BS 544:1969

Incorporating Amendment Nos. 1, 2, 3 and 4

Specification for

Linseed oil putty for use in wooden frames



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Co-operating organizations

The Committee responsible for the preparation of this British Standard consists of representatives from the following Government departments and scientific and industrial organizations:

British Association of Synthetic Rubber Manufacturers British Woodwork Manufacturers' Association Flat Glass Associations Greater London Council Institution of Municipal Engineers Metal Window Federation of Great Britain Ministry of Public Building and Works Ministry of Public Building and Works — Building Research Station National Association of Putty and Building Mastics Manufacturers Research Association of British Paint, Colour and Varnish Manufacturers Royal Institute of British Architects Welwyn Hall Research Association

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Foreword

This standard makes reference to the following British Standards: BS 242, BS 243, BS 259, BS 632, Specifications for linseed oil. BS 245, Specification for mineral solvents (white spirit and related hydrocarbon solvents) for paints and other purposes. BS 410, Specification for test sieves. BS 572, Interchangeable conical ground glass joints. BS 1795, Extenders for paints. BS 2021, Specification for separating and dropping funnels for laboratory use. BS 3406, Methods for the determination of particle size of powders. BS 3591, Industrial methylated spirits. BS 3900, Methods of test for paints. BS 3900-B1, Determination of water by the Dean and Stark method. BS 3900-C2, Surface-drying test (ballotini method). BS 5358, Specification for low-lead solvent-thinned priming paint for woodwork. BS 5922, Specification for glass condensers for laboratory use. BS 6262, Code of practice for glazing for buildings. This British Standard was originally published in 1934 and revised in 1939 and 1969. Subsequently two amendments have been published under the direction of the Elements and Components (of Diverse Materials) for Buildings Standards Committee. Amendment No.2 permits putties based on limestone fillers as well as putties

Amendment No.2 permits putties based on inmestone fillers as well as putties incorporating whiting to comply with the standard. The composition of putties will be periodically reviewed in conjunction with the preparation of a new British Standard giving performance requirements for all types of glazing compound.

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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 specifies the requirements and composition of linseed oil putty for use in glazing wooden frames as recommended in BS 6262¹).

NOTE The paintability test in this standard only covers the compatibility of putties with priming paints complying with BS 5358^{20} . The use of other types of paint with putties should be agreed between the specifier and the manufacturer.

2 Sampling

For the purpose of examination under Clauses 4, 5, 6 and 8 of this specification samples shall be drawn, and prepared by one of the methods described in Appendix A. Only original unopened containers shall be sampled.

NOTE The number of containers sampled should be by agreement between the purchaser and the vendor. As a guide it is suggested

that the number of containers sampled should be $\sqrt{\frac{n}{2}}$ where *n* is the number of containers in each separate batch.

3 Text deleted

4 Composition

The material shall consist of a homogeneous paste composed of the following.

Raw linseed oil complying with BS 243, 632^a or refined linseed oil complying with BS 242^a	Not less than 10.0 % by mass as determined within 3 months from the date of manufacture by the method given in Appendix C
Additives taken as the unsaponifiable matter determined by the method given in Appendix C	Not more than 2.5 % by mass of the extracted oil
Water	Not more than 0.5 % by mass as determined by the method described in BS 3900-B1 ^b
Filler (as specified in Appendix B)	Remainder as determined by the method given in Appendix C

^a BS 242, BS 243, BS 259, BS 632, "Specifications for linseed oil".

^b BS 3900, "Methods of test for paints" — Part B1: "Determination of water by the Dean and Stark method".

5 Paintability

When tested according to the method described in Appendix D, the condition of the material shall be such that:

1) It is capable of being painted after drying for a 48 hours period with white priming paint complying with BS 5358^{2} without any lifting or disturbance of its surface.

2) The coat of white priming paint applied as in 1) shall dry in 24 hours and not show any discoloration, wrinkling, grittiness or other defect.

NOTE Some timber preservatives, particularly those containing wax or silicones and some water thinnable wood primers are found to give adhesion and drying problems when used with putties under certain conditions. The manufacturers' advice should be sought where these types of preservative or primer are used.

6 Workability

The material shall work readily and smoothly in the hand and under a knife when used in glazing.

¹⁾ BS 6262, "Code of practice for glazing for buildings".

²⁾ BS 5358, "Specification for low-lead solvent-thinned priming paint for woodwork".

7 Consistency

The consistency of the material shall be agreed between the purchaser and the vendor. Where the container is of an appropriate size, the method of determining consistency as described in Appendix E shall be used and the consistency of the material shall be such that the depth of penetration is within \pm 20 % of the figure agreed between purchaser and vendor.

