BRITISH STANDARD

CONFIRMED AUGUST 1985 BS 6088:1981

(Reprinted, incorporating Amendments Nos. 1 to 3 inclusive)

Specification for

Solid glass beads for use with road marking compounds and for other industrial uses

UDC 625.094:628.978.73:666.27



Cooperating organizations

The Road Engineering Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following:

Asphalt and Coated Macadam Association
Association of Consulting Engineers
British Quarrying and Slag Federation
British Tar Industry Association
Cement and Concrete Association
Concrete Society Limited
Contractors Plant Association
Convention of Scottish Local Authorities
County Surveyor's Society*
Department of the Environment (PSA)
Department of the Environment (Transport and Road Research Laboratory)
Department of Transport*
Federation of Civil Engineering Contractors
Federation of Manufacturers of Construction

Greater London Council
Institute of Petroleum
Institute of Quarrying
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Institution of Highway Engineers*
Institution of Municipal Engineers*
Institution of Structural Engineers
Ministry of Defence
Refined Bitumen Association Ltd.
Road Emulsion Association Ltd.
Road Surface Dressing Association
Sand and Gravel Association Limited
Society of Chemical Industry*
Trades Union Congress

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the Technical Committee entrusted with the preparation of this British Standard:

British Resin Manufacturers' Association Chemical Industries Association Glass Manufacturers' Federation Oil and Colour Chemists Association

Equipment and Cranes

Road Marking Manufacturers and Contractors Association Zinc Pigment Development Association

This British Standard, having been prepared under the direction of the Road Engineering Standards Committee, was published under the authority of the Executive Board and comes into effect on 31 August 1981

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The following BSI references relate to the work on this standard:
Committee reference RDB/25
Draft for comment 79/14982DC

ISBN 0 580 12171 2

Amendments issued since publication

| Amd. No. | Date of issue | Comments |
|----------|-------------------|---------------------------------------|
| 4047 | September 1982 | |
| 4872 | July 1985 | |
| 5600 | September 1987 | Indicated by a sideline in the margin |
| | | |

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Foreword

This British Standard has been prepared under the direction of the Road Engineering Standards Committee specifically to cover solid, reflective glass beads referred to in BS 3262 and in BS 6044. The beads may be incorporated in the mix before application and/or applied to the surface afterwards. The specification also applies to glass beads used in a number of other industrial applications.

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.

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, 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 amendment table on the inside front cover.

1 Scope

This British Standard specifies requirements for bulk supplies of glass beads for road marking, impacting, plastics reinforcement and general industrial applications.

2 References

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

3 Definitions

For the purposes of this British Standard the following definitions apply.

3.1

foreign matter

non-glass materials including magnetic particles

3.2

grains

glass particles that present sharp angles

3 3

gas inclusions

voids in the interior of the glass beads that affect the optical properties

3.4

roundometer

a vibrating glass plate used to separate mechanically spherical beads from other particles

3.5

spherical beads

glass beads that have the required properties when tested using the roundometer or microscope

3.6

fused particles

two or more glass beads that have joined together

3.7

pavement

a road, runway or other paved area

3.8

moisture proof coating

a coating applied to the surface of the glass beads to ensure ease of flow during application

3.9

flotation coating

a clear coating applied to the surface of the glass beads allowing a greater area of the surface applied glass beads to be exposed

4 Classes of glass beads

This British Standard deals with the following classes of glass beads, based on their application.

Class Application

- A Incorporation in thermoplastic road marking compounds.
- B Surface applied for thermoplastic road marking compounds and pavement marking paints.
- C For impacting, plastics reinforcement and general industrial applications.

5 Sampling and testing

- **5.1 General.** For the purposes of carrying out the testing procedures described in Appendix B to Appendix M adequate and representative samples shall be taken in accordance with Appendix A.
- **5.2 Testing.** The samples shall be tested in accordance with Appendix B to Appendix M.

WARNING. Some of the liquids specified for use in the tests may be toxic, corrosive and/or flammable. It is essential that care be taken in handling all chemicals.

6 Properties

6.1 Properties common to all applications (classes A, B and C)

- **6.1.1** *Particle size distribution.* When tested in accordance with Appendix B the glass beads shall comply with the requirements of Table 1, Table 2 or Table 3 as appropriate.
- **6.1.2** Freedom from defects. When tested in accordance with Appendix C and Appendix D (class A) or with Appendix D (classes B and C), glass beads shall comply with the requirements of Table 1, Table 2 or Table 3 as appropriate.
- **6.1.3** *Chemical composition.* When tested in accordance with BS 2649-1, the chemical composition shall be:

| silicon dioxide calcium oxide magnesium oxide | $\left. \begin{array}{l} \rm SiO_2 \\ \rm CaO \\ \rm MgO \end{array} \right\}$ | not less than 70 % together not less than 8 % | |
|---|--|---|--|
| sodium oxide | Na ₂ O | | |
| potassium oxide | K_2 O | together not more | |
| aluminium oxide | $Al_2 O_3$ | than 18 % | |
| ferric oxide | $\mathrm{Fe_2}\mathrm{O_3}$ | | |

The glass shall be colourless [i.e. have no appreciable tint when spread in a single layer on a white ground (see Appendix N)].

