BS 3262-1: 1989

Incorporating Amendment No. 1

# Hot-applied thermoplastic road marking materials—

Part 1: Specification for constituent materials and mixtures

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# Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Road Engineering Standards Policy Committee (RDB/-) to Technical Committee RDB/25, upon which the following bodies were represented

British Resin Manufacturers' Association

Chemical Industries Association

County Surveyors' Society

Department of Transport (Highways)

Department of Transport (Transport and Road Research Laboratory)

Glass Manufacturers' Federation

Institution of Civil Engineers

Institution of Highways and Transportation

Oil and Colour Chemists' Association

Road Markings Manufacturers' and Contractors' Association

Society of Chemical Industry

This British Standard, having been prepared under the direction of the Road Engineering Standards Policy Committee, was published under the authority of the Board of BSI and comes into effect on 29 September 1989

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### **Foreword**

This British Standard has been prepared under the direction of the Road Engineering Standards Policy Committee. It is published in three Parts:

- Part 1: Specification for constituent materials and mixtures;
- Part 2: Specification for road performance;
- Part 3: Specification for application of material to road surfaces.

This Part of BS 3262 replaces BS 3262-1:1987 which is withdrawn.

There have been many changes in materials and methods of laying since the first issue of this standard in 1960 and the basis of the standard has been changed to include performance requirements. Part 1 specifies the composition and quality control requirements of mixtures. Part 2 specifies the performance requirements for type approval of the materials when they have been applied to the road surface. Part 3 specifies the requirements for the application of material to the road surfaces.

This edition introduces technical changes to bring the standard up-to-date but it does not reflect a full review of the standard, which will be undertaken in due course.

In this new edition, requirements for black thermoplastic material are now included and synthetic resin binders are no longer limited to the hydrocarbon type.

It was previously thought that the measurement of the luminance coupled with the heat stability test would give a sufficient guarantee of the whiteness of a marking. This is not necessarily the case as the luminance factor is a measure of brightness and therefore work is now in hand to develop an ultra-violet light ageing test and a yellowness index test for use in determining the whiteness of the marking.

The 1976 edition of the standard indicated that the skid resistance of a material which has been laid on the road surface would tend to increase once the weathering of any resin film on the surface of the marking had occurred. Experience at a road trial site has shown that this is not necessarily the case and a test is being developed which will give an accurate indication of the change in skid resistance of a material with the passage of time and traffic. A minimum skid resistance value based on laboratory tests has been included in the standard.

A British Standard specification for solid glass beads has been published as BS 6088 and glass beads used in road marking materials are now required to comply with that standard.

The committee is considering the matter of the repeatability and reproducibility of the test methods contained in this standard but at present there is insufficient data on which to base limits.

It has been assumed in the drafting of this British Standard that the execution of its provisions is entrusted to appropriately qualified and experienced people.

Purchasers ordering to this standard are advised to specify in their purchasing contract that the supplier operate a quality system in compliance with the appropriate Parts of BS 5750 to assure themselves that the products claimed to comply with this Part of BS 3262 consistently achieve the required levels of quality.

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This standard was confirmed in November 1995 on publication of amendment No. 1. The changes incorporated did not reflect a full review of the standard, but were those felt necessary to maintain its currency for the foreseeable future pending its withdrawal and supersession by European Standards currently in course of preparation.

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 to iv, pages 1 to 14, 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.

iv blank

#### 1 Scope

This Part of BS 3262 specifies the requirements for white, yellow and black thermoplastic road marking materials and their constituents that are melted and applied hot to road surfaces, using screeded or sprayed application, as thin superimposed layers for centre lines, edge lines, pedestrian crossing stripes and the like. It does not apply to thermoplastic materials intended to be inset into the road surface.

The marking material consists of an aggregate which is light coloured except for black thermoplastic material, pigment and extender, bound together with a thermoplastic resin, plasticized as necessary. Provision is made for reflectorization using solid glass beads of a suitable grade complying with BS 6088 to improve the visibility of road markings.

The information to be given and requested with the invitation to tender is listed in appendix A.

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

#### 2 Definitions

For the purposes of this Part of BS 3262 the following definitions apply.

#### 2.1 Aggregates

#### 2.1.1

#### silica sand

a naturally occurring sand that consists of grains of substantially pure silica

### 2.1.2 calcite

a naturally occurring form of crystalline calcium carbonate

#### 2.1.3

#### quartz

a naturally occurring form of crystalline silica

#### 2.1.4

#### calcined flint

a prepared material made by heating pure flints to a sufficiently high temperature for change to the crystalline state to occur with an accompanying change in colour from black to white

#### 2.2

#### pigment

a fine powder added primarily to impart colour to the mixture

#### 2.3

#### extender

powder added to assist the dispersion of the pigment and impart body to the mixture

#### 2.4 Binding materials

#### 2.4.1

#### binder

the thermoplastic resinous material (together with any included oils or other thermoplastics) which provides adhesion to the road surface and cohesion between the other components (i.e. filler, pigment, aggregate and solid glass beads where specified)

#### 2.4.2

#### natural resin

a member of the group of glassy, amorphous, organic solids that are secreted by certain plants and insects and are insoluble in water but soluble in many organic solvents

#### 2.4.3

#### synthetic resin

a synthetic product resembling in some ways natural resin

#### 2.4.4

#### rosin

a specific kind of natural resin originating together with other organic liquids in pine trees; there are two forms:

- a) **gum rosin**. The natural resin which is obtained from the oleo-resin collected from living traces
- b) **wood rosin**. The natural resin which is obtained from the oleo-resin contained in dead wood such as stumps.

#### 2.5

#### reflectorization

the use of solid glass beads complying with BS 6088 in road marking materials

#### 2.6

#### luminance factor

the ratio of the luminance of a non-luminous body, under specified conditions of illumination and observation, to the luminance of a perfect diffuser receiving the same illumination

#### 2.7

#### maximum application temperature

the maximum temperature at which the material can be maintained for at least 6 h in an open-topped melting-machine or open-topped application-machine with constant agitation by an efficient stirring device, without serious degradation or discoloration occurring

#### 2.8

#### maximum safe heating temperature

the temperature above which the material is not to be heated at any time

#### 3 Aggregate

For white or yellow thermoplastic, the aggregate shall consist of light-coloured silica sand, calcite, quartz or calcined flint. For black thermoplastic, calcined bauxite or other dark coloured aggregate may be used as all or part of the aggregate.