NOTE The consistency is specified as the depth of penetration in a certain time with a stated load. The requirements of most glaziers will be met by materials which are stiffer than those where the penetration is 300 mm, 25 s, 2 500 g and softer than 200 mm, 15 s, 4 000 g where the weight is the superimposed load. A method of determining consistency is given in Appendix E.

8 Skins and coarse particles

The material shall not yield skins and coarse particles in excess of the following limits when tested in the manner described in Appendix F:

0.1 % on a sieve of nominal aperture $425 \,\mu m \, (36 \, mesh)^{3)}$

7.0 % on a sieve of nominal aperture 63 μ m (240 mesh)³).

9 Keeping qualities and storage stability

The material when stored in the original unopened container for a period not exceeding 3 months in conditions free from undue vibration and at a temperature of 15 ± 10 °C, shall not be caked or hardened. Any surface skin shall be continuous and easily removable.

10 Marking

Each container in which the material is packed shall be clearly and indelibly marked with the following information:

a) name of identification mark of the supplier or manufacturer;

- b) number and date of this British Standard;
- c) batch number, manufacturing date and shelf life;
- d) storage temperature range and conditions relevant to the stated expiry date.

³⁾ BS 410, "Specification for test sieves".

Appendix A Methods for sampling

A.1 Method 1: (For sampling kegs and large containers)

A.1.1 Apparatus. The following apparatus is required:

1) Auger, of mild steel as described in Figure 1a.

2) Tommy bar, of silver steel, dimensions 200 mm × 9.5 mm (8 in × ¾ in) (see Figure 1a).

3) Tube, of 1.63 mm (0.064 in, 16 SWG) aluminium alloy, dia. 29 mm (11/2 in) and

length 520 mm (201/2 in) fitted with a handle (see Figure 1b).

A.1.2 *Procedure.* Remove any surface skin. Then remove any oil layer on the surface of the putty by means of a pipette and carefully weigh and retain. Calculate the oil as a percentage of the total weight of the putty. Slowly screw the auger into the putty at the centre of the surface so as to avoid producing a large depression at the surface of the putty and making sure the auger reaches to the bottom of the container.

Gently press the area around the auger with a palette knife to ensure that the top $13 - 25 \text{ mm} (\frac{1}{2} - 1 \text{ in})$ of the putty is in contact with the auger and allow the auger to remain in the putty for 10 minutes.

Then insert the tube into the putty so that it is concentric with the auger and withdraw both the auger and tube.

Without rotating the auger withdraw it from the tube. Remove the putty from the auger and mix it thoroughly, adding the proportionate amount of the retained oil layer previously removed as above. Divide the putty into 3 portions, each weighing not less than 450 g (1 lb) and pack each portion in a clean,

dry, airtight metal or glass container of such size that it is nearly as possible filled by the putty.

Seal each container and mark it with full details and date of sampling.

A.2 Method 2:

(To be used where the size of the container makes it inappropriate to use Method 1.)

Remove any surface skin and, if necessary remove the entire contents of the container and thoroughly mix. The samples of putty should then be packed, and labelled as described above in Method 1.

Appendix B Specification for filler

The material shall consist of a finely divided powder prepared from calcium carbonate and complying with the following specification:

Size of particles (µm)	% undersize	% cumulative residue	Test method described in
$500 \\ 125 \\ 63 \\ 45$	99.99 or more 99.00 or more 94.00 or more 84.00 or more	0.01 max. 1.0 max. 6.0 max. 16.0 max.	Clause 8 of BS 1795 ⁴⁾ :1976
$\begin{array}{c} 32 \\ 4 \end{array}$	92.0 or less 40 to 65		Clause 9 of BS 1795 ⁴⁾ :1976

1) Particle size distribution.

2) Calcium carbonate content. When tested as described in clause 15 of BS 1795^{4} :1976 the material shall have a calcium carbonate content of not less than 94 %.

3) pH value of aqueous suspension. When tested as described in clause **13** or BS 1795⁴):1976 the pH value of the aqueous suspension of the material shall be in the range 8 to 10.

4) *Matter volatile at* 105 °C. When tested as described in clause **10** of BS 1795⁴):1976 the percentage loss in mass of the material as delivered shall be not greater than 0.25 %.

⁴⁾ BS 1795, "Specification for extenders for paints".

Appendix C Method for the determination of oil content, whiting content and the unsaponifiable matter on the extracted oil

NOTE Full laboratory precautions should be taken when using diethyl ether.