- **6.1.4** *Magnetic particles*. When tested in accordance with Appendix F, magnetic particles shall not exceed 0.1 %.
- **6.1.5** Resistance to acid. When tested in accordance with Appendix J, glass beads shall not develop surface haze or dulling (see Appendix N).
- **6.1.6** Resistance to calcium chloride. When tested in accordance with Appendix K, glass beads shall not develop surface haze or dulling (see Appendix N).
- **6.1.7** Resistance to sodium sulphide. When tested in accordance with Appendix L, the sodium sulphide solution shall not darken the glass beads (see Appendix N).
- **6.1.8** Water resistance. Class A and Class B glass beads shall be tested in accordance with Appendix M. The glass beads shall not develop surface haze or dulling (see Appendix N) and titration to the end point shall not require more than 4.5 ml of 0.1 N hydrochloric acid.

6.2 Optical properties: refractive index for roadmarking applications (classes A and B).

When tested in accordance with the method of Appendix E the glass shall have a refractive index of not less than 1.50.

6.3 Properties of coatings for road marking applications (classes A and B)

- **6.3.1** *General*. Coated glass beads shall be tested in accordance with either **6.3.2** or **6.3.3** as specified by the purchaser.
- **6.3.2** *Moisture proofing.* When the glass beads are tested in accordance with Appendix G, the presence of a coating for moisture proofing shall be shown.
- **6.3.3** Flotation coating. When the glass beads are tested in accordance with Appendix H the presence of flotation coating shall be shown.

Table 1 — Class A glass beads: particle size distribution, roundness and defects

| Sieve | % retained | Minimum % spherical beads by mass tested in accordance with Appendix C | Maximum % of defective beads as tested in accordance with Appendix D |
|-------------|------------|--|--|
| 1.18 µm | 0 to 3 | | |
| 850 µm | 5 to 20 | | |
| $425~\mu m$ | 65 to 95 | 70 | 30 |
| below | | | |
| $425~\mu m$ | 0 to 10 | | |

Table 2 — Class B glass beads: particle size distribution, roundness and defects

| Sieve | % retained | Minimum % spherical beads by microscope tested in accordance with Appendix D | Maximum % of defective beads tested in accordance with Appendix D |
|-------|------------|--|---|
| μm | | | |
| 850 | 0 to 5 | | |
| 600 | 5 to 20 | | |
| 300 | 30 to 75 | | |
| 180 | 10 to 30 | 80 | 20 |
| Below | | | |
| 180 | 0 to 15 | | |

NOTE These glass beads may be applied by either gravity feed or air assisted feed and may have a moisture proof coating or a flotation coating. Flotation coatings are most useful for paint applications.

7 Containers

7.1 Material. Containers shall be made of material that does not contaminate the contents and that protects the contents from contamination.

 NOTE . Unless otherwise agreed with the purchaser the solid glass beads are normally supplied either

- a) in bags each containing 25 kg + 0.5 % 0 %, or
- b) on pallets each carrying 1 tonne + 0.5 % 0 % in bags each of 25 kg \pm 0.5 %.

8 Marking

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

- a) the class of glass bead;
- b) the application of glass bead as described in clause 4;
- c) in the case of class B beads, the type of coating i.e.

"Moisture proofing" or (M)

"Flotation" or (F);

- d) the number of this British Standard, i.e. BS 6088¹⁾;
- e) the mass of the contents of the container;
- f) the name, trade mark or other means of identification of the manufacturer;
- g) the batch number;
- h) the date of manufacture.

¹⁾ Marking BS 6088 on or in relation to a product is a claim by the manufacturer that the product has been manufactured in accordance with the requirements of the standard. The accuracy of such a claim is therefore solely the manufacturer's responsibility. Enquiries as to the availability of third party certification to support such claims should be addressed to the Director, British Standards Institution, Maylands Avenue, Hemel Hempstead, Herts HP2 4SQ in the case of certification marks administered by BSI or to the appropriate authority for other certification marks.