#### 4 Pigment

White pigment material shall either consist of titanium dioxide type A (anatase) or type R (rutile) complying with BS 1851.

The lead chromate content of the road marking material shall not exceed 5 % by mass.

For black material, carbon black or a similar suitable pigment shall be used and the pigment may be predispersed.

NOTE There is no requirement for the composition of yellow pigment material. A suitable yellow pigment should be substituted for all or part of the titanium dioxide.

#### 5 Binder

The binder shall comprise plasticized synthetic resin, plasticized natural resins or rosins.

#### 6 Reflectorization

NOTE Reflectorization of products with improved wet night visibility may necessitate the use of large diameter glass beads in which case the gradation requirements of BS 6088 do not apply. For products where improved wet night visibility is required, glass beads of size 1 mm to 5 mm but otherwise complying with the requirements of BS 6088 may be specified and substituted for part or all of the incorporated and surface applied glass beads.

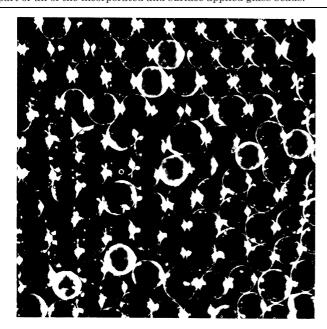
- **6.1** Solid glass beads to be incorporated in marking materials prior to application to road surfaces shall comply with the class A requirements of BS 6088.
- **6.2** Solid glass beads for additional surface reflectorization shall comply with the class B requirements of BS 6088.

NOTE  $\,$  Figure 1 illustrates the roundness requirement for class A beads specified in BS 6088.

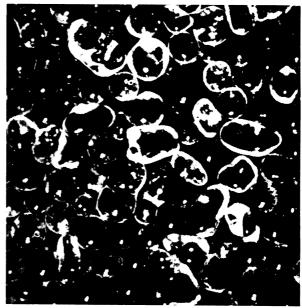
#### 7 Composition of marking material

NOTE For products where the nominal size of glass beads has been increased for the improvement of wet night visibility, the requirements for gradation do not apply.

- 7.1 When samples of the marking material taken in accordance with appendix B are tested in accordance with appendices C and D, the proportions of the constituents of the mixture obtained by the analysis shall be in accordance with Table 1.
- **7.2** When tested in accordance with appendix D, the grading of the aggregate, pigment, extender and, where specified, solid glass beads in the marking material, sampled in accordance with appendix B, shall be as follows:
  - a) 100 % by mass passes a 2.80 mm BS 410 test sieve;
  - b) 65 % to 95 % by mass passes a 600  $\mu m$  BS 410 test sieve.



(a) Good sample. Nearly all reasonably clear and spherical. Complies with roundness requirements for class A of BS 6088.



(b) Poor sample. Approximately 70 % reasonably clear and spherical, i.e. lower permissible limit for roundness of class A beads in BS 6088.

Figure 1 — Solid glass beads

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Table 1 — Proportions of constituents of marking material (see note)

. , ,			
Constituent(s)	Percentage by mass of total mixture		
	%		
Binder (resin and oil)	$20 \pm 2$		
Solid glass beads <sup>a</sup>	20 min.b		
Aggregate together with pigment, extender and solid glass beads <sup>a</sup>	$80 \pm 2$		

NOTE Black material is to be non-reflectorized and the binder is to be  $19 \pm 3$  % by mass of the total mixture.

#### 8 Properties of marking material

NOTE Whilst it is common for yellow materials used for pavement marking to be an approximate match to colour no. 353 Lemon of BS 381C:1980, the Department of Transport permits the use of other yellows (e.g. colour nos. 310 Primrose and 355 Deep Cream) in certain environmentally sensitive areas.

#### 8.1 Softening point

The softening point of the thermoplastic material, when measured in accordance with appendix E on samples taken and prepared in accordance with appendix B, shall be at a temperature of not less than 65 °C.

#### 8.2 Luminance

NOTE The measurement of the luminance factor on site may be acceptable for normal purposes but is deprecated for use as a reference method. The surface texture of the markings and irregularity in the substrate can affect the results; the rougher the surface, the lower the results obtained.

It is, however, permissible to take measurements of the luminance factor on the underside of samples, of the dimensions given in appendix F, which have been cast directly from material from the outlet of the melter or the laying apparatus. The method of casting and test procedure should be as described in appendix F.

#### 8.2.1 White material

**8.2.1.1** White marking material sampled "as delivered". When tested in accordance with appendix F the luminance factor of white marking material, sampled "as delivered" in accordance with appendix B, shall be not less than 70.

**8.2.1.2** White marking material re-melted for use. When tested in accordance with appendix F the luminance factor of white marking material, sampled after re-melting for use in accordance with appendix B shall be not less than 65.

### 9 Containers<sup>1)</sup>

The thermoplastic road marking materials shall be kept in containers made of material which does not contaminate the contents, and which protects the

#### 8.2.2 Yellow material

**8.2.2.1** Yellow marking material sampled "as delivered". When tested in accordance with appendix F the luminance factor of yellow marking material sampled "as delivered" in accordance with appendix B, shall be not less than 50.

**8.2.2.2** *Yellow marking material re-melted for use.* When tested in accordance with appendix F the luminance factor of yellow marking material sampled after re-melting for use in accordance with appendix B, shall be not less than 45.

**8.2.3** Black marking material re-melted for use. When tested in accordance with appendix F the luminance factor of black marking material, sampled after re-melting for use in accordance with appendix B, shall be not greater than 10.

#### 8.3 Heat stability

**8.3.1** White marking material. When tested in accordance with appendix G the luminance factor of white marking material sampled "as delivered" or after re-melting for use in accordance with appendix B, shall be not less than 65.

