

Calibration of rubber and plastics test equipment —

Part 3: Calibration schedules —

Section 3.11 Rubber polymer
identification and chemical properties

ICS 83.020

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Committees responsible for this British Standard

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- British Rubber Manufacturers' Association Ltd.
- National Physical Laboratory
- GAMBICA (BEAMA) Ltd.
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Foreword

This Section of Part 3 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 requirements, 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>

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 28, 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 identification of rubber polymers and the determination of their chemical properties and constituents.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of this British Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. For undated references, the latest edition of the publication referred to applies.

BS 572, *Specification for interchangeable conical ground glass joints.*

BS 903-A19, *Physical testing of rubber. Heat resistance and accelerated ageing tests.*

BS 903-A54, *Physical testing of rubber. Methods for exposure to artificial light, for use in determining resistance to weathering.*

BS 903-A64, *Physical testing of rubber. Method for the preparation, mixing and vulcanization of rubber test mixes.*

BS 1752, *Specification for laboratory sintered or fritted filters including porosity grading.*

BS 6057-3.19, *Determination of total bound styrene content of reinforced styrene-butadiene rubber latices.*

BS 4181-1:1985, *Method for identification of hydrocarbon, chloroprene, nitrile and chlorosulphonated polyethylene rubbers.*

BS 6630:1985, *Method for identification of antidegradants in rubber and rubber products by thin layer chromatography.*

BS 7164-2-2.2:1990, *Chemical tests for raw and vulcanized rubber — Part 2: Sample preparation — Section 2.2: Vulcanized rubber.*

BS 7164-3:1992, *Chemical tests for raw and vulcanized rubber — Part 3: Methods for determination of solvent extract.*

BS 7164-4:1990, *Chemical tests for raw and vulcanized rubber — Part 4: Methods for dissolution of rubber by acid digestion.*

BS 7164-5:1991, *Chemical tests for raw and vulcanized rubber — Part 5: Methods for determination of ash content.*

BS 7164-7.1:1990, *Chemical tests for raw and vulcanized rubber — Part 7: Methods for determination of polymer content — Section 7.1: Polyisoprene content.*

BS 7164-22-22.2:1992, *Chemical tests for raw and vulcanized rubber — Part 22: Determination of chlorine content — Section 22.2: Oxygen flask combustion technique for determination of bromine and chlorine.*

BS 7164-23.1:1993, *Chemical tests for raw and vulcanized rubber — Part 23: Methods for determination of total sulfur content — Section 23.1: Oxygen combustion flask method.*

BS 7164-25:1996, *Chemical tests for raw and vulcanized rubber — Part 25: Method for determination of sulfide sulfur content.*

BS 7164-26-26.1:1997, *Chemical tests for raw and vulcanized rubber — Part 26: Methods for determination of manganese content — Section 26.1: Atomic absorption spectrometry.*

BS 7164-27-27.1:1991, *Chemical tests for raw and vulcanized rubber — Part 27: Methods for determination of iron content — Section 27.1: Atomic absorption spectrometry.*

BS 7164-28-28.1:1997, *Chemical tests for raw and vulcanized rubber — Part 28: Methods for determination of copper content — Section 28.1: Atomic absorption spectrometry.*

BS 7164-28-28.2:1995, *Chemical tests for raw and vulcanized rubber — Part 28: Methods for determination of copper content — Section 28.2: Photometric method.*

BS 7164-29-29.1:1992, *Chemical tests for raw and vulcanized rubber — Part 29: Methods for determination of zinc content — Section 29.1: Atomic absorption spectrometry. (Identical with ISO 6101-1:1991)*

BS 7164-30-30.1:1997, *Chemical tests for raw and vulcanized rubber — Part 30: Methods for determination of lead content — Section 30.1: Atomic absorption spectrometry. (Identical with ISO 6101-2:1986)*

BS EN 30012-1:1994, *Metrological confirmation system for measuring equipment.*

BS EN ISO 105-A01, *Textiles. Tests for colour fastness. General principles of testing.*

BS EN ISO 658:1995, *Oilseeds. Determination of impurities content.*

BS ISO 1042, *Specification for one-mark volumetric flasks.*

BS ISO 3865:1997, *Rubber, vulcanized or thermoplastic. Methods of test for staining in contact with organic material.*