Table 3 — Class C glass beads: particle size distribution

| Designation | Sieve | % retained | Minimum % spherical beads by microscope tested in accordance with Appendix D | Maximum % of defective beads tested in accordance with Appendix D |
|-------------|-----------|------------|--|---|
| | μm | | | |
| 1 | 850 | 0 to 10 | | |
| | 425 | 80 to 100 | 70 | 30 |
| | Below 425 | 0 to 20 | | |
| 2 | 425 | 0 to 10 | | |
| | 250 | 80 to 100 | 70 | 30 |
| | Below 250 | 0 to 20 | | |
| 3 | 300 | 0 to 10 | | |
| | 180 | 80 to 100 | 80 | 20 |
| | Below 180 | 0 to 20 | | |
| 4 | 250 | 0 to 10 | | |
| | 150 | 80 to 100 | 80 | 20 |
| | Below 150 | 0 to 20 | | |
| 5 | 212 | 0 to 10 | | |
| | 106 | 80 to 100 | 80 | 20 |
| | Below 106 | 0 to 20 | | |
| 6 | 150 | 0 to 10 | | |
| | 75 | 80 to 100 | 80 | 20 |
| | Below 75 | 0 to 20 | | |
| 7 | 106 | 0 to 10 | | |
| | 53 | 80 to 100 | 80 | 20 |
| | Below 53 | 0 to 20 | | |
| 8 | 90 | 0 to 10 | | |
| | 45 | 80 to 100 | 80 | 20 |
| | Below 45 | 0 to 20 | | |
| 9 | 53 | 0 to 5 | | |
| | 45 | 0 to 20 | 80 | 20 |
| | Below 45 | 80 to 100 | | |

Appendix A Preparation for testing

A.1 General. From every batch or part of a batch take three containers at random for sampling. If one fails take samples from another two; if these pass, the consignment shall be passed.

A.2 Apparatus

- **A.2.1** *Riffle box*, consisting of an even number of rectangular chutes with 12 mm apertures, discharging alternately on opposite sides of the box (see Figure 1).
- **A.2.2** *Two containers*, that will each hold a minimum of 30 kg of glass beads.
- **A.3 Procedure.** Empty the glass beads from the selected container into one of the 30 kg containers, and then pour the glass beads into the other 30 kg container. Repeat this three times to ensure that the glass beads are thoroughly mixed before reduction.

Take a 25 kg sample of glass beads and, ensuring that the glass beads are surface dry, thoroughly mix the sample and pass it through the riffle box. Retain one portion, pass it through the riffle box again and repeat the procedure until the original sample has been reduced to approximately 1 200 g.

NOTE For convenience, a 16 to 1 sample reducer may be used as an alternative to obtain the first reduction (see Figure 2).

A.4 Sample. Retain the sample obtained from the procedure given in **A.3** for use in all the tests.

NOTE It is important that a truly representative sample is taken; surveys have shown that the most common cause of incorrect results in testing has been due to the improper selection of the sample, therefore every care should be taken in the selection of the sample.

Appendix B Test to determine particle size distribution

B.1 Apparatus

- **B.1.1** *Test sieves*, complying with the requirements of BS 410.
- B.1.2 Balance, accurate to 0.1 g.
- **B.1.3** *Containers*, large enough to hold the residue from each sieve.
- **B.1.4** *Riffle box*, with 12 mm apertures (see Figure 1).
- **B.1.5** *Oven*, capable of heating the glass beads to 80 °C to 105 °C.

B.2 Procedure

B.2.1 When testing material containing glass beads less than approximately 200 μm in size, dry it at 80 °C to 105 °C for not less than 5 min before commencing sieving.

Test the glass beads for particle size distribution in accordance with **6.2.5** of BS 1796:1976 using a dry sieving procedure. In the event of dispute, the sieving shall be done by hand.

Using the riffle box reduce the sample obtained by the procedure given in **A.3**, to a size of 50 g to 100 g.

- **B.3 Test report.** The test report shall include the following:
 - a) results of the sieve analysis reported as the total percentage retained on each sieve to the nearest whole number;
 - b) method of sieving, i.e. hand or machine.

Appendix C Test to determine the percentage of spherical beads by weighing

C.1 Principle. To determine the percentage of spherical beads by mechanically separating them into spherical beads and other particles by controlled vibration on a glass plate fixed to a predetermined slope.