**8.3.2** *Yellow marking material.* When tested in accordance with appendix G the luminance factor of yellow marking material, sampled "as delivered" or after re-melting for use in accordance with appendix B, shall be not less than 45.

#### 8.4 Flow resistance

When tested in accordance with appendix H the flow resistance, measured as the mean slump, of white or yellow marking material, sampled "as delivered" or after re-melting for use in accordance with appendix B, shall be not more than 25 %.

#### 8.5 Skid resistance

When tested in accordance with appendix J the skid resistance of white or yellow marking material sampled "as delivered" or after re-melting for use in accordance with appendix B shall be not less than 45 and for black material it shall be not less than 55.

NOTE The skid resistance of a marking material is not a fixed property for any given formulation. For a new marking it may vary considerably according to the temperature at which it is laid and it will also vary throughout its life according to conditions such as traffic and weather.

If extremely high skid resistance is an essential requirement, special aggregates such as calcined bauxite or fused alumina may be necessary in the formulation and this should be subject to special prior arrangements between the purchaser and the supplier.

contents from contamination. Each container shall contain not less than 20 kg and not more than 110 kg of material.

a Where specified.

<sup>&</sup>lt;sup>b</sup> At least 20 % by mass is to be maintained even in the case of material to which solid glass beads are to be surface applied by pressure or by gravity (see note 2 to **D.4.3**)

#### 10 Marking

Each container shall be clearly and indelibly marked with the following information.

- a) the name, trade mark or other means of identification of the manufacturer;
- b) batch number;
- c) date of manufacture;

- d) the number and date of this British Standard, i.e. BS 3262-1:1989<sup>2)</sup>;
- e) whether reflectorized;
- f) colour (white, yellow or black);
- g) chemical description and type of resin;
- h) maximum application temperature and maximum safe heating temperature;
- i) the relative density;
- j) if applicable, the class of its contents (see BS 3262-2);
- k) if applicable, a warning about the use of lead pigment (see Figure 3, appendix K).

 $<sup>^{1)}\,\</sup>mathrm{See}$  BS 1133.

<sup>&</sup>lt;sup>2)</sup> Marking BS 3262-1:1989 on or in relation to a product represents a manufacturer's declaration of conformity, i.e. a claim by or on behalf of the manufacturer that the product meets the requirements of the standard. The accuracy of the claim is therefore solely the responsibility of the person making the claim. Such a declaration is not to be confused with third party certification of conformity, which may also be desirable.

# Appendix A Guidance on information to be given and requested with invitation to tender

The following information should be given by the purchaser with the invitation to tender:

- a) the colour of the marking material (i.e. white, yellow or black);
- b) the chemical description and type of resin (see clause 5);
- c) whether the material is required to be reflectorized (see clause **6**);
- d) whether additional surface reflectorization is required (see **6.2**);
- e) whether a tack coat is to be used (see clause 3 of BS 3262-3:1989);
- f) the method by which the material is to be applied to the road surface (i.e. screed or spray);
- g) whether the material is to be supplied only or supplied and laid in accordance with BS 3262-3;
- h) details of any samples of the material required for testing.

The following information should be requested from the manufacturer by the purchaser:

- 1) maximum application temperature and maximum safe heating temperature;
- 2) the temperature range for apparatus and method of laying to be used.

# Appendix B Sampling and preparation of specimens

#### **B.1** General

This appendix describes the procedure for preparing specimens used in the methods of test described in appendices C to K inclusive.

For the purposes of carrying out the tests given in this standard it is essential that adequate and representative samples be taken in accordance with this appendix.

Marking material shall be sampled in accordance with **B.2.1** if samples are to be taken at the manufacturer's plant or are to be taken of the "as delivered" materials, or with **B.2.2** if samples are to be taken of the re-melted mixture prepared for use.

NOTE 1 Sampling from a preheater is the preferred source for samples as the greater number of bags in the preheater compensates for lack of uniformity in the mix.

NOTE 2 Where the manufacturer recommends that the bag in which the material is contained does not need to be removed no allowance should be made for the presence or absence of the bag in the determination of the constituents of the mix.

#### **B.2 Sampling**

#### B.2.1 Sampling before re-melting for use

**B.2.1.1** *Powdered material.* Select three bags at random of the material, each bag bearing the same batch number. Divide each bag separately using a riffle box having a maximum aperture of 50 mm to obtain three samples each having a mass of approximately 4 kg. Combine the three 4 kg samples and clearly label the combined sample with the date and batch number from the bags together with the grade name, source of sample and any other relevant information. Keep half the sample in case of dispute.

B.2.1.2 Thermoplastic compound. Select at random three containers of the thermoplastic compound. Break up the material from all three containers and discard any pieces that by visual examination are not uniform in texture and colour. Take a portion of not less than 4 kg from the material near the centre of each of the three containers and combine these portions in a clean container clearly labelled with the relevant details, e.g. supplier, batch number, type and date. Do not apply heat at any stage of the sampling process.

### B.2.2 Sampling of re-melted mixture prepared for use

Take three portions, each having a mass of not less than 4 kg, from the outlet of a preheater or laying apparatus, discarding the first and last 5 % of the charge. Combine the three portions in a clean container clearly labelled with the contractor's name, type of material, site and date. Do not apply heat at any stage of the sampling process once the material has issued from the melting or laying apparatus. Record the temperature of the material at the time of sampling. Keep half the sample in case of dispute.

 ${
m NOTE}\ {
m Because}$  black material may be a two part system, it is essential that samples be taken from the pre-heater.

#### **B.2.3** Certificate of sampling

**B.2.3.1** *General*. A record shall be prepared at the time of sampling. The information shall be recorded in the form shown in **B.2.3.2** and a copy shall accompany each laboratory sample.

**B.2.3.2** *Typical certificate of sampling.* The following information should be given on the certificate:

- a) job or contract;
- b) name of contractors;
- c) location where sample was taken (where possible):
- d) contractor's vehicle number;
- e) required specification;

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- f) method of sampling (i.e. **B.2.1.1**, **B.2.1.2** or **B.2.2**);
- g) date when sample was taken;
- h) data from container:
  - 1) identity of manufacturer;
  - 2) batch number;
  - 3) whether reflectorized;
  - 4) colour;
  - 5) chemical description and type of resin;
  - 6) maximum application and maximum safe heating temperatures;
- i) where the material was riffled (**B.2.1.1**) or broken up (**B.2.1.2**), where applicable (e.g. on site or in the laboratory);
- j) type of container used and approximate weight of sample taken;
- k) where the remainder of the materials is being kept;
- l) name of sampler (block capitals);
- m) signature of sampler;
- n) signature of contractor's representative.

