BRITISH STANDARD

BS 388:1972

Specification for

Aluminium pigments

Confirmed
February 2011

Co-operating organizations

The Pigments, Paints and Varnishes Industry Standards Committee, under whose supervision this British Standard was prepared, consists of representatives from the following Government departments and scientific and industrial organizations:

British Colour Makers' Association
British Railways Board
Chemical Industries Association
Crown Agents for Oversea Governments and Administrations
Department of the Environment
Department of Trade and Industry
Department of Trade and Industry, Laboratory of the Government Chemist
Greater London Council
London Transport Executive
Ministry of Defence, Army Department*
Ministry of Defence, Navy Department
National Federation of Builders' and Plumbers' Merchants
Oil and Colour Chemists' Association
Paint Manufacturers and Allied Trades Association
Paintmakers' Association of Great Britain Ltd.*
Post Office
Research Association of British Paint, Colour and Varnish Manufacturers*
Royal Institute of British Architects
Royal Institute of Public Health and Hygiene
Society of Chemical Industry
Titanium Pigment Manufacturers' Technical Committee
White Lead Manufacturers' Association
Zinc Development Association
Zinc Pigment Development Association

The Government department and scientific and industrial organizations marked with an asterisk in the above list, together with the following, were directly represented on the committee entrusted with the preparation of this British Standard:

Aluminium Federation
Amalgamated Society of Painters and Decorators
Decorators of England and Wales
National Federation of Master Painters

This British Standard, having been approved by the Pigments, Paints and Varnishes Industry Standards Committee, was published under the authority of the Executive Board on 16 May 1972

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Amendments issued since publication

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Contents

Foreword

This standard makes reference to the following British Standards:

BS 245, *White spirit.*

BS 410, *Test sieves.*

BS 1728, *Methods for the analysis of aluminium and aluminium alloys — Part 5: Determination of copper (absorptiometric method) — Part 8: Determination of iron (absorptiometric 1 : 10 phenanthroline method) — Part 10: Determination of manganese (absorptiometric method) — Part 12 Silicon (absorptiometric molybdenum blue method) — Part 18 Method for the determination of zinc (ion-exchange-volumetric EDTA or polarographic method).*

BS 1752, *Laboratory sintered or fritted filters.*

BS 2511, *Methods for the determination of water (Karl Fischer method).*

BS 3483, *Methods for testing pigments for paints.*

BS 3978, *Water for laboratory use.*

BS 4726, *Sampling raw materials for paints and varnishes.*

The British Standard for aluminium pigments was first issued in 1930 and was subsequently revised in 1938, 1952 and 1964.

The present revision is technically identical with ISO/R 1247, *"Aluminium pigments"*, except that in this standard there is an additional requirement that the leafing power of the leafing material shall be not less than 90 % of that of the agreed sample.

NOTE The use of chlorinated hydrocarbon solvents with aluminium powder or paste can be hazardous if heating is involved, since a rapid exothermic reaction may take place. Experience has shown, however, that chloroform presents no danger when used as described in Appendix G.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 14 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 the corresponding methods of test for aluminium pigments used in paints including

1) general purpose, decorative and protective paints, and

2) speciality finishing paints.

NOTE The titles of the British Standards referred to in this standard are listed on page ii.

2 Sampling

For the purposes of testing a pigment in accordance with this standard, a sample representative of the bulk material shall be taken by an appropriate procedure, as described in BS 4726. The sample shall not be less than 250 g in mass.

For both pastes and powders a sampling tube should be used that enables the whole depth of the container to be sampled (and preferably, the longest diagonal of the container). For powders, which may be compacted, a sharply pointed sampling tube should be used, and the container should be rolled vigorously before sampling.

Suitable designs of sampling tubes are illustrated in Figure 1.

3 Agreed sample

Except where otherwise agreed between the purchaser and the vendor, the agreed sample referred to in Table 2 shall comply in all respects with the requirements of this standard. The mass of the sample shall be not less than 250 g and shall be packed in the manner described in BS 4726.

4 Description

The material shall be composed essentially of finely divided aluminium metal. The particles of aluminium metal shall be lamellar, which can be confirmed by microscopical examination. The material shall be in the form of a powder or a homogeneous paste.

NOTE Mica and other adulterants shall be absent. If when preparing a solution of the sample in hydrochloric acid in **H.1.3.3** a non-fatty residue is obtained, the residue should be examined further.

5 Classification

5.1 Types. This British Standard covers four types of aluminium pigments, as follows:

Type 1. Aluminium powder, leafing

Type 2. Aluminium paste, leafing

Type 3. Aluminium powder, non-leafing

Type 4. Aluminium paste, non-leafing

5.2 Classes. Pigments of Types 1 and 2 are further classified by their water-covering capacity as shown in Table 1.

Table 1 — Classification of materials

Type	Class	Water-covering capacity m^2/g
1	a	up to 0.8
	b	over 0.8 up to 1.5
	c	over 1.5 up to 2.2
	d	over 2.2
2	p	up to 1.7
	q	over 1.7 up to 2.4
	r	over 2.4

NOTE Attention is drawn to the reproducibility limits given in **D.6.2**.

6 Required characteristics and their tolerances

The material shall have the characteristics given in the appropriate column of Table 2.