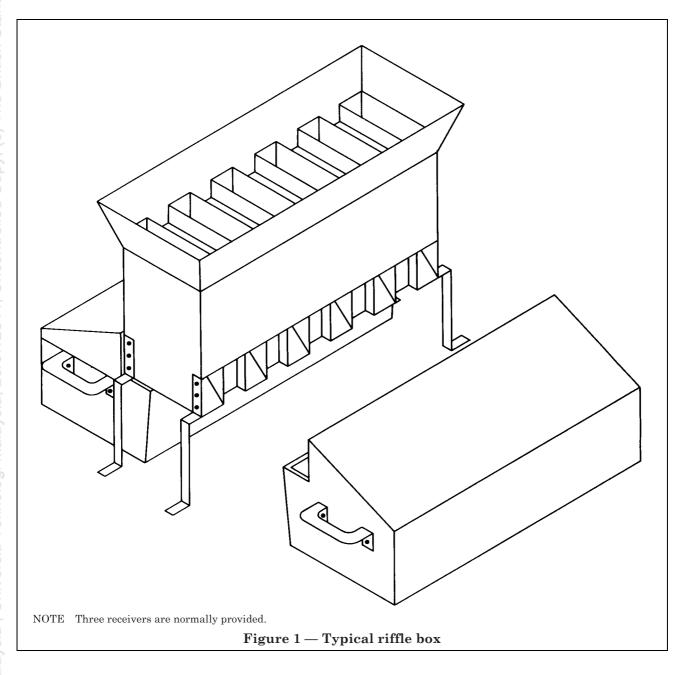
C.2 Apparatus

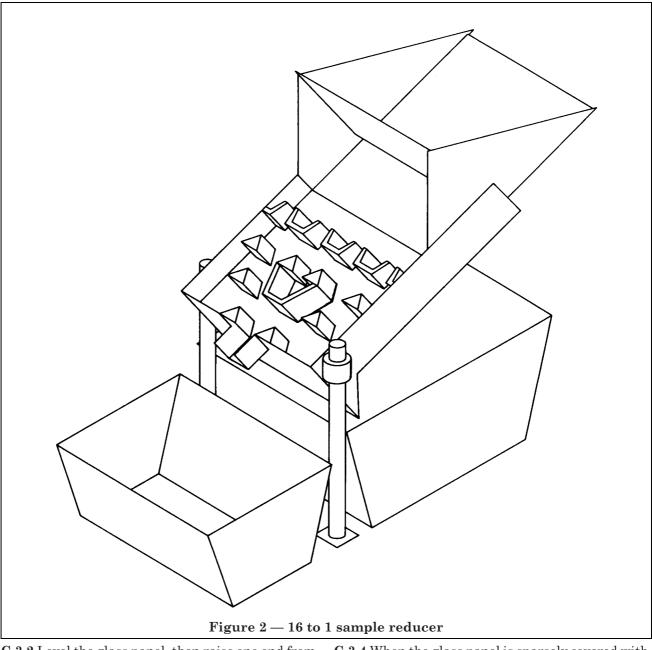
- **C.2.1** *A roundometer*, consisting of the following.
 - a) Electrical feeder-vibrator, upon which is mounted a smooth glass panel, 150 mm wide and 355 mm long.
 - b) Hinged base, supporting the vibrator and panel in such a manner that the angle of the glass panel with the horizontal may be varied and fixed in any predetermined position.
 - c) Vibrator, providing a means of varying the amplitude from nil to 0.5 mm of the longitudinal vibrations transmitted to the glass panel, at a fixed frequency of 50 Hz. The vibration shall be in such a mode that irregular particles move up the slope.
- **C.2.2** Collecting pans or containers, at either end of the sloping panel, in which to collect the spheres and irregular particles.
- C.2.3 Camel hair brush.
- **C.2.4** *Riffle box*, with 6.35 mm apertures (see Figure 1).
- C.2.5 *Riffle box*, with 12 mm apertures (see Figure 1).

C.3 Procedure

NOTE $\;\;$ This procedure is for use with glass beads above 300 μm only.

C.3.1 Reduce the retained sample (see Appendix A) to approximately 10 g by using the riffle boxes.





C.3.2 Level the glass panel, then raise one end from the horizontal to an angle of 1° $35' \pm 10'$. Clean the glass panel with industrial alcohol or methylated spirit. Start the vibrator and feed the glass beads on to the upper third of the slope, from a height not exceeding 15 mm so that they form a single layer on the glass panel.

C.3.3 Adjust the vibrator amplitude control to such a position that irregular particles on the upper half of the panel will move slowly up the slope, while the true spheres roll down. Feed slowly at such a rate that no "bunching up" or flooding of glass beads on the panel occurs.

C.3.4 When the glass panel is sparsely covered with glass beads, stop feeding until separation of the spherical beads has occurred. Stop the vibrator and, after all the spherical beads have rolled down the slope into the sphere pan, brush or scrape all the particles remaining on the panel into the upper pan containing the irregular particles. For the purposes of this test, all particles not rolling freely down the slope shall be considered as irregular.

C.3.5 Repeat the procedure given in C.3.3 and C.3.4 until the sample has been completely separated. Remove the spherical beads and irregular particles from the collecting pans into separate containers.

- C.3.6 Re-run the spherical beads collected in the primary separation, and repeat the procedure given in C.3.3 and C.3.4.
- **C.3.7** Re-run the irregular particles collected in the primary separation and repeat the procedure given in **C.3.3** and **C.3.4**.
- **C.3.8** Determine and record the total mass of the spherical beads and of the irregular particles obtained by the secondary separations described in **C.3.6** and **C.3.7**.
- **C.3.9** From the total mass of spherical beads obtained from both groups, calculate the percentage of spherical beads in the total sample, using as 100 % the total mass of spherical beads plus irregular particles collected in the test.

Appendix D Test to determine the percentage of spherical beads by microscopic count

D.1 Principle. To determine the percentage of spherical beads by counting defective beads in a projected magnified image of a sample on a glass slide. Beads with gas inclusions are counted in a separate examination using a liquid covering to show up the inclusions.