PD 6461-1:1995, *Basic and general terms (international).*

ISO 105-A02, *Textiles — Tests for colour fastness — Part A02: Grey scale for assessing change in colour.*

ISO 105-B01, *Textiles — Tests for colour fastness — Part B01: Colour fastness to light: daylight.*

ISO 383, *Laboratory glassware; Interchangeable conical ground joints.*
ISO 385-1, *Laboratory glassware; Burettes; Part 1 :General requirements.*
ISO 648, *Laboratory glassware; One-mark pipettes.*
ISO 1772, *Laboratory crucibles in porcelain and silica.*
ISO 2454:1995, *Rubber products — Determination of zinc content—EDTA titrimetric method.*
ISO 5478:1990, *Rubber — Determination of styrene content — Nitration method.*
ISO 6528-2:1992, *Rubber — Determination of total sulfur content — Sodium peroxide fusion method.*
ISO 6528-3:1988, *Rubber — Determination of total sulfur content — Furnace combustion method.*
ISO 7270:1987, *Rubber — Identification of polymers (single polymer and blends) — Pyrolytic gas chromatographic method.*
ISO Guide 30 : 1992

3 Definitions

For the purposes of this section of BS 7825 the definitions, where relevant, given in BS EN 30012-1, BS 5233, PD 6461-1, 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 BS 7825-2, 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 BS 7825-1
	U	In use

In-use checks additional to the schedule are listed separately.

A list of reference materials is given, 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 the size has no particular significance, other than convenience of use, these items will not require calibration.

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:

BS 7164-3:1992	<i>Chemical tests for raw and vulcanized rubber — Methods for determination of solvent extract</i> (Identical with ISO 1407:1992)
ISO 2454:1995	<i>Rubber products — Determination of zinc content — EDTA titrimetric method</i>
BS ISO 3865:1997	<i>Rubber, vulcanized or thermoplastic. Methods of test for staining in contact with organic material</i>
BS 6630:1985	<i>Method for identification of antidegradants in rubber and rubber products by thin layer chromatography</i> (Identical with ISO 4645:1984)
BS 4181-1:1985	<i>Method for identification of hydrocarbon, chloroprene, nitrile and chlorosulphonated polyethylene rubbers</i> (Identical with ISO 4650:1984)
BS 7164-2-2.2:1990	<i>Chemical tests for raw and vulcanized rubber — Part 2: Sample preparation — Section 2.2: Vulcanized rubber</i> (Identical with ISO 4661-2:1987)
ISO 5478:1990	<i>Rubber — Determination of styrene content — Nitration method</i>
BS 7164-7-7.1:1990	<i>Chemical tests for raw and vulcanized rubber — Part 7: Methods for determination of polymer content — Section 7.1: Polyisoprene content</i> (Identical with ISO 5945:1989)
BS 7164-29-29.1:1992	<i>Chemical tests for raw and vulcanized rubber — Part 29: Determination of zinc content — Section 29.1: Atomic absorption spectrometry</i> (Identical with ISO 6101-1:1991)
BS 7164-30-30.1:1997	<i>Chemical tests for raw and vulcanized rubber — Part 30: Determination of lead content — Section 30.1: Atomic absorption spectrometry</i> (Identical with ISO 6101-2:1986)
BS 7164-28-28.1:1997	<i>Chemical tests for raw and vulcanized rubber — Part 28: Methods for determination of copper content — Section 28.1: Atomic absorption spectrometry</i> (Identical with ISO 6101-3:1997)
BS 7164-26-26.1:1997	<i>Chemical tests for raw and vulcanized rubber — Part 26: Methods for determination of manganese content — Section 26.1: Atomic absorption spectrometry</i> (Identical with ISO 6101-4:1997)
BS 7164-27-27.1:1991	<i>Chemical tests for raw and vulcanized rubber — Part 27: Methods for determination of iron content — Section 27.1: Atomic absorption spectrometry</i> (Identical with ISO 6101-5:1990)
BS 7164-23-23.1:1993	<i>Chemical tests for raw and vulcanized rubber — Part 23: Methods for determination of total sulfur content — Section 23.1: Oxygen combustion flask method</i> (Identical with ISO 6528-1:1992)
ISO 6528-2:1992	<i>Rubber — Determination of total sulfur content — Sodium peroxide fusion method</i>
ISO 6528-3:1988	<i>Rubber — Determination of total sulfur content — Furnace combustion method</i>
ISO 7270:1987	<i>Rubber — Identification of polymers (single polymer and blends) — Pyrolytic gas chromatographic method</i>