The liquid contained in paste pigment shall be white spirit complying with the requirements of BS 245, or other appropriate liquid as may be agreed between the purchaser and the vendor.

7 Packing

The material shall be packed in air-tight containers.

Table 2 — Required characteristics and their tolerances

Type	Type 1 leafing powder	Type 2 leafing paste	Type 3 non-leafing powder	Type 4 non-leafing paste	Test method
Matter volatile at 105 °C,% max.	1.0	35.0[a]	1.0	35.0[a]	BS 3483
Matter soluble in organic solvents, % max.	6.0	4.0	1.5	6.0	Appendix A (Types 1 and 2, Method 1; Types 3 and 4, Method 2)
Appearance of paint prepared in an agreed vehicle	To match closely the appearance of paint prepared similarly from the agreed sample				Appendix B
Residue on sieve[b]	Nil on 250 µm	Nil on 180 µm	Nil on 250 µm	Nil on 180 µm	Appendix C
Water-covering capacity m²/g	Within the limits for the agreed class (see clause **5**)		—	—	Appendix D
leafing power	65 % min. and not less than 90 % of that of the agreed sample	65 % min. and not less than 90 % of that of the agreed sample	Nil	Nil	Appendix E (Types 1 and 2) Appendix F (Types 3 and 4)
Water content, % max.	0.2	0.15	0.2	0.15	Appendix G
Metallic impurities, % max., on dry pigment	1.0 for Cu + Fe + Pb + Si + Zn. 0.03 for Pb. Separate limits for metals other than lead may be agreed between the purchaser and the vendor.		Limits to be agreed between the purchaser and the vendor.		Appendix H

[a] Alternative limits for volatile content of pastes may be agreed between the purchaser and the vendor.

[b] Additional limits for residue on sieves of smaller apertures may be agreed between the purchaser and the vendor.

Appendix A Method for the determination of matter soluble in organic solvents

A.1 Method 1 (for use with leafing pigments, Types 1 and 2)

A.1.1 *Principle.* The sample is treated with hydrochloric acid to dissolve the metal and the residual oily and fatty matter is extracted with acetone and weighed.

A.1.2 *Reagents.* The reagents used shall be of recognized analytical quality. Water that complies with the requirements of BS 3978 shall be used.

1) *Hydrochloric acid solution,* approximately 6N.

2) *Acetone.*

A.1.3 *Procedure*

A.1.3.1 *Test portion.* Weigh, to the nearest milligram, about 2 g of the test sample into a 400 ml beaker.

A.1.3.2 *Determination.* Add 100 ml of hot water to the test portion and cover the beaker, e.g. with a watch glass. Add the hydrochloric acid in small portions, heating gently to complete the reaction after each addition until all the metal is dissolved. Not more than 60 ml of acid should be required.

Cool the beaker and contents to room temperature and filter the contents through an acid-washed, grease-free filter paper. Thoroughly wash the beaker, the cover and the filter paper with cold water.

Allow the paper to drain and dry completely in the filter funnel, heating gently, if necessary, to a temperature not exceeding 50 °C. Remove as much water as possible from the beaker by shaking it.

Place a weighed 100 ml beaker under the funnel. Wash the original beaker and cover with warm acetone and transfer the washings to the filter paper. Wash the paper at least five times with warm acetone filling it about half full each time. Finally, rinse the tip of the funnel.

Gently warm the beaker and its contents on a water-bath without using a free flame, until the acetone has been evaporated as completely as is possible. Continue the evaporation by heating the beaker at a temperature of 105 ± 2 °C for 1 h, cool and weigh.

A.1.4 *Calculation.* Calculate the matter soluble in organic solvents, as a percentage by mass, by means of the following formula:

$$\frac{100\ m_1}{m}$$

where m = mass of test portion (g)

m_1 = mass of residue (g).

A.2 Method 2 (for use with non-leafing pigments, Types 3 and 4)

A.2.1 *Principle.* The sample is dispersed in solvent and the solvent extractable matter is filtered off, dried and weighed.

A.2.2 *Apparatus.* *Sintered glass filter crucible,* of porosity Grade 4 that complies with the requirements of BS 1752.

A.2.3 *Reagents*

1) *Solvent mixture.* Mix 3 parts by volume of toluene with 1 part of diethyl ether, $d = 0.720$.

2) *Light petroleum,* boiling range of from 40 to 60 °C.

A.2.4 *Procedure*

A.2.4.1 *Test portion.* Weigh, to the nearest milligram, about 2 g of the test sample into a 250 ml beaker and disperse it in 20 ml of the solvent mixture, with frequent intermittent swirling of the contents of the beaker.

A.2.4.2 *Determination.* When complete dispersion has been obtained add a further 10 ml of the solvent mixture, thoroughly agitate by swirling the beaker and then allow to stand for 1 h to enable the metallic flakes to settle.

Decant the supernatant liquid into the sintered glass filter crucible and filter by suction into a clean flask.

When all the liquid has been filtered, add a further 30 ml of the solvent mixture to the residue in the beaker and repeat the swirling so as to redisperse the aluminium pigment. Filter the dispersion through the sintered glass filter crucible, washing the beaker with the light petroleum spirit.