D.2 Apparatus and reagents

- D.2.1 Riffle box, with 6.35 mm apertures.
- **D.2.2** *Microscope*, with attached screen with test area division to allow evaluation of picture with a minimum magnification of 10.
- **D.2.3** *Glass slides*, 25 mm wide, complying with the requirements of BS 3836-1.
- D.2.4 Transparent adhesive tape, 20 mm wide.
- **D.2.5** *Liquid*, of low volatility and refractive index of or near to 1.5^{2}).
- **D.2.6** Eye dropper or syringe.
- **D.2.7** *Oven*, capable of heating the glass beads up to 80 °C to 105 °C.

D.3 Procedure

D.3.1 Take the sample prepared as described in Appendix A and separate further by sieving to give a specimen of sufficient size to allow all the glass beads to be placed on the adhesive tape.

Before sieving material containing uncoated glass beads of less than approximately 200 μm in size, dry at 80 °C to 105 °C for not less than 5 min before commencing sieving.

When the glass beads are of class B (see Table 2) use $180~\mu m$ and $600~\mu m$ sieves and retain the portion retained on the $180~\mu m$ sieve.

When the glass beads are of class C (see Table 3) use the nominal sieves and retain the portion on the smaller sieve.

- **D.3.2** Distribute the whole sample uniformly as a monolayer on the transparent adhesive tape, then attach the tape to a glass slide, with the glass beads facing the slide. Secure the tape to the opposite side of the slide.
- **D.3.3** Project an image of the glass beads on to the screen and adjust magnification so that 250 to 300 particles cover the test area of the screen. If the magnification does not allow this then check several points on the slide until three times the required number is reached and the percentage averaged. If sufficient particles are covered, three points at least 10 mm apart shall be checked on each slide, and averaged.

NOTE To assist the operator a chinagraph pencil may be used to mark off the particles.

- **D.3.4** Defective beads may have several different defects and shall only be reported once on the most obvious one.
- **D.3.5** To identify gas inclusions cover the glass beads with a liquid of refractive index approximately 1.5. Carefully inject the liquid between the tape and slide using the eye dropper or syringe. The gas inclusions appear black and the glass bead outline dark. Glass beads are defective where the black portion exceeds 25 % of its projected surface area.
- **D.3.6** The following shall be classed as defects:
 - a) grains (see 3.2);
 - b) oval or deformed particles;
 - c) fused particles (see 3.6);
 - d) opaque or milky particles;
 - e) foreign matter (see 3.1);
- f) spheres with gas inclusions (see 3.3).
- **D.3.7** Calculate the number of imperfections as follows:
 - a) count the total number of particles in the test area;
 - b) report the quality as:

% imperfections =
$$\frac{\text{number of defective beads}}{\text{total number of particles}} \times 100$$

% spherical beads = 100 % of particles — % of defective beads.

 $^{^{2)}}$ Examples of suitable liquids include glycerol in accordance with BS 2623 or BS 2625 and turpentine in accordance with type 2 of BS 244 & 290.

Appendix E Test to determine the refractive index of solid glass beads

E.1 Principle. To determine whether the refractive index of solid glass beads is higher or lower than that of a comparator liquid of refractive index 1.50.

This method utilizes the principle of the bending of light as it passes from a medium with one index of refraction to another with a different index. No bending is apparent if the different media have the same index of refraction.

E.2 Apparatus and reagents

E.2.1 *Microscope or projector*, with minimum magnification of 10.

E.2.2 *Glass slide*, of medium thickness, having spherical concavities which are ground, polished, and have the approximate dimensions of 10 mm diameter, and 0.8 mm deep.

E.2.3 Microscope illuminator, with iris diaphragm.

E.2.4 *Liquid*, of certified refractive index 1.50.

E.3 Procedure

E.3.1 Place enough glass beads to cover a circle 3 mm in diameter with a single layer in the cavity of the glass slide.

E.3.2 Take the liquid of refractive index 1.50 and place enough of this liquid in the cavity in order to completely cover the glass beads.

E.3.3 Adjust the light from the microscope illuminator so that a limited portion of the area covered by the glass beads is illuminated by a dim light from below.

E.3.4 Focus the microscope initially on the glass beads. Then vary the focus slowly, first in one direction then in the other, while observing the appearance of individual glass beads through the microscope. If, while adjusting the focus in such a direction as to decrease the distance between the microscope stage and the objective lens, a dark ring appears on the circumference of a bead and light concentrates in the centre, the glass beads have an index of refraction lower than 1.50. Adjustment of the focus in the opposite direction shows the glass beads becoming blurred with no ring and bright centre spot appearing.

NOTE $\,$ In this context adjusting the focus of the microscope, means moving the objective lens through a distance of 1 ± 0.5 mm.

If a dark ring and bright centre spot appear when the distance between the microscope stage and the objective lens is increased, then the glass beads have a refractive index higher than 1.50. **E.3.5** If the glass beads have refractive index of 1.50 they shall be almost invisible when perfectly focused and shall have a blurred outline when the focus is moving in either direction.

Appendix F Test to determine the percentage of magnetic particles by weighing

F.1 Principle. To determine the percentage of magnetic particles by passing a sample over paper laid on magnets and collecting the residue on the paper.