#### **B.3** Consolidation of sample

#### **B.3.1** Apparatus

**B.3.1.1** Clean heat resistant glass or metal containers.

NOTE  $\,$  Suitable metal containers are new 2% litre lever lid paint tins.

**B.3.1.2** *Oven*, of a capacity to hold at least two of the above containers. The oven shall be capable of heating to 200 °C within two hours the two containers plus at least 1 kg of material and shall also be capable of holding a preset temperature to an accuracy of  $\pm$  5 °C.

NOTE A short temperature recovery time is beneficial.

**B.3.1.3** *Electronic thermometer*, with a fast response immersion probe accurate to  $\pm$  1 °C. Alternatively a mercury-in-glass thermometer of the same accuracy may be used.

#### B.3.2 Procedure for powdered material

**B.3.2.1** If the softening point of the material is known from past results proceed to **B.3.2.3**.

NOTE If subsequent determination of the softening point (i.e. after preparing in accordance with  $\bf B.4$ ) reveals that the value is more than 10 °C from the value taken then the procedure in  $\bf B.3.2.3$  should be repeated with a fresh 6 kg sample using the revised softening point.

**B.3.2.2** If the softening point of the material is not known take a sample of approximately 1 kg from the combined sample (see **B.2.1.1**) and determine the softening point in accordance with appendix E. When preparing specimens heat until homogeneous and do not exceed the manufacturer's safe heating temperature.

NOTE If subsequent determination of the softening point (i.e. after preparing in accordance with  ${\bf B.4}$ ) reveals that the value is more than 10 °C from the value found in  ${\bf B.3.2.2}$ , then the procedure in  ${\bf B.3.2.3}$  should be repeated with a fresh 6 kg sample, using the revised softening point value.

**B.3.2.3** Preheat the oven to a temperature of  $(S+70)\pm 10$  °C, where S is the softening point, and place the combined sample from **B.2.1.1**, in one or two containers, into the oven. After 20 min and subsequently at 10 min to 15 min intervals remove the sample from the oven, quickly stir thoroughly with a palette knife, measure the temperature, and replace the sample in the oven. When the temperature of the sample reaches  $(S+50)\pm 5$  °C, pour the material on to a suitable release surface. The time from commencement of heating to pouring shall not exceed two hours.

**B.3.2.4** If a sample is not homogeneous at  $(S+50)\pm 5$  °C raise the oven temperature to  $(S+120)\pm 10$  °C. Continue to remove the sample at 10 min to 15 min intervals and stir and measure temperature as in **B.3.2.3**. When the sample is homogeneous pour it on to a suitable release surface.

**B.3.2.5** Report the temperature at pouring and the time from first commencing heating to pouring.

#### $B.3.3\ Procedure\ for\ block\ or\ re-melted\ material$

The samples obtained in **B.2.1.2** and **B.2.2** need no consolidation and further preparation shall be in accordance with **B.4**.

#### **B.4 Preparation of specimens**

#### **B.4.1** Apparatus

The apparatus shall be as specified in **B.3.1** except that the clean heat resistant containers shall be of sufficient size to prepare the size of specimens required.

#### B.4.2 Procedure

**B.4.2.1** Take the block or re-melted sample from **B.2** or **B.3** and, without the application of heat, prepare a subsample by breaking a sufficient number of pieces of material, each weighing not more than 50 g, from various parts of the sample, allowing for a small excess for wastage.

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**B.4.2.2** Follow the procedure described in **B.3.2.3**, except that the time from commencement of heating to pouring shall not exceed one hour. Stir thoroughly immediately before pouring the appropriate specimens for testing.

NOTE The softening point should always be determined (see note to B.3.2.1 and B.3.2.2). An exception can be made when samples from the same source are being tested very frequently or when more than one sample has been taken from the same batch.

## Appendix C Determination of binder content

#### C.1 General

- **C.1.1** This appendix describes three methods for determining the binder content of thermoplastic road marking materials sampled in accordance with appendix B.
  - a) an extraction bottle method using dichloromethane (methylene chloride);
  - b) a hot extractor method using a suitable solvent;
  - c) an ignition method.
- C.1.2 Any of the methods listed in C.1.1 may be used except that in the case of dispute method C.1.1 c) shall be used for the binder content determination and method C.1.1 a) for the determination of glass bead content and grading.

NOTE Some binders contain polymers and these may be difficult to dissolve. Complete solution can usually be obtained by prolonged exposure to the specified solvents; in cases of difficulty the manufacturer of the product should be consulted.

#### C.2 Extraction bottle method

#### C.2.1 Apparatus

- **C.2.1.1** *Metal bottle*, having a capacity of between 600 mL and 2 000 mL with a wide mouth and suitable closures.
- **C.2.1.2** *Rotating machine*, which will rotate the bottle about its longitudinal axis at a speed of  $20 \pm 10$  r/min.
- **C.2.1.3** *Volumetric flasks*, of appropriate capacity, e.g. 250 mL, 500 mL and 1 000 mL.
- **C.2.1.4** Centrifuge or filtration apparatus. The centrifuge shall comply with BS 4402 and be capable of developing an acceleration a of  $25~000 \pm 100~\text{m/s}^2$  calculated in accordance with the following equation:

$$a = 1.097 n^2 r \times 10^{-5}$$

where

- n is the number of revolutions per minute;
- r is the radius (in mm) to the inside base of the tubes when rotating.

The tubes shall be closed with caps to prevent loss of solvent during centrifuging.

NOTE 1  $\,$  A typical centrifuge suitable for this method carries two or more buckets fitted with centrifuge tubes of between 50 mL and 100 mL capacity.