C.1 Apparatus

The following apparatus is required:

- 1) Centrifuge.
- 2) Centrifuge tube, 100 ml capacity.
- 3) *Flask*, 500 ml, flat-bottomed, fitted with a B24 cone complying with BS 572^{5} .
- 4) Condenser, glass, complying with BS 5922⁶⁾ and fitted with a B24 cone complying with BS 572.
- 5) Separating funnel (2), 250 ml, complying with BS 2021⁷⁾.

C.2 Reagents

The reagents used shall be of recognized analytical reagent quality. Distilled water or water otherwise prepared of equal purity shall be used throughout.

1) *Potassium hydroxide*. 0.5N solution in 95 % (v/v) ethanol⁸⁾. The strength should be approximately but not less than 0.5N and its colour not darker than pale yellow. Dissolve 35-40 g of potassium hydroxide pellets in 20 ml of water and mix the solution with 1 000 ml of 95 % (v/v) ethanol. Allow the solution to stand for several hours, preferably overnight, then decant or filter off the clear supernatant liquid. Keep the clarified solution in a dark place.

2) Diethyl ether.

3) Acetone.

4) Potassium hydroxide, approximately 0.5N aqueous solution.

5) Sodium hydroxide, 0.1N solution in ethanol⁸⁾. Dissolve 5–6 g of sodium hydroxide pellets in 10 ml of water and mix the solution with 1 000 ml of 95 % (v/v) ethanol. Allow the solution to stand for several hours, preferably overnight, then decant or filter off the clear supernatant liquid. Keep the filtered solution in a dark place.

- 6) *Hydrochloric acid*, d = 1.18.
- 7) $Ethanol^{(8)}$, 95 % (v/v).

8) Phenolphthalein indicator, 1.0 % solution in 95 % (v/v) ethanol⁸⁾.

C.3 Procedure

C.3.1 Determination of oil content. Weigh to the nearest 0.1 g, about 18 g of the material into a previously tared centrifuge tube. Add 15 ml of the diethyl ether, mix thoroughly with a stirring rod; wash the stirring rod with diethyl ether to make a total volume of 75 ml. Place the centrifuge tube in the centrifuge and whirl at a moderate rate until all the pigment is well settled. Decant the supernatant liquid into the tared flask. Repeat the extraction three times more using a similar amount of diethyl ether for each extraction. Decant the supernatant liquid into the flask after each extraction. When the extraction procedure is complete, fit the flask with a condenser and distil off the diethyl ether on a steam-bath. Carry out this process in a fume-cupboard. When distillation is complete continue heating the open flask, occasionally passing in a stream of air from a rubber blowing ball until only a trace of solvent remains.

Then place the flask in a ventilated oven at 80 $^{\circ}$ C for 1 hour, cool in a desiccator and weigh. Reheat at 80 $^{\circ}$ C for a further half hour, cool and reweigh. Continue with this procedure until the difference between two successive weighings is not more than 1 mg.

Calculate the percentage of oil in the material.

⁵⁾ BS 572, "Interchangeable conical ground glass joints".

⁶⁾ BS 5922, "Specification for glass condensers for laboratory use".

⁷) BS 2021, "Specification for separating and dropping funnels for laboratory use".

⁸⁾ Ethanol may be replaced by 66 OP industrial methylated spirit complying with BS 3591, "Industrial methylated spirits".

It should be noted that the use of industrial methylated spirit is governed by the Methylated Spirits Regulations, 1983 (S.I. 1983/252).

C.3.2 Determination of whiting content. Dry the residue in the centrifuge tube by placing the tube and residue on a steam-bath and heating until no smell of diethyl ether can be detected. Then transfer the tube and its contents to a ventilated oven and heat at 105 °C for 1 hour. Cool in a desiccator and weigh. Reheat at 105 °C for a further half hour, cool and reweigh. Continue heating at 105 °C until the difference between two successive weighings is not more than 1 mg. Calculate the percentage of whiting in the material.

C.3.3 Determination of unsaponifiable matter on extracted oil

Weigh to the nearest milligramme, 2.0 - 2.5 g of the extracted oil into a 250 ml flask or use the flask and oil from C.3.1, and add 25 ml of the alcoholic potassium hydroxide solution. Attach the flask to a reflux condenser and heat on a boiling water bath for 1 hour, swirling at frequent intervals to ensure complete saponification.