F.2 Apparatus

F.2.1 Balance, sensitive to 0.01 g.

F.2.2 Four permanent magnets, mounted in a frame (see Figure 3).

F.2.3 Soft brush, of 12 mm camel hair.

F.2.4 Sample pan, of aluminium.

F.2.5 *Sample cup*, of aluminium.

F.3 Procedure

F.3.1 Take approximately 200 g of the sample from the first reduction for the test (see Appendix A), and weigh to the nearest 0.01 g.

F.3.2 Place a sheet of white paper over the magnets.

F.3.3 Place the sample pan under the end of the paper to catch the beads.

F.3.4 Holding the paper with one hand, slowly pour the sample over the magnet area with the other hand. After the sample has been poured over the paper, the magnetic material will remain on the paper.

F.3.5 Bring the paper at the lower end into a horizontal position and brush the magnetic material into an aluminium cup.

F.3.6 Repeat the whole procedure until the sample has been passed over the magnets three times, or until no particles are visible on the paper. Weigh the total amount of magnetic material to the nearest 0.01 g.

F.3.7 Calculate the percentage of magnetic particles in the sample as follows:

% magnetic particles =
$$\frac{\text{mass of residue}}{\text{mass of sample}} \times 100$$

Appendix G Test to determine the presence of moisture proof coating

G.1 Principle. To test for the presence of a moisture proof coating by either of the following procedures as applicable: procedure A which is time consuming but more accurate, and should be used when precise results are required; procedure B which gives a quick indication that moisture proofing is present and only takes a few seconds.

G.2 Apparatus

- **G.2.1** *Funnel*, with a top diameter of 150 mm, 120 mm deep and a 6.25 mm inside diameter stem.
- **G.2.2** Washed cotton bag with 48×48 thread count (size approximately $450 \text{ mm} \times 250 \text{ mm}$).
- **G.2.3** *Bucket* with a minimum capacity of 4 litres filled with potable water at room temperature.
- G.2.4 Beaker, 500 ml.

G.3 Procedure

G.3.1 Procedure A

- G.3.1.1 Using the beads taken from the initial reduction (see Appendix A) check the mass which should be approximately $400~\rm g.$
- G.3.1.2 Turn the cotton bag (G.2.2) inside out and pour in the sample.
- **G.3.1.3** Immerse the bag containing the sample in the bucket of water for 30 s or until the bag is completely immersed (whichever is longer).
- **G.3.1.4** Remove the bag and sample from the water and squeeze the excess water out of the bag by twisting the neck of the bag.
- **G.3.1.5** With the neck of the bag still twisted tight, hang the bag up to drain at room temperature for 2 h.
- **G.3.1.6** At the end of the 2 h, mix the sample thoroughly by releasing the tension on the neck and shaking the bag, thus loosening the beads from the bottom and sides of the bag.
- **G.3.1.7** Transfer the sample to a clean dry funnel (**G.2.1**). The entire sample should flow through the funnel without stoppage. Failure to flow shall be considered as failure to pass the test.
- NOTE $\,$ In case the beads block the funnel when first introduced, it is permissible to tap the funnel stem lightly to initiate the flow.
- **G.3.1.8** If the result is as described in **G.3.1.7** as flowing without stoppage, the glass beads shall pass the test

G.3.2 Procedure B

G.3.2.1 Pour water into the beaker until it is approximately three quarters full.

- **G.3.2.2** Take 20 g to 25 g of glass beads from the initial reduction (see Appendix A) and pour them slowly but continuously on to the surface of the water
- **G.3.2.3** When the glass beads break the surface they should spiral to the bottom of the beaker in a continuous string, or in lumps, which indicates a moisture proof coating is present.
- **G.3.2.4** If the glass beads fall to the bottom of the beaker individually this indicates insufficient, or ineffective coating.
- **G.3.2.5** If the result is as described in **G.3.2.3** the glass beads shall pass the test. If the result is as described in **G.3.2.4** they shall have failed, and procedure A shall then be conducted.

Appendix H Test to determine the presence of flotation coating

H.1 Principle. To determine the presence of flotation coating by estimating the percentage of glass beads floating on the surface of xylene or *n*-heptane.