Transfer the filtrate from the flask to a 250 ml beaker and evaporate to minimum bulk (about 50 ml). Transfer the concentrated filtrate to a tared 100 ml beaker and wash the 250 ml beaker with the light petroleum, transferring the washings into the 100 ml beaker. Evaporate the contents of the 100 ml beaker just to dryness, heat in an oven at a temperature of 105 ± 2 °C for 1 h, then cool and weigh.

A.2.5 ***Calculation.*** Calculate the matter soluble in organic solvents, as a percentage by mass, by means of the following formula:

$$\frac{100\ m_1}{m}$$

where m = mass of test portion (g)

m_1 = mass of residue (g).

Appendix B Method for the comparison of appearance

B.1 Procedure

B.1.1 ***Test portion.*** Weigh an amount of test sample that has been agreed between the purchaser and the vendor.

B.1.2 ***Assessment.*** Disperse the test portion in a paint vehicle to be agreed between the purchaser and the vendor (see Note) in the agreed proportions, by simple mixing without grinding. Store in a covered container for a period to be agreed between the purchaser and the vendor, e.g. 24 h at a temperature also to be agreed, preferably 20 °C. After this period remove any surface skin, mix well by shaking or stirring, or both, and apply a coat of the mixture by a suitable method to a smooth, clean, non-absorbent panel, and allow to dry thoroughly in a clean atmosphere. Treat the agreed sample in a similar way on the same day. When dry, compare visually the two panels for colour, opacity, finish and brightness.

NOTE For leafing pigments, the vehicle should have an acid value less than 7.5 mg of KOH per gram and should not contain lead driers.

Appendix C Method for the determination of residue on sieve

C.1 Apparatus

The following apparatus is required.

1) *Containers,* 3, of suitable size to accommodate the sieve.

2) *Beaker,* 400 ml.

3) *Test sieve* of nominal aperture 180 µm (for pastes) or 250 µm (for powders) complying with BS 410 (see Note to **C.3.1**).

4) *Sintered glass filter,* of porosity Grade 4 that complies with the requirements of BS 1752.

C.2 Reagents

The following reagents are required.

1) *White spirit,* complying with the requirements of BS 245.

2) *Acetone,* of recognized commercial quality.

C.3 Procedure

C.3.1 ***Test portion.*** Weigh 10 g of the sample, to the nearest 0.1 g, into a 400 ml beaker.

NOTE If it is required by agreement between the purchaser and the vendor to determine the residue on a sieve of nominal aperture other than 180 µm (for pastes) or 250 µm (for powders), the procedure to be adopted is similar to that described, except that the mass of the test portion should be correspondingly reduced for sieves of smaller nominal aperture.

C.3.2 ***Determination.*** Half fill two of the containers with the white spirit and half fill the third container with the acetone.

Mix the test portion with 100 ml of the white spirit. Add a further 50 ml of the white spirit with vigorous stirring. Pour the suspension slowly onto the surface of the test sieve, adjusting the speed of transference so that the majority of the suspension passes through. Wash the residue on the sieve by holding the sieve in the first container at a slight angle to the surface of the white spirit and shaking the sieve backwards and forwards so that the screen surface passes just under and just above the level of the liquid. Continue this operation for 1 min and then repeat the procedure in the second container for about 2 min.

When it is evident that no more material passes through the sieve, repeat the procedure in the acetone container for 2–3 min. Wash down the sides of the sieve with a small stream of acetone and collect the residue on one side. Transfer the residue, by washing with a minimum quantity of acetone, to the tared sintered glass filter and apply suction.

As soon as filtration is complete and the surface of the residue is apparently dry, place the filter in an oven at 105 ± 2 °C and heat at this temperature for 1 h. Weigh the residue to the nearest milligram and calculate the percentage of the mass of the test portion.

C.4 Calculation

Calculate the residue on sieve, as a percentage by mass, by the following formula:

$$\frac{100 m_1}{m}$$

where m = mass of test portion (g)

m_1 = mass of residue on sieve (g).

Appendix D Method for the determination of water-covering capacity

D.1 Principle

The water-covering capacity is measured in the standard apparatus on a sample that has been carefully washed with petroleum spirit and filtered under vacuum. The test involves a comparative trial to determine the number of washings (between 3 and 6) that gives the maximum result for water-covering capacity.

NOTE Experience has shown that it is essential to carry out the test precisely as described below if reproducible results are to be achieved.

D.2 Apparatus

Usual laboratory equipment and in particular the following apparatus are required.

1) *Evaporating dish,* porcelain, approximately 200 mm in diameter.

2) *Brushes,* small, camel hair.

3) *Sintered glass filter crucible,* of porosity Grade 4 that complies with the requirements of BS 1752.

4) *Trough,* rectangular in shape, supported on levelling screws, approximately 650 mm long, internal width 120 mm and 13 mm to 15 mm deep with vertical sides about 13 mm thick, machined and finished smooth on the upper surface (see Figure 2). (The trough used may conveniently be fabricated from block aluminium, in which case the bottom may be covered internally with black adhesive tape so as to exclude reflections that may interfere with measurement of the length of the pigment film.)