BS 7164-22-22.2:1992	<i>Chemical tests for raw and vulcanized rubber — Part 22: Determination of chlorine content — Section 22.2: Oxygen flask combustion technique for determination of bromine and chlorine</i> (Identical with ISO 7725:1991)
BS 7164-28-28.2:1995	<i>Chemical tests for raw and vulcanized rubber — Part 28: Methods for determination of copper content — Section 28.2: Photometric method</i> (Identical with ISO 8053:1995)
BS 7164-25:1996	<i>Chemical tests for raw and vulcanized rubber — Method for determination of sulfide sulfur content</i> (Identical with ISO 8054:1996)
BS 7164-4:1990	<i>Chemical tests for raw and vulcanized rubber — Part 4: Methods for dissolution of rubber by acid digestion</i> (Identical with ISO 9028:1989)

Table 1 — BS 7164-3:1992 (Identical with ISO 1407:1992)

Parameter	Requirement	Procedure	Verification frequency	Notes
Extraction apparatus	3 types described, any similar accepted	C	U	6.1a), b), and (c)
Oven	(100 ± 2) °C	6	S	—
Sieve	150 µm (100 mesh)	3.6	N	—
Method A				—
Mass of test portion	3 g to 5 g weighed to 0.1 mg	—	—	—
Mass of dry flask	Nearest 0.1 mg	—	—	—
Distillation rate: method A	10 to 20 times per h	—	U	—
Extraction time: method A	(16 ± 0.5) h	—	—	—
Mass of flask and content	Nearest 0.1 mg	—	—	—
Method B				—
Mass of test portion	90 mg to 110 mg weighed to nearest 0.1 mg	—	—	—
Mass after extraction	Nearest 0.1 mg	—	—	—
Reagents				
All of analytical quality	Acetone Ethanol/Toluene azeotropic mixture as 5.2 Propan-2-ol (Isopropanol) Methanol Desiccant Absorbent tissue			

Table 2 — ISO 2454:1995

Parameter	Requirement	Procedure	Verification frequency	Notes
Balance	± 0.1 mg	11.1	S	—
Pipette	25 cm ³ Grade A*	7.1	N	—
Volume of HCl	5 cm ³	—	—	—
Muffle furnace	(550 $\pm$ 25) °C	6	S	—
Burette	10 cm ³ graduated 0.02 cm ³ as in ISO 385-1 Class A	7.1	N	Glassware marked in “ml” or “cc” is acceptable
Burette	50 cm ³ graduated 0.1 cm ³ as in ISO 385-1 Class A	7.1	N	—
Crucible	Platinum 50 cm ³	C	N	Volume is not critical
Mass of test piece	Approximately 1 g weighed to ± 0.1 mg	11.1	S	—
Ash	Method B or C of BS 7164-5 (ISO 247)	—	—	—
Volumetric flask	100 cm ³ Grade A*	—	—	—
Aliquot portions	25 cm ³ Grade A* or 10 cm ³ Grade A	—	—	—
Reagents All of analytical quality	Distilled water or water of equivalent purity Acetone 2,4-Pentanedione 10 % (V/V) solution in acetone Hydrochloric acid of density 1.18 Mg/m ³ Sulfuric acid of density 1.84 Mg/m ³ Hydrofluoric acid 48 % (m/m) solution Ammonium hydroxide of density 0.91 Mg/m ³ solution Buffer solution (as in 4.7) Aluminium chloride c0,1 mol/dm ³ solution (as in 4.8) Magnesium chloride c0.1 mol/dm ³ solution (as in 4.9) Ammonium fluoride c3 mol/dm ³ solution (as in 4.10) Zinc, standard reference solution 1 g Zn/dm ³ (as in 4.11) Ethylenedinitrilotetra-acetic acid disodium salt dihydrate, approximately 0.01 mol/dm ³ standard volumetric solution Dithiozone indicator (as in 4.13) Desiccant, universal indicator paper, ashless filter paper, etc.			