NOTE 2 It is strongly recommended that the speed of rotation should be verified regularly to ensure that the centrifuge maintains its performance at all times. The centrifuge should be maintained in accordance with appendix B of BS 4402:1969.

The filtration apparatus comprises a metal bottle as specified in C.2.1.1, a porous filter thimble, tubing and a 50 mL burette. The porous filter is of porcelain, alumina or similar material, 90 mm long by 20 mm diameter and of up to 4 µm pore diameter. The filter is closed by sealing in, to within 5 mm of the bottom, a length of metal tubing, approximately 300 mm long by 5 mm bore, that passes through a supporting ring of cork or metal that is mounted just inside the open end of the filter. A cement paste composed of copper oxide powder, prepared by the direct oxidation of copper wire, of about 425 µm particle size, and phosphoric acid is used to seal the joint. The joint is left to dry for a few hours. Alternatively, a rubber stopper may be used. Care has to be taken to ensure that the seal does not split the filter thimble.

**C.2.1.5** Recovery apparatus, comprising a water bath with an electric heater capable of maintaining boiling water in the bath throughout the recovery procedure, a flat-bottomed flask of 200 mL or 250 mL capacity, a vacuum gauge, a vacuum reservoir and a means of maintaining reduced pressure, e.g. a filter pump.

 ${\bf C.2.1.6}\ Balance$ , capable of weighing to within an accuracy of  $0.005\ {\bf g.}$ 

#### C.2.2 Solvent

Use dichloromethane (methylene chloride), complying with BS 1994.

WARNING. In appendix A of BS 1994:1953, the requirements for the condenser includes asbestos board. The use of asbestos products is controlled by the Asbestos Regulations 1969, the Control of Asbestos at Work Regulations 1987 and the approved Code of Practice relating to those Regulations.

The Regulations require that any dust liberated during the handling and working of these products should be maintained at a low level. Adequate methods exist to control levels of dust during such operations and these are detailed in the Control and Safety Guides issued by the Asbestos Research Council.

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#### C.2.3 Procedure

**C.2.3.1** Take a subsample of 100~g to 150~g as described in appendix B, break it into small pieces, and weigh to an accuracy of 0.05~% of the total mass taken.

**C.2.3.2** Insert the pieces into the bottle and add dichloro-methane, measured by means of volumetric flasks, to give a solution of the binder having a concentration of about 3 %.

**C.2.3.3** Close the bottle and rotate it on the rotary mixer for a minimum of 15 min, until a complete solution of the binder is obtained.

**C.2.3.4** Separate a portion of the binder solution from insoluble matter either by centrifuging for 20 min to 30 min in completely closed tubes, or by means of the filtration apparatus using a dry, binder free filter. Take care to minimize loss of solvent during this separation.

NOTE 1 If filtration is prolonged, owing to fine mineral matter being present in the binder solution, inaccurate results may be obtained and in this case centrifuging is essential.

NOTE 2 When the amount of binder in the sample is unknown, it is advisable to recover the binder from one aliquot portion of solution before proceeding with the duplicate recovery described in C.2.3.6 to C.2.3.10 to ensure compliance with the mass restrictions given in C.2.3.11.

C.2.3.5 Weigh two flat-bottomed flasks (see C.2.1.5) to the nearest 0.01 g.

**C.2.3.6** Measure from the burette a sufficient amount of the centrifuged or filtered solution into each flask to give a residue of 0.75 g to 1.25 g of binder after evaporation of the solvent.

NOTE An estimate of the volume of solution [aliquot portion, A (in mL)] required is given by the following equation:

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

where

*B* is the total volume of solvent (in mL);

*C* is the mass of sample (in g);

D is the estimated percentage of soluble binder in the specimen

**C.2.3.7** Remove the solvent from the binder solution by connecting each flask to the recovery apparatus. Immerse the flasks to approximately half their depth in the boiling water, and distil off the solvent at atmospheric pressure or at a reduced pressure of not less than 600 mbar<sup>3)</sup>. While the distillation is proceeding shake the flasks with a rotary motion so that the binder is deposited in a thin layer on the walls of the flasks. Do not allow pressure above atmospheric to develop in the flask during the evaporation of the solvent.

**C.2.3.8** At the stage that relatively rapid reduction in pressure occurs, usually accompanied by frothing, when the bulk of the solvent has been removed, reduce the pressure to 200 mbar in 1.5 min and maintain the pressure at 200 mbar for a further 3.5 min.

**C.2.3.9** Remove the flasks from the bath and admit air to the apparatus to increase the pressure to atmospheric. Wipe each flask dry and disconnect it, taking care to prevent the entry into the flasks of any water that may have collected where the rubber stopper joins the flask. Remove the last traces of solvent that remain in the flasks by a gentle current of clean, oil and water free air.

**C.2.3.10** Cool the flasks in a desiccator and weigh them to the nearest 0.01 g. If the quantity of soluble binder recovered in either flask lies outside the limits 0.75 g and 1.25 g, repeat the recovery with another portion of the solution having the volume suitably adjusted.

**C.2.3.11** If the difference between the duplicate recoveries is greater than 0.02 g reject these results and repeat the recovery of the binder in duplicate on further aliquot portions.

**C.2.3.12** Note the results of the duplicate determinations in which the mass of binder recovered lies within the required limits.

**C.2.3.13** When the aggregate is required for a grading test or for analysis, filter the whole of the solution remaining after determination of the binder content. Use the funnel method described in BS 598-2, but with a No. 40 Whatman filter paper or equivalent or using a sintered glass Buchner funnel (porosity No. 4) or a pressure filter.

#### C.2.4 Calculation of results

**C.2.4.1** Calculate the mean of the results obtained in **C.2.3.11**.

**C.2.4.2** Calculate the binder content C (% by mass) of the sample from the following equation:

$$C = \frac{100zV}{vm} \left(1 + \frac{z}{dv}\right)$$

where

m is the mass of sample (in g);

z is the average mass of binder recovered from the two aliquot portions (in g);

*V* is the total volume of solvent (in mL);

v is the volume of each aliquot portion (in mL);

d is the relative density of the binder.