5) *Barriers* (for trough), 2, made of glass or polished plastics materials (e.g. transparent acrylic resin) of thickness approximately 7 mm, width approximately 25 mm and length slightly more than the width of the trough, with the extremities of one edge slightly inset so that when a barrier is resting on the sides of the trough the lower edge lies slightly below the sides of the trough.

6) *Watch-glass,* approximately 50 mm in diameter.

D.3 Reagents

The following reagents are required.

1) *Petroleum spirit,* boiling range of from 80 °C to 100 °C.

2) *Butan-2-ol,* laboratory grade.

3) *Paraffin wax,* laboratory quality. The melting point of the wax should be about 50 °C.

D.4 Pre-treatment of sample

Place the test sample (about 0.5 g for powder, about 1.0 g for paste) in the evaporating dish; add in a few portions, 50 ml of the petroleum spirit and thoroughly disperse with a brush. Allow to stand for 10 min. Filter through the sintered glass crucible and apply suction. Disconnect the vacuum line.

Transfer the filter cake to the evaporating dish with a brush, redisperse it with 50 ml of the petroleum spirit, part of which may be used to wash the dish. Filter as before.

Repeat the whole operation of the preceding paragraph (i.e. a total of three dispersing operations) and leave the filter cake under suction for 30 min after it appears to be dry. Then set aside a small portion of the filter cake.

Repeat these same operations once, twice and three times more (i.e. 4, 5 and 6 dispersing operations, respectively), setting aside small portions of the filter cake each time.

Place the small portions of filter cake on separate pieces of gloss paper and mix each with a dry brush.

Allow the portions to dry in a clean, dry atmosphere for 2 h at room temperature, occasionally mixing with a dry brush.

D.5 Preparation of trough

Prepare the trough for use by cleaning, drying and rubbing with the paraffin wax and polishing with a soft cloth.

Run water into the trough until the level of the meniscus is appreciably above the sides of the trough, adjusting the levelling screws as necessary.

Sweep the water surface from end to end until it is visibly free from dust, using one barrier. Place the barrier near one end of the trough and blow away any remaining dust. Place the second barrier alongside the first and slide it along toward the other end of the trough so that there is a clear stretch of water between the two barriers.

NOTE It is essential that the two barriers are placed sufficiently wide apart, otherwise the pigment film will not be satisfactorily formed when carrying out the procedure described in **D.6.2**.

Adjust the water level so that it is just below the upper edges of the trough and in contact with the lower edges of the barriers.

D.6 Procedure

D.6.1 ***Test portion.*** Weigh, to the nearest 0.1 mg, on the watch-glass, a quantity of the treated sample that has been slurried and filtered three times (as described in **D.4**) and which will give a final film length on the trough of between 150 mm and 300 mm. In order to determine the actual quantity of the treated sample to use, carry out a preliminary test using 20 mg of the treated sample.

NOTE It has been found by experience that the manipulation of the film in the trough is facilitated if the length of film, when measured, is between 150 mm and 300 mm.

D.6.2 ***Determination.*** Add from a dropping bottle such a quantity of the butan-2-ol to the sample that, after stirring (for at least 30 s) with a glass rod, a slurry of smooth consistency is obtained (at least 2 ml will usually be required).

Distribute this slurry on the water surface of the trough between the barrier by holding the watch-glass and slurry in a sloping position in the water of the trough so that the watch-glass just dips into the water. The slurry should be distributed upon the water surface immediately and almost completely. Raise the rim of the watch-glass just clear of the water and wash the remaining slurry into the trough with water from a wash bottle.

When the surface film ceases moving, raise the water level by running additional water into the trough until the level is appreciably above the sides of the trough. Complete the distribution of the test portion on the water by stirring with a glass rod. It is important that as little work as possible should be done at this stage to avoid overlapping of particles.

Move one barrier towards the other, gently sweeping the pigment film before it and move the barrier backwards and forwards, while wrinkles alternately form and disappear. Adjust the barrier in the position in which the wrinkles have just disappeared.

NOTE 1 It should be noted that the barriers should not be used for stirring the film.

NOTE 2 Draughts should be avoided at all times and no attempt should be made to distribute the film by blowing on it.

NOTE 3 Movement of the barriers should be minimal, consistent with obtaining an unbroken pigment film. Overworking of the film is likely to produce low results.

Repeat the operations of the previous paragraph, using the other barrier. The two barriers should now be positioned parallel to one another and at right angles to the edge of the trough.

Measure the length in millimetres, of the pigment film between the barriers. After the measurement has been completed check that only a negligible quantity of pigment is left adhering to the barriers, the glass rod and the sides of the trough. If this is not the case, discard the result.

Repeat the whole of the previous operation with a portion of the treated sample that has been dispersed and filtered four times (see **D.4**). Again repeat the whole operation with portions of the treated sample that have been dispersed and filtered five and six times. Note the number of dispersions and filtrations required to give a maximum value for the length of the pigment film.

Repeat the complete test from the beginning of **D.4** with portions of sample that have been dispersed and filtered for the number of times previously found to give the maximum value until three values are obtained, which evaluated according to **D.7** yield results differing from their mean by not more than ± 0.05 m^2/g.

D.7 Calculation

Calculate the water-covering capacity in square metres per gram by the following formula:

$$\frac{L \times W}{m \times 10^6}$$

where L = length of pigment film (mm)
W = width of pigment film (mm)
m = mass of the test portion (g).

