Table 1. ISO 289-1 : 1994 and ISO 289-2 : 1994

Parameter	Requirement	Procedure	Verification frequency	Notes
Rotor diameter	(38.10 ± 0.03) mm	3.2	N	—
Rotor thickness	(30.48 ± 0.03) mm (5.54 ± 0.03) mm		N	—
Cavity diameter	(50.9 ± 0.1) mm	3.2	N	—
Cavity depth	(10.59 ± 0.03) mm	3.2	N	—
Die and rotor material	Hardened steel, Rockwell 60 HRC minimum	8.1	N	—
Radial V grooves	20° intervals, within diameters of 47 mm and 7 mm for the upper die and to within 1.5 mm of the hole in the lower die.	3.9	—	—
		3.2	—	—
		3.2	—	—
	Each groove to form a 90° angle with the die surface and be (1 ± 0.1) mm wide	3.9	N	—
Rectangular grooves	(0.80 ± 0.02) mm wide, (0.30 ± 0.05) mm deep and spaced (1.60 ± 0.04) mm apart Flat surfaces to have two sets at right angles, the edges to have vertical grooves, 75 for large rotors and 60 for small rotors	3.2	N	—
		C	N	—
Shaft	(10 ± 1) mm diameter such that top and bottom clearances do not differ by more than 0.25 mm.	3.2	N	—
	To bear on the spindle	3.2	S	—
Shaft clearance	Small enough to prevent rubber leaving	C	S	—
Rotor eccentricity	0.1 mm max.	3.2	S	—
Angular velocity	(0.209 ± 0.002) rad/s	12.4	S	—
Heating device	Within ± 0.5 °C of test temperature.	6	S	—
	Return to ± 0.5 °C of temperature within 4 min	C	S	—
Thermocouple probes and temperature sensors	Positioned as in figures 1 and 4	C	N	—
	Accurate to ± 0.25 °C	6	S	—
Die closure pressure	(11.5 ± 0.5) kN	10.2	S	—
	Give even pattern on tissue paper	C	S	—
Torque measurement device	0.083 N·m = 1 MU Scale read to 0.5 MU Zero variation less than 0.5 MU	10.4	S	—
Reference material	Butyl rubber designated IIR-NIST-SRM 388	—	—	—

Table 2. BS 2782 : Method 733A : 1996 (identical with ISO 307 : 1994)

Parameter	Requirement	Procedure	Verification frequency	Notes
Vacuum drying cabinet	Pressure < 100 kPa	4.4	S	Temperature of 80 °C to 100 °C
Balance	Accurate to 0.1 mg	11.1	S	—
Volumetric flask	50 ml to ISO 1042	7.1	N	—
Sintered glass filter or stainless steel sieve	40 µm to 100 µm (grade P100) apertures about 0.075 mm ²	3.6	N	—
Ubbelohde viscometer with formic acid	Conforming to ISO 3105 Type 1	4.2	S	—
with sulfuric acid	Type 2	4.2	S	Diameter 0.58 mm ± 2 %
with m-cresol	Type 2	4.2	S	Diameter 1.03 mm ± 2 %
Location of viscometer	Vertical with graduation mark E (as in figure 1) at least 30 mm below surface of liquid in the bath	3.2	U	—
Thermostatic bath	(25 ± 0.05) °C	6	S	—
Stop watch	Accurate to 0.1 s	12.1	S	—
Thermometer	Reading to 100 °C	6	S	—
Centrifuge	Frequency approx. 50 s ⁻¹	12.3	S	Alternative (10.3)
Reagents				
Solvents	All analytical grade Distilled water or water of equivalent purity	—	—	—
	Sulfuric acid (96 ± 0.15) % (m/m) solution checked at least every two weeks	—	—	—
	Formic acid (90 ± 0.15) % (m/m) solution checked at least every two weeks	—	—	—
	M-cresol, clear and colourless 99 % (m/m) min.	—	—	—
	0-cresol content 0.3 % (m/m) max. water content 0.13 % (m/m) max. May be obtained by distillation of chemically pure m-cresol (preferably in vacuo)	—	—	—
	Nitrogen shall be used for pressure compensation. Its purity may be checked by gas chromatography	—	—	—