 $<sup>^{3)}</sup>$  1 mbar = 0.1 kPa =  $10^2$  N/m $^2$ 

BS 3262-1:1989

NOTE The density of resin/oil binders containing between 20 % and 30 % of oil lies between 1.03 and 1.08, hence a figure of 1.05 may be assumed when the exact nature of the binder is not known

#### C.2.5 Test report

Report the binder content of the sample.

#### C.3 Hot extractor method

#### C.3.1 Apparatus

**C.3.1.1** Specimen container, cylindrical in shape and made from brass or copper gauze with an aperture size of between 1 mm and 2 mm. The diameter of the container shall be 44.5 mm and its depth shall be 44.5 mm and it shall be capable of being suspended by wire hooks from the cork of the conical flask (see **C.3.1.2**).

**C.3.1.2** *Conical flask*, made of heat resistant glass and of 750 mL capacity.

**C.3.1.3** *Graduated receiver*, of suitable size, complying with BS 756, and fitted with a suitable reflux condenser.

**C.3.1.4** *Heating arrangement,* comprising either a laboratory gas burner and tripod or other suitable form of electrical heating arrangement.

**C.3.1.5** *Drying oven*, ventilated and capable of maintaining temperatures of  $120 \pm 50$  °C.

**C.3.1.6** *Desiccator*, containing self-indicating silica gel.

**C.3.1.7** *Balance*, capable of weighing to an accuracy of 0.005 g.

#### C.3.2 Solvent

Use any solvent which dissolves the entire binder component.

#### C.3.3 Procedure

**C.3.3.1** Take a subsample of about 30 g as described in appendix B.

**C.3.3.2** Fit a filter paper<sup>4)</sup> into the cylindrical container to form a complete lining and dry the whole at a temperature of  $110 \pm 10$  °C. Cool the container in a desiccator and weigh.

**C.3.3.3** Carefully place the sample in the lined container and weigh the whole to the nearest 0.05 % of the mass taken,  $m_1$ . Carry out the weighing operations involving the dried filter paper as rapidly as possible to prevent undue absorption of moisture from the atmosphere.

**C.3.3.4** Assemble the apparatus with 200 mL of solvent in the flask and reflux the solvent until the liquid running from the basket is free from binder.

**C.3.3.5** Stop the refluxing and allow the apparatus to cool and drain. Remove the washed aggregate with its container and dry at a temperature of between 100 °C and 120 °C. Weigh the container at 30 min intervals during drying. Continue drying until the difference between successive weighings does not exceed 0.05 %. Note the final mass,  $m_2$ .

**C.3.3.6** Collect any fine material present in the solution at the end of the test by filtering the whole of the solution through a suitable filter paper<sup>5)</sup> or by centrifuging it. Weigh the insoluble matter,  $m_3$ .

**C.3.3.7** Determine the mass of water collected, *W*, by weighing the water collected in the reflex condenser (see **C.3.1.3**).

#### C.3.4 Calculation of results

Calculate the binder content C (% by mass) of the sample from the following equation:

$$C = 100 \times \frac{m_1 - (m_2 + W + m_3)}{m_1 - W}$$

where

 $m_1$  is the mass of sample before drying (in g);

 $m_2$  is the mass of recovered aggregate in cylindrical container (in g);

W is the mass of water collected (in g);

 $m_3$  is the mass of residue obtained on filtering or centrifuging the solution (in g).

#### C.3.5 Test report

Report the binder content of the sample.

#### C.4 Ignition method

#### C.4.1 Apparatus

**C.4.1.1** *Two crucibles*, made of porcelain or other suitable material, having a capacity of 30 mL to 50 mL.

**C.4.1.2** *Electric muffle furnace*, with a temperature range of up to 800 °C and capable of being controlled to within  $\pm 25$  °C.

NOTE  $\,$  A muffle furnace with an internal volume of 2.4~L and a maximum power rating of 1.5~kW is suitable.

**C.4.1.3** *Desiccator*, containing silica gel or other suitable drying agent.

**C.4.1.4** *Balance*, capable of weighing to an accuracy of 0.001 g.

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 $<sup>^{4)}</sup>$  A Whatman No. 1 filter paper or equivalent.

<sup>&</sup>lt;sup>5)</sup> A Whatman No. 5 filter paper or equivalent.

#### C.4.2 Procedure

**C.4.2.1** Take a subsample of material prepared in accordance with appendix B and cast a slab approximately 100 mm in diameter and 10 mm thick on a clean, flat silicone rubber mould.

**C.4.2.2** Allow the material to cool to room temperature.

**C.4.2.3** Break the material into small pieces and weigh to an accuracy of 0.01 g duplicate specimens, each of approximately 10 g, into a pre-weighed dry crucible

**C.4.2.4** Place the weighed specimens contained in the crucibles in the centre of the muffle furnace and ignite for a minimum of 1 h at a temperature of 500 °C to constant mass. Do not heat the specimens to a temperature in excess of 550 °C to avoid decomposition of any inorganic carbonates.

**C.4.2.5** Cool the specimens in a desiccator after ignition and re-weigh to an accuracy of 0.01 g.

#### C.4.3 Calculation and expression of results

Calculate the binder content from the loss in mass and express as a percentage of the original sample mass.

#### C.4.4 Test report

Report the binder content to one decimal place as the mean of the results of the duplicate samples. If however the difference between the results is greater than 0.3 % absolute repeat the binder content determination.

# Appendix D Determination of the grading of constituents and glass bead content

#### D.1 General

This appendix describes the methods for determining the grading of the constituents and the glass bead content of marking materials sampled in accordance with appendix B for the whole of the material obtained as described in C.2 (see C.2.3.1 and C.2.3.2).

#### D.2 Apparatus

2.80 mm,  $600~\mu m$  and  $425~\mu m$  test sieves complying with BS 410 shall be used.

#### D.3 Determination of grading of constituents

**D.3.1** Ignite the filter paper and thoroughly mix the ash with the main bulk of the aggregate.

**D.3.2** Carry out the grading test using 2.80 mm and 600  $\mu$ m test sieves in accordance with the method specified in BS 812-103. If the glass bead content is to be determined use a 425  $\mu$ m test sieve also.