D.7.1 ***Expression of results.*** Report the mean value, rounded to the nearest 0.01 m^2/g, as the water-covering capacity.

D.7.2 *Precision*

D.7.2.1 *Repeatability.* The results of tests on the same sample by the same operator with the same apparatus, should not be considered suspect unless the extreme values differ by more than 0.1 m^2/g.

D.7.2.2 *Reproducibility.* The results of tests on the same sample in different laboratories should not be considered suspect unless the extreme values differ by more than 0.3 m^2/g.

Appendix E Method for the determination of leafing power

E.1 Apparatus

The following apparatus is required.

1) *Steel strip,* of length not less than 140 mm, width 13 ± 0.5 mm, thickness not more than 1.0 mm, rectangular and square ended. The strip should be abraded to a satin finish by using aluminium oxide, silicon carbide or emery powder, of grade 000 or equivalent, wetted with white spirit.

2) *Glass cylinder,* with foot, preferably without spout, about 200 mm in height and 40 mm in internal diameter.

3) *Corks,* 2, to fit the glass cylinder, one slotted to hold the steel strip when suspended vertically in the cylinder.

4) *Test tubes,* about 150 mm in length and 19 mm to 20 mm in external diameter.

5) *Evaporating dish or spouted capsule,* 35 ml to 50 ml capacity.

E.2 Reagents

The following reagents are required.

1) *White spirit,* the aromatics content of which shall have been adjusted to 20 % (v/v) by the addition of xylene.

NOTE Normally the white spirit supplier will be able to provide the figure for the aromatic content but failing this a method for determining it is described in BS 245.

2) *Coumarone-indene or hydrocarbon resin,* with an acid value not greater than 0.5, completely soluble in the solvent [reagent 1)] when made up as the leafing test vehicle [reagent 3) below] without re-precipitation after standing for 24 h at 20 ± 2 °C.

NOTE In the laboratory tests that were carried out to establish ISO Recommendation R 1247, a coumarone-indene resin, designated B2-TN/75 (obtainable from Verkaufsvereinigung für Teerverwertung, Essen, Germany), was used and found to be satisfactory but other resins may also be found to be satisfactory.

In the UK a resin from British Steel Corporation, Chemical Division, P.O. Box 8, Port Clarence, Middlesbrough, designated coumarone-indene resin M.P. 70–80 °C, has been found to be satisfactory.

3) *leafing test vehicle,* obtained by dissolving 50 g of the resin [reagent 2)] in 100 ml of the white spirit [reagent 1)].

The solution should be prepared slowly at a temperature not exceeding 50 °C and any loss of solvent being made up on a mass basis.

E.3 Procedure

The day before the tests are to be carried out place 5 ml of the leafing test vehicle in the glass cylinder and close with the unslotted cork, then leave overnight.

E.3.1 ***Test portion.*** Remove the top surface layer from the pigment and weigh a quantity of the sample according to Table 3 in the evaporating dish or spouted capsule.

Table 3 — Test portions for leafing test

Type	Class	Mass of test portion, in g[a]
1	a	3.0
	b	2.5
	c	2.0
	d	1.5
2	p	3.0
	q	2.5
	r	2.0

[a] Where the water-covering capacity of the material is such that the result (see **5.2**) is borderline between two classes, two series of leafing power tests should be carried out using the mass of pigment indicated for each of the two classes and the results of both sets of tests should be reported.

E.3.2 ***Determination.*** Measure out 25 ml of the leafing test vehicle and pour about 1 ml (for paste) or 2 ml (for powder) of it on to the pigment. Mix with a small brush until a uniform mixture has been obtained. Repeat with a further similar quantity of vehicle. Mix well and gradually add the remainder of the 25 ml, mixing carefully without introducing air bubbles into the mixture. Immediately transfer an amount to the test tube held at an angle of 45°, such that the height in the test tube is 112 mm, avoiding the formation of bubbles. Adjust the temperature as quickly as possible to 20 ± 2 °C and dip the steel strip immediately to the bottom of the mixture in the tube.

Rotate the steel strip gently for 10 s at about one-quarter turn (90°) per second. The direction of rotation should be reversed once per second and excessive splashing should be avoided. Withdraw the strip at a uniform rate (total time 6 ± 1 s) without touching the sides of the test tube.

NOTE Not more than 2 or 3 drops of the mixture should drain from the strip.

Using the slotted cork to hold the strip, suspend it vertically in the glass cylinder, sheltered from sunlight, the atmosphere in the cylinder being saturated with the vapour from the leafing test vehicle. The strip should not be in contact with the vehicle at the bottom of the cylinder at any time. Allow to stand for 6 min and then measure the length of the leafed area, i.e. the completely covered surface free from cracks or breaks (see Figure 3) and the total immersed length. Carry out two determinations with each successive test portion, taking readings to the nearest millimetre on both sides of the steel strip. Calculate the mean of the readings for each test portion.

E.3.3 ***Calculation.*** Calculate the leafing power, as a percentage, by means of the following formula:

$$\frac{100\,L_1}{L_0}$$

where L_1 = length of the leafed area (mm)

L_0 = total immersed length (mm).