Table 3 — BS ISO 3865:1997

Parameter	Requirement	Procedure	Verification frequency	Notes
Air circulating oven	As in BS 903-A19 (ISO 188)	—	—	—
Xenon arc lamp	As in BS 903-A54 (ISO 4665-3), methods A, B and C with radiant flux density of 1 000 W/m ² in the wavelength range 300 nm to 830 nm	—	—	—
Thermocouple or black panel thermometer	For surface temperature methods A, B and C	—	—	See BS 903-A54 (ISO 4665-3)
Apparatus for light intensity	Wavelengths (as in 8.1) methods A, B or C	5.1	S	Recommended, not mandatory
Blue dyed wool standards	As in ISO 105-B01, methods A, B and C	—	—	—
Grey scale	As ISO 105-A02, methods A, B and C	—	—	—
Reflectance spectrometer	40 nm to 600 nm, methods A, B and C	7.3	U	—
Rubber mill	Preferably as in BS 903-A64:1995 (ISO 2393)	—	—	—
Protection	E.g. starched cambric or polyethylene	C	U	—
Panels	White acrylic-based stove enamel	C	N	—
Lacquer drying	30 min at 125 °C	6	S	—
Panel dimensions	At least 20 mm around the test piece	C	U	—
Pressure	(7 ± 1) kPa	4.6	U	—
Distance between test pieces	≥40 mm	C	U	—
Oven temperature	(70 ± 2) °C	6	S	—
Storage time	24 (+0, -2) h	—	—	—
Drip rate	1 dm ³ in 24 h	C	U	—
Dripping apparatus	As in Figure 1	C	U	—
Irradiation	Xenon	—	—	e.g. 1 500 W
Radiant flux density	(1 000 ± 200) W/m ²	5.1	S	—
Wavelength range	300 nm to 830 nm	C 5.1 —	N S —	— — —
Irradiation period: Xenon	— 24 h, 48 h, 150 h	— — —	— — —	— Or alternative as in 8.2 —
Surface temperature	(55 ± 3) °C with black panel thermometer	6	S	—

Table 3 — BS ISO 3865:1997 (continued)

Parameter	Requirement	Procedure	Verification frequency	Notes
Variation in intensity of radiation	Not more than 10 % from the mean	5.1	U	Rotation helps
Annular zone	90 mm to 300 mm dia.	C	N	—
Rotation	3 r/min	C	U	—
Visual assessment: using grey scale using reflectance spectrometer	As in BS EN ISO 105:A01 As in ISO 105:A02, 400 nm to 600 nm	— 5.3	— U	— Relative to blank
Calibration of reflectance spectrometer: Minimum 3 wavelengths	Using barium sulfate e.g. 445 nm, 555 nm and 600 nm	7.3 5.3	U U	— —
Colour change	Reading ρ minus Blank reading ρ_0	Calculated as in 10.3	— —	Negative values indicate darkening Positive values indicate lightening
Reagent	Water containing 2 % of a non-alkaline detergent			

Table 4 — BS 6630:1985 (Identical with ISO 4645:1984)

Parameter	Requirement	Procedure	Verification frequency	Notes
Thickness of coating	250 μm to 300 μm	C	U	Thinner coatings permissible providing they give good separation
Oven	(100 $\pm$ 5) $^{\circ}\text{C}$	6	S	—
Desiccator	—	—	—	—
Chromatographic developing tanks	Large enough (not the small “sandwich type”)	C	U	—
Extraction apparatus	As in BS 7164-3 (ISO 1407)	—	—	—
Short liquid-solid chromatographic column	E.g. 5 cm^3 hypodermic syringe barrel with needle approximately 35 mm long and 1.25 mm in diameter, or glass tubes 120 mm long, 10 mm long and 10 mm to 12 mm in diameter holding approximately 5 cm^3 silica gel	C	U	—
Spray apparatus	Mask optional	—	—	—
Micro pipettes	2 mm^3 , 5 mm^3 and 10 mm^3	7.1	N	—
Extraction	As in BS 7164-3 (ISO 1407)	—	—	—
Rubber pieces	Length of edges <2 mm	—	—	—
Hot plate	$\leq 50^{\circ}\text{C}$	6	S	—
Silica gel column	Freshly prepared or stored in desiccator for not more than 2 h	C	U	—