Remove the flask from the bath, detach the condenser and transfer the contents of the flask to a 250 ml separating funnel. Wash the flask with 50 ml of water and add the washings to the solution in the funnel. Then wash the flask with 50 ml of diethyl ether and add this cautiously to the solution in the funnel. Stopper the funnel, invert and ventilate by opening the tap, close the tap, shake vigorously, ventilate and then allow the funnel to stand until the two layers of liquid separate and clarify. Run off the aqueous alcoholic layer into the flask used for the saponification.

Pour the ethereal layer from the top of the funnel into a second 250 ml separating funnel containing 20 ml of water. Extract the aqueous alcoholic soap solution twice more, each time with 50 ml of diethyl ether in the same manner and combine the three extracts in the second funnel. If the extracts contain solid suspended matter, pass them through a small, dry, fat-free filter into the second separating funnel, washing the filter subsequently with warm diethyl ether and adding to the main diethyl ether extract.

Rotate the extracts gently in the second funnel, without violent shaking, with the 20 ml of water and, after allowing to separate, run off the water.

If the presence of metallic soap in the extracted oil is known or suspected, the second separating funnel should be empty instead of containing 20 ml of water.

In this case add 5 drops of hydrochloric acid d = 1.18, and shake vigorously. Wash the combined extracts successively with two quantities of 20 ml of water, employing vigorous shaking on each occasion.

After one or other of these preliminary treatments, wash the ethereal solution twice with 20 ml of water, shaking vigorously on each occasion. Then successively wash with 20 ml of the 0.5N aqueous potassium hydroxide solution, 20 ml of water, 20 ml of the 0.5N aqueous potassium hydroxide solution, 20 ml of water and again with 20 ml of the 0.5N aqueous potassium hydroxide solution, and at least twice more with 20 ml of water. Continue washing with water until the wash water no longer turns pink on addition of phenolphthalein indicator.

Transfer the ethereal solution to a previously tared flask and evaporate to small bulk. Add 2–3 ml of acetone and completely remove the solvent from the flask, for example by means of a gentle current of air, the flask being almost entirely immersed, held obliquely and rotated in a boiling-water bath. Dry the flask and contents to constant weight⁹⁾ at a temperature not exceeding 80 °C.

Dissolve the contents in 10 ml of freshly boiled and neutralized 95 % ethanol and titrate with the 0.1N alcoholic sodium hydroxide solution, using phenolphthalein indicator.

The titration should not exceed 0.1 ml. If it does, reject the test and repeat the determination from the beginning.

If there is any reason to suspect the incomplete separation of saponifiable matter, subject the weighed residue to re-saponification, re-extraction and washing, under the conditions specified in the method. If, by the treatment, the amount of unsaponifiable matter differs from that first obtained in the first determination, within the limits of manipulative error¹⁰⁾ reject the test and repeat the determination from the beginning.

Calculation. Unsaponifiable matter, per cent by weight = $\frac{100W_1}{W}$

where W_1 = weight of unsaponifiable residue (g)

and W = weight of extracted oil (g).

⁹⁾ In certain unusual cases, the unsaponifiable matter appears to give a continuous loss during drying, owing to the presence of some matter difficult to volatilize, as, for example, residual solvent fractions. In such instances transfer the washed ethereal extract to a flask containing about 2 g of neutral oil, such as ground nut oil, previously brought to constant weight at 80 °C, and then proceed as in an ordinary determination. Under these conditions the neutral oil serves to minimize any loss. ¹⁰⁾ The limits of experimental error in terms of weight will naturally vary with the amount of unsaponifiable matter in the sample.

Appendix D Method for the determination of paintability

D.1 Apparatus

The following apparatus is required:

1) Putty knife.

2) Wood moulding. Length approximately 250 mm with a 12.5 mm \times 12.5 mm rebate, made from a piece of softwood which is free from knots.

D.2 Procedure

Apply a coat of white priming paint complying with BS 5358^{11} to the wood moulding and allow to dry for 2 days. Then fill the moulding rebate with the putty and smooth off with one stroke of the putty knife to a clean surface so that a cross section of the putty presents a triangular area.

After allowing the putty to dry for 7 days at a temperature of 20 ± 2 °C and a relative humidity of 65 ± 5 %, coat the surface of the putty with a priming paint complying with BS 5358^{11} , using a brush, and allow to dry for 24 h at the same temperature and relative humidity. During the coating operation ensure that there is no lifting or disturbance of the putty surface.

After the 24 h drying period, determine whether the undercoat is surface dry by carrying out the test described in BS $3900 \cdot C2^{12}$ and also whether there is any discoloration, wrinkling and grittiness or other defect of the paint film.