E.3.4 ***Expression of results.*** Report as the percentage leafing power, the mean (rounded to the nearest 1 %) of 3 or 4 results agreeing within ± 3 % absolute.

E.3.5 *Precision*

E.3.5.1 *Repeatability*. The results of tests on the same sample by the same operator with the same apparatus should not be considered suspect unless the extreme values differ by more than 6 % absolute.

E.3.5.2 *Reproducibility*. The results of tests on the same sample in different laboratories should not be considered suspect unless the extreme values differ by more than 10 % absolute.

Appendix F Test for absence of leafing power

F.1 Reagent

The following reagent is required.

1) *Xylene,* commercial quality.

F.2 Procedure

Add 5 g of the sample to 50 ml of the xylene in a suitable container and thoroughly disperse it by vigorous stirring. Allow to settle for 5 min, and then observe the surface of the xylene for flakes of pigment. Absence of flotation of flakes of pigment at the surface is taken as denoting absence of leafing power.

Appendix G Method for the determination of water content

NOTE The use of chlorinated hydrocarbon solvents with aluminium powder or paste can be hazardous if heating is involved, since a rapid exothermic reaction may take place. Experience has shown, however, that chloroform presents no danger when used as described below but precautions should be taken when disposing of waste.

Either of the methods described in BS 2511, Clauses **3** and **4**, may be used subject to the modifications given below.

G.1 Electrometric titration method (BS 2511 Clause 3)

Add to the reaction vessel sufficient dry chloroform[1] to cover the electrodes when inserted, switch on the stirrer and titrate with the Karl Fischer reagent until a large deflection of the galvanometer is obtained, which remains above the half scale reading for at least 30 s. Ignore this titration. Immediately add 10 g of the sample (or such other quantity as will give a titration of not more than 10 ml of the reagent) using chloroform, if necessary, to aid introduction. Allow the material 30 s to disperse and titrate with the Karl Fischer reagent (V ml).

G.2 Visual titration method (BS 2511, Clause 4)

Add to the reaction vessel 10 g of the sample (or such other quantity as will give a titration of not more than 10 ml of reagent) using chloroform if necessary to aid introduction. Add sufficient chloroform to allow complete dispersion of the pigment and titrate with the Karl Fischer reagent (V ml) until the first permanent appearance of a brown colour.

G.2.1 ***Calculation.*** Calculate the water content, as a percentage by mass, by means of the formula:

$$\frac{F \times V}{10m}$$

where F = water equivalent of the Karl Fischer reagent (mg/ml)

V = volume of Karl Fischer reagent (ml)

m = mass of test portion (g).

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[1] Alternatively, if the chloroform used is not dry, allowance should be made for its water content.

Appendix H Methods for the determination of metallic impurities

The following methods are recommended for referee purposes, but other methods may be used by agreement between the purchaser and the vendor.

H.1 Determination of lead (spectrophotometric method)

H.1.1 ***Apparatus.*** Usual laboratory equipment, and in particular:

1) *Filter absorptiometer,* with Cenco green filter cat. No. 87309 B or equivalent, or a spectrophotometer with a blue sensitive phototube.

2) *Cells* of 10 mm or 50 mm.

NOTE All glassware should be free from metals reacting with dithizone. It is advisable to rinse each beaker, funnel or flask with a mixture of dilute ammonia solution, reagent [**H.1.2** 7)] and dithizone solution, 0.02 % [**H.1.2** 8)] the same mixture being used for all articles unless the dithizone turns red.

H.1.2 ***Reagents.*** The reagents used shall be of recognized analytical quality. Water complying with the requirements of BS 3978 shall be used.

1) *Hydrochloric acid,* 6N.

2) *Hydrochloric acid,* 0.5N

3) *Citric acid solution.* Dissolve 200 g of citric acid in water, and dilute to 1 litre. Keep in a borosilicate glass container.

4) *Hydroxylammonium chloride solution.* Dissolve 10 g of hydroxylammonium chloride in water and dilute to 100 ml. It is essential that this solution is freshly prepared just before use.

5) *Potassium cyanide solution,* 100 g/l	Dissolve the required amount of potassium cyanide in water and dilute to 1 litre.
6) *Potassium cyanide solution,* 5 g/l	

NOTE Take the usual precautions in preparing and storing these solutions because of the poisonous character of potassium cyanide. Store in borosilicate glass containers.

7) *Ammonia solution.* Dilute 1 part by volume of ammonia solution, concentrated, 35 % (m/m) with 200 parts of water.

8) *Dithizone solution,* 0.02 g/l. Dissolve 0.200 g of dithizone in 200 ml of chloroform and agitate in a 1 litre separating funnel with 300 ml of dilute ammonia solution [reagent 7)]. Separate the layers and repeat first with 100 ml and then with 50 ml of dilute ammonia solution. Discard the chloroform and filter the combined aqueous layers on a wet paper if necessary. Transfer to a clean 1 litre separating funnel with 200 ml of chloroform and add the 0.5N hydrochloric acid drop by drop with frequent shaking until the colour turns to green and passes into the chloroform layer. Separate and rinse the aqueous layer a few times with 50 ml portions of chloroform that are then added to the main solution. Filter through a dry paper and dilute to 1 litre with pure chloroform. Store in a brown bottle and cover with a 10 mm or 20 mm layer of sulphur dioxide solution (dilute 20 ml of saturated sulphur dioxide solution to 100 ml). Under these conditions, the dithizone solution is stable for several months.