**D.3.3** Report the mass passing each sieve as a percentage of the combined mass of the aggregate, pigment and extender and (where present) glass beads.

#### D.4 Determination of glass bead content

#### D.4.1 Apparatus

**D.4.1.1** *Metal tray,* of approximate dimensions 150 mm  $\times$  355 mm inclined at an angle of  $5 \pm 1^{\circ}$  to the horizontal.

**D.4.1.2** *Brush*, small and soft.

#### D.4.2 Method

Take all the material obtained in **D.3.2**, place increments of 5 g to 10 g on the upper end of the tray and gently brush the material until all the glass beads have been moved to the bottom of the tray.

#### D.4.3 Calculation and expression of results

Report the total mass of the round glass beads collected as a percentage of the mass of the original sample of thermoplastic material × 100/70.

NOTE 1 The accurate determination of the proportion of glass beads in the mixture can be complicated by the chemical composition and relative density of the materials in it. The method given above is the simplest and although rather tedious does give quite accurate results.

NOTE 2 The intention of this standard is that a minimum of 20 % glass beads complying with BS 6088 should be incorporated in the mix at the time of manufacture.

NOTE 3 An alternative method for determining glass bead content is to use a roundometer as described in BS 6088.

## Appendix E Determination of softening point

#### E.1 Procedure

Determine the softening point of two specimens of the material, obtained in accordance with appendix B, by the ring and ball method described in BS 2000-58 except that the material shall not be sieved through a 300  $\mu m$  sieve and shall be heated as described in appendix B.

#### E.2 Expression of results

Report the mean value of the results obtained to the nearest 1  $^{\circ}\mathrm{C}.$ 

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### Appendix F Determination of luminance factor

#### F.1 General

This appendix describes methods for determining the luminance factor of marking materials sampled in accordance with appendix B. The luminance factor shall be that for a CIE standard observer<sup>6)</sup> under CIE illuminant "C" at 45° to the surface with viewing at right-angles to the surface.

#### F.2 Apparatus<sup>7)</sup>

**F.2.1** General. The apparatus for comparison of materials under test with calibrated reference panels shall consist essentially of a light source arranged at an angle of 45° to the specimen and a photo-detector positioned to view the specimen at right angles.

**F.2.2** *Light source.* The light source shall be CIE standard source "C"<sup>6)</sup> with an international colour temperature of 6  $774 \pm 200$  °K representing average daylight viewing.

**F.2.3** Filtered photo-detector. The filter shall be CIE type "y"<sup>6</sup>).

NOTE This is a separate filter to that used for conversion of "A" to "C" source illumination.

#### F.3 Calibrated reference panels

#### F.3.1 General

Calibrate the apparatus used for measuring luminance factors using calibrated panels with "Y" luminance factors traceable to national standards.

#### F.3.2 White materials

For white materials, calibrate using a panel having a CIE "Y" luminance factor  $^{6)}$  in the range of 65 % to 95 %.

#### F.3.3 Yellow materials

For yellow materials calibrate using a panel yellow in colour approximating to colour no. 353 of BS 381C:1988 and having a CIE "Y" luminance factor within the range 50 % to 70 %.

#### F.4 Test procedure

**F.4.1** Take material prepared in accordance with appendix B and cast a slab approximately 100 mm in diameter at least 10 mm thick on a clean, flat silicone rubber mould.

NOTE For routine purposes, specimens may be cast on either a flat, smooth metal sheet having a permanent release coating of polytetra-fluoroethylene (PTFE) or a clean, flat, smooth metal sheet having a light coating of glycerol. However, the silicone rubber mould is to be used for referee purposes. If, after having been tested, the specimen is to be kept for further measurement, it should be returned to its mould and stored at low temperature, preferably in a refrigerator, in order to avoid distortion. Provided the cast face of the specimen is used for measurement, the presence of solid glass beads in the material is unlikely to affect its luminance.

**F.4.2** Allow the specimen to cool to room temperature and then remove it from the mould and immediately measure the reflectance value of the cast face and record the value obtained.

**F.4.3** Repeat the measurement on two different parts of the specimen and note the values obtained.

#### F.5 Expression of results

Calculate the mean of the three values obtained to the nearest unit and record the luminance factor of the specimen.

# Appendix G Determination of heat stability

#### G.1 General

This appendix describes a method for determining the heat stability of road marking materials sampled in accordance with appendix B.

#### G.2 Apparatus

**G.2.1** *Oil bath*, capable of maintaining temperatures of up to 200 °C to within  $\pm$  2 °C.

**G.2.2** *Beaker*, made of heat resistant glass with a capacity of 250 mL and having nominal dimensions 110 mm high and 65 mm diameter.

<sup>&</sup>lt;sup>6)</sup> As defined in the proceedings of the 8th session of the Commission Internationale de L'Éclairage (CIE), Paris 1931. Publications available from the National Illumination Committee of Great Britain, c/o The Library, Thorn Lighting Ltd., Great Cambridge Road, Enfield, Middlesex EN1 1UL.

<sup>&</sup>lt;sup>7)</sup> For information on the availability of suitable apparatus apply to Enquiries Section, BSI, Linford Wood, Milton Keynes MK14 6LE enclosing a stamped addressed envelope for reply.

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**G.2.3** *Paddle stirrer*, electrically driven and controlled to rotate at  $150 \pm 10$  r/min. The shaft of the stirrer is a 6.5 mm diameter rod of suitable length to fit the stirrer motor, fitted with a single blade paddle of  $40 \pm 0.5$  mm length,  $30 \pm 0.5$  mm depth and  $1.5 \pm 0.5$  mm thickness.

#### **G.3 Procedure**

- **G.3.1** Accurately weigh a specimen of the material prepared in accordance with appendix B of mass approximately 250 g and place it in the beaker.
- **G.3.2** Control the temperature of the oil bath to within  $\pm$  2 °C, at 200 °C or the maximum application temperature whichever is the lower.
- **G.3.3** Immerse the beaker in the heated oil bath so that the surface of the sample, when molten, will be below that of the oil.
- **G.3.4** When the sample is molten, lower the stirrer to within 15 mm of the base of the beaker and commence stirring.
- **G.3.5** Maintain the test conditions for 6 h after which remove the beaker from the oil bath and pour the contents on to a clean flat silicone rubber mould (but see note to **F.4.1**).
- G.3.6 When the material has cooled to room temperature remove it from the mould, invert it and measure the luminance factor in accordance with appendix F.