H.2 Apparatus and reagents

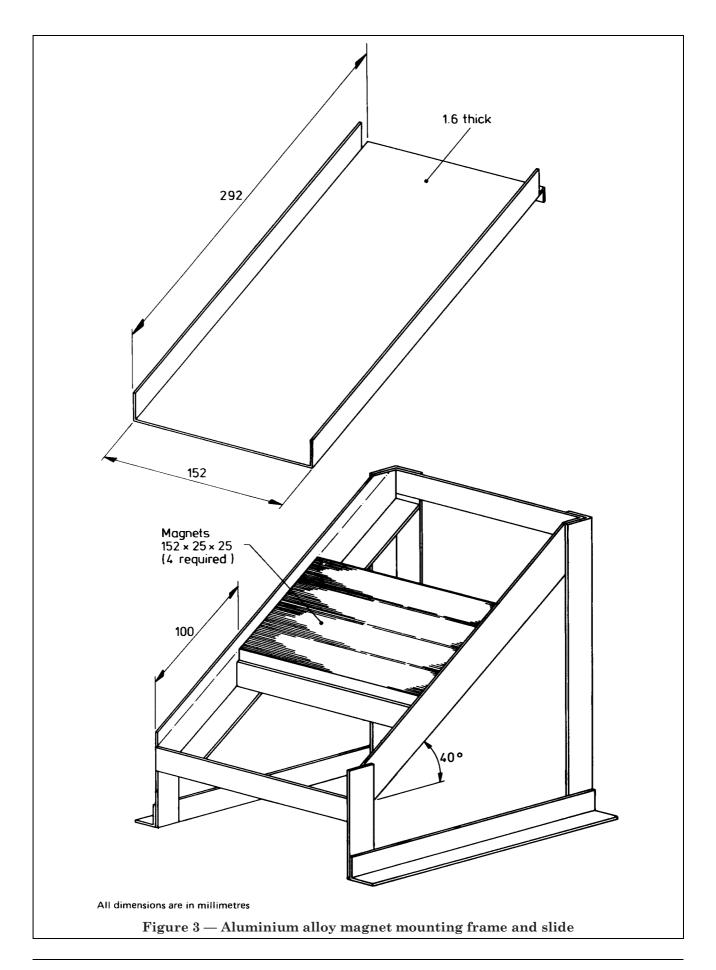
- **H.2.1** Watch glass or petri dish, 50 mm to 75 mm in diameter complying with the requirements of BS 611-1.
- **H.2.2** Syringe, pipette or eye dropper, 5 ml to 10 ml capacity.
- **H.2.3** *Test sieves*, complying with the requirements of BS 410.
- H.2.4 Xylene, of reagent grade.
- **H.2.5** *n-Heptane*, of reagent grade.

H.3 Procedure

- **H.3.1** Sieve out from the retained sample (see Appendix A) the fraction passing a 300 μm sieve but retained on a 180 μm sieve.
- **H.3.2** Spread a monolayer of glass beads on to the clean watch glass and, using the syringe, slowly introduce the xylene at the edge of the watch glass until the liquid is deep enough to allow the beads to float. Care should be taken to avoid agitation of the glass beads whilst the xylene is being added.
- **H.3.3** Visually estimate the percentage of glass beads floating on the surface of the xylene.
- **H.3.4** Repeat **H.3.1** and **H.3.2** using *n*-heptane in place of xylene, using a new sample of glass beads.
- **H.4 Results.** In order to pass the test the minimum percentage of glass beads floating shall be as follows.

Liquid Minimum % floating

Xylene 90 n-Heptane 75



Appendix J Test to determine the resistance of glass beads to acid

J.1 Principle. To determine the resistance of glass beads to acid by treating them with sulphuric acid and examining them for surface haze or dulling.

J.2 Apparatus and reagent

- J.2.1 Sulphuric acid solution, 1.0 N.
- J.2.2 Glass beaker, 100 ml.
- J.2.3 Distilled water.
- **J.2.4** *Microscope*, with minimum magnification of 10.

J.3 Procedure

- **J.3.1** Take approximately 10 g of glass beads from the retained sample (see Appendix A).
- **J.3.2** Place the glass beads in a 100 ml glass beaker, cover with the sulphuric acid solution and allow to stand for 5 min.
- **J.3.3** Pour off the sulphuric acid solution and then rinse the glass beads three times with distilled water.
- **J.3.4** Dry the glass beads in an oven controlled at 100 ± 5 °C and using the microscope compare these with an untreated sample.
- **J.4 Results.** When compared with an untreated sample (see Appendix N) the glass beads shall not develop surface haze or dulling.

Appendix K Test to determine the resistance of glass beads to calcium chloride

K.1 Principle. To determine the resistance of glass beads to acid by treating them with calcium chloride solution and then examining them for surface haze or dulling.

K.2 Apparatus and reagents

- K.2.1 Glass beaker, 100 ml.
- K.2.2 Calcium chloride solution, 1.0 N.
- K.2.3 Distilled water.
- **K.2.4** *Microscope*, with minimum magnification of 10.

K.3 Procedure

- **K.3.1** Take approximately 10 g of glass beads from the retained sample (see Appendix A).
- **K.3.2** Place the glass beads in the 100 ml glass beaker, cover with the calcium chloride solution and allow to stand for three hours.
- **K.3.3** Pour off the calcium chloride solution and rinse three times with distilled water.

- **K.3.4** Dry the glass beads in an oven controlled at 100 ± 5 °C and using the microscope compare these with an untreated sample.
- **K.4 Results.** When compared with an untreated sample (see Appendix N) the glass beads shall not develop surface haze or dulling.

Appendix L Test to determine the resistance of glass beads to sodium sulphide

L.1 Principle. To determine the resistance of glass beads to acid by treating them with sodium sulphide and then examining them for darkening.