9) *Dithizone solution,* 0.01 g/l. Dilute a given volume of dithizone solution 0.02 g/l [reagent 8) above] with an equal volume of chloroform.

10) *Nitric acid,* concentrated, 70 % (m/m).

11) *Ammonia solution,* concentrated, 35 % (m/m).

12) *Chloroform.*

H.1.3 ***Procedure***

H.1.3.1 *Test portion.* Weigh 0.5 ± 0.001 g of the sample in a 250 ml beaker.

H.1.3.2 *Blank test.* Perform a blank test at the same time as the determination.

H.1.3.3 *Determination.* Dissolve the test portion in 15 ml of 6N hydrochloric acid [**H.1.2** 1)]. When dissolution is complete, add 5 drops of nitric acid [**H.1.2** 10)] and boil for 2 to 3 min. Filter if necessary, washing five times with hot water, combining the filtrate and washings. Adjust the volume to 50 ml, add 15 ml of citric acid solution [**H.1.2** 3)] and neutralize with ammonia solution [**H.1.2** 11)] using litmus as the indicator adding 4 drops in excess. Add 2 ml freshly prepared hydroxylammonium chloride solution [**H.1.2** 4)] and 5 ml of potassium cyanide solution, 100 g/l [**H.1.2** 5)]. Boil for 2 to 3 min and cool in a water trough.

Introduce the solution into a 250 ml separating funnel, add 10 ml of dithizone solution, 0.02 g/l [**H.1.2** 8)] and shake for 30 s. Draw off the chloroform layer into another separating funnel. Continue to shake the aqueous layer with 5 ml portions of dithizone solution 0.01 g/l [**H.1.2** 9)] drawing off the chloroform layer into the second funnel before each new addition, until the chloroform layer remains pure green. Make one further extraction with 5 ml of pure chloroform [**H.1.2** 12)].

Wash the combined chloroform extracts with 25 ml of potassium cyanide solution, 5 g/l [**H.1.2** 6)] and then with 10 ml portions of this solution until successive washings have the same faint amber colour, drawing off the chloroform layer carefully each time into another funnel and discarding the washings.

Wash once in a clean separating funnel, with water, and filter the chloroform layer through a dry paper. Collect the filtrate in a 100 ml one-mark volumetric flask, wash the paper with chloroform and dilute to the mark with chloroform. Mix well, transfer to a 10 mm or 50 mm cell, and read the transmittance with a filter absorptiometer, using the green filter, or read the absorbance with a spectrophotometer at 515 nm (blue-sensitive phototube), with chloroform in the reference cell.

Determine the lead concentration from a calibration graph of absorbance against percentage lead.

H.2 Method for the determination of iron

See BS 1728-8.

H.3 Method for the determination of copper

See BS 1728-5.

H.4 Method for the determination of silicon

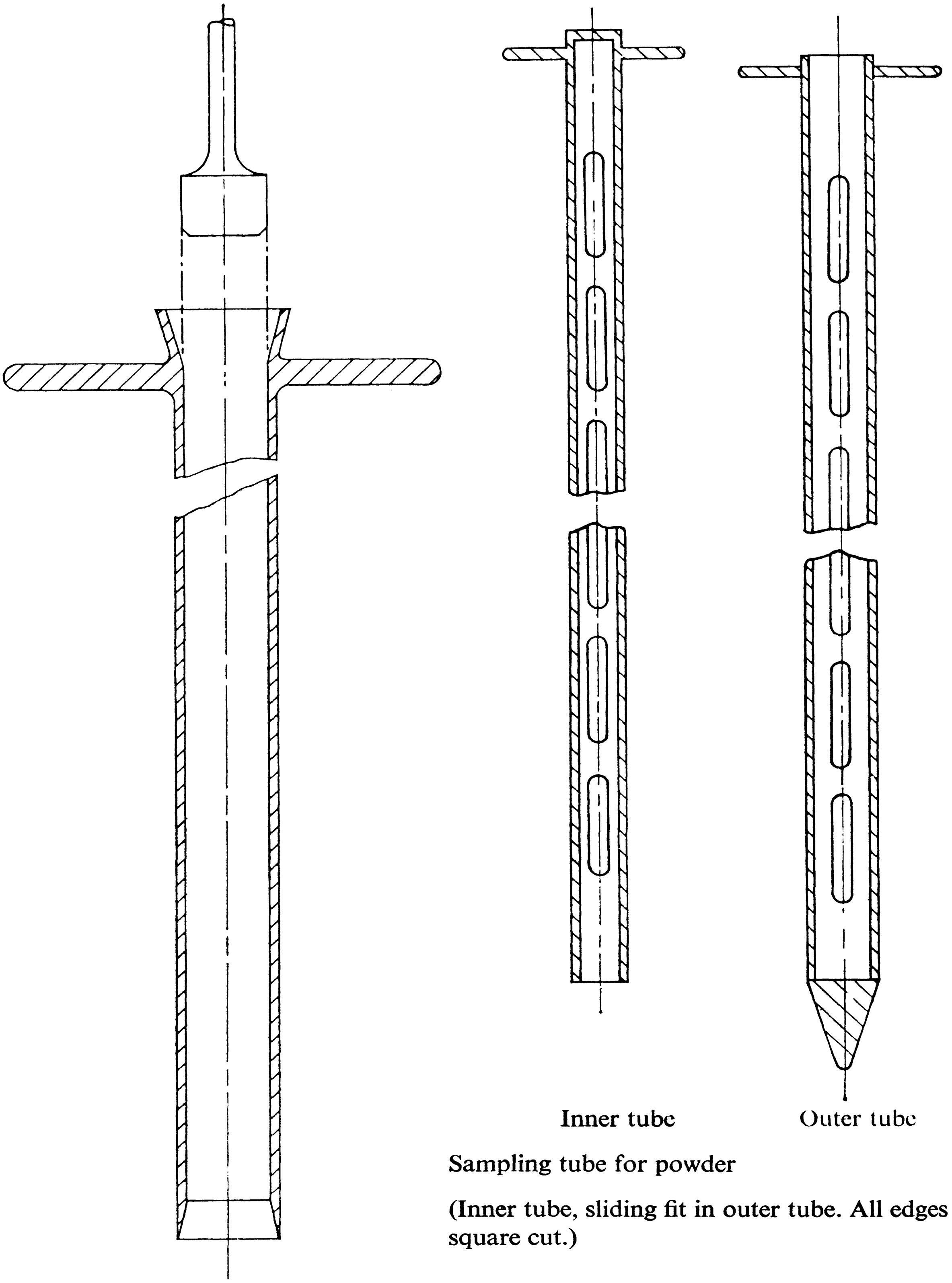
See BS 1728-12.