#### **G.4** Expression of results

Express the luminance factor to the nearest unit.

## Appendix H Determination of flow resistance

#### H.1 General

This appendix describes a method for determining the flow resistance of road marking materials sampled in accordance with appendix B.

#### **H.2 Procedure**

- **H.2.1** Cast two conical specimens of the material, prepared in accordance with appendix B, so that each has an angle of nominally  $60^{\circ}$  at its apex and a vertical height of  $100 \pm 5$  mm.
- **H.2.2** After cooling and settling for 24 h remove the specimen from the mould and place it point upwards on a flat level surface in a room or box maintained at a temperature of  $23 \pm 2$  °C for 48 h.

- **H.2.3** Measure and record the height of the cone nearest millimetre.
- **H.2.4** Measure and record the height of the cone following the 48 h conditioning period.

#### H.3 Expression of results

Calculate the decrease in height of the two specimens as a percentage and report the average of the two percentages to the nearest 1 % as the mean slump

### Appendix J Determination of skid resistance

#### J.1 General

This appendix describes a method for determining the skid resistance of marking materials sampled as described in appendix B.

#### J.2 Apparatus

- **J.2.1** *Steel sheet,* approximately 1.6 mm thick, at least 150 mm wide and 850 mm long.
- **J.2.2** *Screed box*, as shown in Figure 2. The gap at the base is  $3 \pm 0.1$  mm when used for thermoplastic materials intended for screed application and  $1.5 \pm 0.1$  mm for spray applied thermoplastic materials.
- **J.2.3** *Portable skid resistance tester*<sup>8)</sup>, manufactured under licence to the design of the Transport and Road Research Laboratory (TRRL). The calibration of the tester shall be checked at least once a year<sup>9)</sup>.

#### J.3 Procedure

- **J.3.1** From a sample of approximately 1 kg prepare two specimens in accordance with appendix B.
- **J.3.2** Pour the material into the screed box, with the screed box and steel sheet at a temperature of  $20 \pm 10$  °C, and immediately draw the box at a steady speed of  $37 \pm 12$  mm/s over the steel sheet to give a coating approximately 100 mm wide and at least 800 mm long.
- NOTE  $\;$  It is of great importance that the material is used at the temperature specified in appendix B and that there is no delay in the screeding operation.
- **J.3.3** Allow the screeded material to cool to room temperature and determine the skid resistance at three different parts of the screeded material, excluding 150 mm from either end, using the skid resistance tester in accordance with the instructions supplied with the instrument.

<sup>&</sup>lt;sup>8)</sup> For information on the availability of suitable apparatus apply to Enquiries Section, BSI, Linford Wood, Milton Keynes MK14 61E enclosing a stamped addressed envelope for reply.

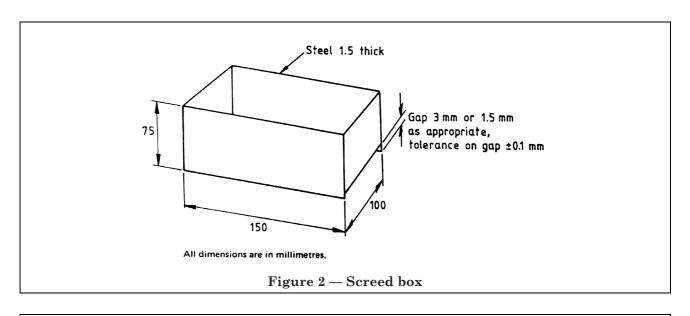
<sup>9)</sup> The calibration of the tester is carried out at The Test Centre, BSI, Maylands Avenue, Hemel Hempstead, Herts HP2 4SQ.

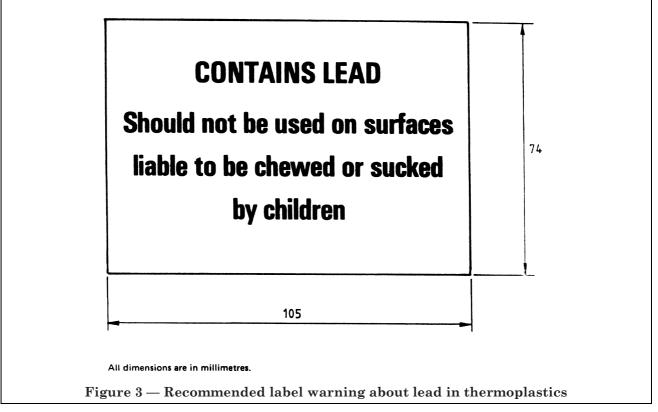
#### J.4 Expression of results

Report as the skid resistance of the material the mean results of the three readings for the two specimens to the nearest whole number.

# Appendix K Recommended label warning about lead in thermoplastics

If the thermoplastics material includes lead pigment the warning label shown in Figure 3 should be used on the container.





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### Publications referred to

BS 381C, Specification for colours for identification, coding and special purposes.

BS 410, Specification for test sieves.

BS 598, Sampling and examination of bituminous mixtures for roads and other paved areas.

BS 598-2, Methods for analytical testing.

BS 756, Specification for Dean and Stark apparatus.

BS 812, Testing aggregates.

BS 812-103, Methods for determination of particle size distribution.

BS 1133, Packaging code.

BS 1851, Titanium dioxide pigments.

BS 1994, Dichloromethane (methylene chloride).

BS 2000, Methods of test for petroleum and its products.

BS 2000-58, Softening point of bitumen (ring and ball).

BS 3262, Hot applied thermoplastic road marking materials.

BS 3262-2, Specification for road performance.

BS 3262-3,  $Specification\ for\ application\ of\ material\ to\ road\ surfaces.$ 

BS 4402, Specification for safety requirements for laboratory centrifuges.

BS 5750, Quality systems.

BS 6088, Specification for solid glass beads for use with road marking compounds and for other industrial uses.

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