L.2 Apparatus and reagents

- L.2.1 Bottle, 50 ml with a glass stopper.
- **L.2.2** *Solution*, containing by mass 50 % sodium sulphide as Na₂ S 48 % distilled water, 2 % anionic wetting agent³⁾.
- L.2.3 Distilled water.
- **L.2.4** *Microscope*, with minimum magnification of 10.

L.3 Procedure

- **L.3.1** Take 10 g of glass beads from the retained sample (see Appendix A).
- **L.3.2** Place the glass beads in a stoppered bottle and cover with the solution containing the sodium sulphide and allow to stand for 1 h.
- **L.3.3** Pour off the solution containing the sodium sulphide and rinse three times with distilled water.
- **L.3.4** Dry the glass beads in an oven at 100 ± 5 °C and, using the microscope, compare these with an untreated sample.
- **L.4 Results.** When compared with an untreated sample (see Appendix N) the glass beads shall not darken.

Appendix M Test to determine the resistance of glass beads to water

M.1 Principle. To determine the resistance of glass beads to water by refluxing a sample in a Soxhlet extractor and by examining the dried beads afterwards for surface haze and dulling by comparison with an untreated sample, and by measuring the basicity of the residual liquid in the boiling flask by titration.

M.2 Apparatus and reagents

- **M.2.1** Extraction thimble, $25 \text{ mm} \times 80 \text{ mm}$.
- **M.2.2** No. 3 Soxhlet extractor, with a 125 ml boiling flask.

³⁾ Agents in which the base is a linear alkyl benzene sulphonate, a linear alkyl sulphate or a linear alkyl ethoxy sulphate. (For the method of determination see BS 3762.)

- **M.2.3** *Distilled water*, or water of equivalent purity complying with the requirements of BS 3978.
- | M.2.4 Hydrochloric acid, 0.1 N.
 - M.2.5 1% phenolphthalein indicator.

M.3 Procedure

- **M.3.1** Take 10 g of glass beads from the sample retained (see Appendix A).
- **M.3.2** Place the sample on the extraction thimble and place in the Soxhlet extractor with the 125 ml boiling flask.
- M.3.3 Add 100 ml of distilled water and reflux for 2 h.
- **M.3.4** Dry the glass beads in an oven at 100 ± 5 °C and using the microscope compare these with an untreated sample (see Appendix N) for surface haze and dulling.
- **M.3.5** Add 5 drops of 1 % phenolphthalein indicator to the contents of the boiling flask and titrate with the hydrochloric acid solution to the indicator end point.
- **M.3.6** Titration to the end point shall not require more than 4.5 ml of 0.1 N hydrochloric acid.
- **M.4 Results.** When compared with an untreated sample (see Appendix N) the glass beads shall not develop surface haze or dulling.

Appendix N Dispute procedure for subjective tests

N.1 General. In the event of difficulty over the interpretation of the subjective tests described in Appendix J, Appendix K, Appendix L and Appendix M the following procedure shall be adopted under the supervision of an arbitrator who is acceptable to the two parties and whose decision shall be final.

N.2 Procedure

- **N.2.1** Take 10 samples of glass beads before treatment and form from each a monolayer in a 100 mm diameter petri dish, on the floor of which has been laid flat a round of white filter paper.
- **N.2.2** Where the glass beads are being examined for freedom from colour, take these 10 comparator samples from an agreed, colourless consignment of similar size grading.
- **N.2.3** Place a sample of the glass beads under examination similarly in an eleventh identical petri dish.

- **N.2.4** The two parties without the arbitrator shall now place the 11 dishes in random order in a circle of radius 200 mm, and illuminated by an automobile filament headlamp complying with the requirements of BS 941 and BS AU 40-3 placed 2 400 mm vertically above the circle centre. The two parties shall record the position of the sample under examination.
- **N.3 Results.** The arbitrator shall then examine by the unaided eye the samples. If he can identify the sample under test in each of five such trials the defect has been established.

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Publications referred to

BS 244 & 290, Turpentine for paints.

BS 410, Specification for test sieves.

BS 611, Petri dishes.

BS 611-1, Specification for glass petri dishes.

BS 941, Filament lamps for road vehicles.

BS 1796, Method for test sieving.

BS 2623, Technical glycerol.

BS 2625, Chemically pure glycerol.

BS 2649, Methods for the analysis of glass.

BS 2649-1, Recommended procedure for the analysis of glasses of the soda-lime-magnesia-silica type.

BS 3262, Hot applied thermoplastic road marking materials.

BS 3762, Methods of sampling and testing detergents.

BS 3836, Components of microscopes.

BS 3836-1, Microscope cover slips and slides and immersion fluid.

BS 3978, Water for laboratory use.

BS 6044, Pavement marking paints⁴⁾.

BS AU 40, Motor vehicle lighting and signalling equipment.

BS AU 40-1, Slide, rear, parking, marker and stop lamps, and direction indicators.

⁴⁾ Referred to in the foreword only.

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