Appendix E Method for the determination of consistency (50 kg (1 cwt) kegs only)

E.1 Principle

The results obtained by submitting the putty to the penetration test will enable an assessment to be made of the property usually referred to as the consistency of the putty.

The penetration is the distance in millimetres which a loaded 38 mm ($1\frac{1}{2}$ in) ball mounted on a shaft penetrates vertically into the keg of putty under standard conditions of loading, time and temperature.

E.2 Apparatus

The following apparatus is required:

1) Penetration device. A polished shaft of rust resisting steel 635 mm (25 in) long and 8 mm $(^{5}/_{16}$ in) diameter terminating at one end in a 38 mm (1½ in) diameter monel metal ball and at the other in a circular plate of aluminium alloy approximately 150 mm (6 in) in diameter and

approximately 5 mm $({}^{3}\!/_{16}$ in) thick on which weights can be placed (see Figure 2), the weights being adjustable to give superimposed loading of 2 500 g to 4 000 g in steps of 500 g.

2) *Penetrometer*. A penetrometer apparatus which will allow the penetration device to move without appreciable friction and which is fitted with a device capable of indicating the depth of penetration in mm. The sides of the guide should be vertical. A suitable hole is provided in the baseboard through which the surface of the putty may be observed when lowering the ball (see Figure 2 for a suitable apparatus).

3) Constant temperature enclosure. An enclosure capable of accommodating 1 cwt kegs of putty and regulated at a temperature of 20 \pm 1 °C unless otherwise agreed.

4) *Timing device*. An audible seconds counter is recommended.

E.3 Preparation of specimen

Immediately prior to carrying out the determination, place the keg of unopened putty in the constant temperature enclosure¹³⁾ for one week or until the putty has attained the agreed test temperature.

 $^{^{11)}\,\}mathrm{BS}$ 5358, Specification for low-lead solvent-thinned priming paint for woodwork.

¹²⁾ BS 3900, "Methods of test for paints", Part C2, "Surface-drying test (ballotini method)".

¹³⁾ For convenience in routine testing a correction for temperature can be determined by the manufacturer for his particular formulation. Where a constant temperature enclosure is not available, the putty can be allowed to cool from a higher temperature until the temperature indicated by a thermometer midway between the side of the keg and its centre, inserted to a depth of six inches reaches the test temperature.

Usually only one keg out of a mixer load need be tested and the contents of that keg should be incorporated in a subsequent batch

E.4 Procedure

Examine the penetrometer and establish that the penetration device falls freely.

Then after removing the lid from the keg and the paper from the surface of putty, place the penetrometer with the base board resting on the rim of the keg with the penetrometer device raised above the putty surface. Load the device by placing weights on the top plate and lower slowly until the ball just makes contact with the surface of the putty. Note the scale reading. Release the penetration device, read and record the depth of penetration in the specified time, using the specified superimposed load. Make four determinations, each midway between the centre and the side of the keg and equally spaced around the keg. Clean the ball between each determination.

NOTE When withdrawing the penetration device care must be taken not to introduce oil or putty into the bearings of the guide. If the difference between any individual reading and the mean is greater than 50 mm the test should be repeated with a fresh keg.

E.5 Reporting

Record the values of the superimposed load, time and penetration depths. Average the penetration depth figures obtained and report whether the average falls within 20 % of the specified penetration depth figure.

Appendix F Method for the determination of skins and coarse particles

NOTE This test should be carried out within 48 hours of taking the sample otherwise the results may be inaccurate. **F.1 Apparatus**

The following apparatus is required:

- 1) Sieve. Nominal aperture 425 μ m (36 mesh) complying with BS 410¹⁴).
- 2) Sieve. Nominal aperture 63 µm (240 mesh) complying with BS 410.
- 3) Camel-hair brush.

F.2 Reagent

White spirit, complying with BS 245^{15} .

F.3 Procedure

Weigh, to the nearest 0.01 g, 50 g of the material and mix with 100 ml of the white spirit. Pour the mixture through the 425 μ m mesh sieve and then through the 63 μ m mesh sieve. Wash the residue on the 425 μ m mesh sieve with the white spirit (not under pressure) and gently brush with the camel-hair brush until the washings are clean. Pour the washings through the 63 μ m mesh sieve and wash the residue on this sieve in the same way as used previously for the 20 μ m mesh sieve. Dry the residues on the two sieves for 1 hour at 100 ± 2 °C, cool and weigh.

¹⁴⁾ BS 410, "Specification for test sieves".

¹⁵⁾ BS 245, "Specification for mineral solvents (white spirit and related hydrocarbon solvents) for paints and other purposes".









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