H.5 Method for the determination of manganese

See BS 1728-10.

H.6 Method for the determination of zinc

See BS 1728-18.



Sampling tube for powder

(Inner tube, sliding fit in outer tube. All edges square cut.)

Sampling tube for paste

Figure 1 — Suitable sampling tubes

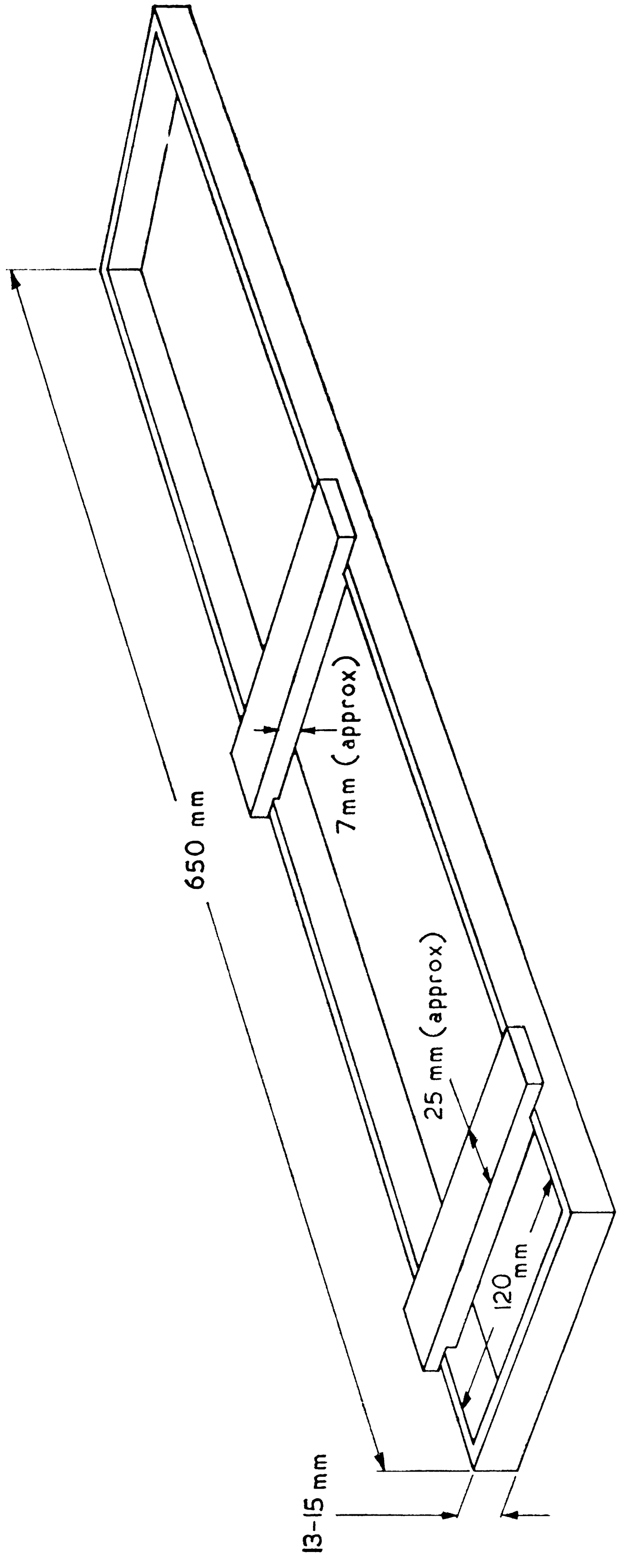


Figure 2 — Trough for determination of water-covering capacity

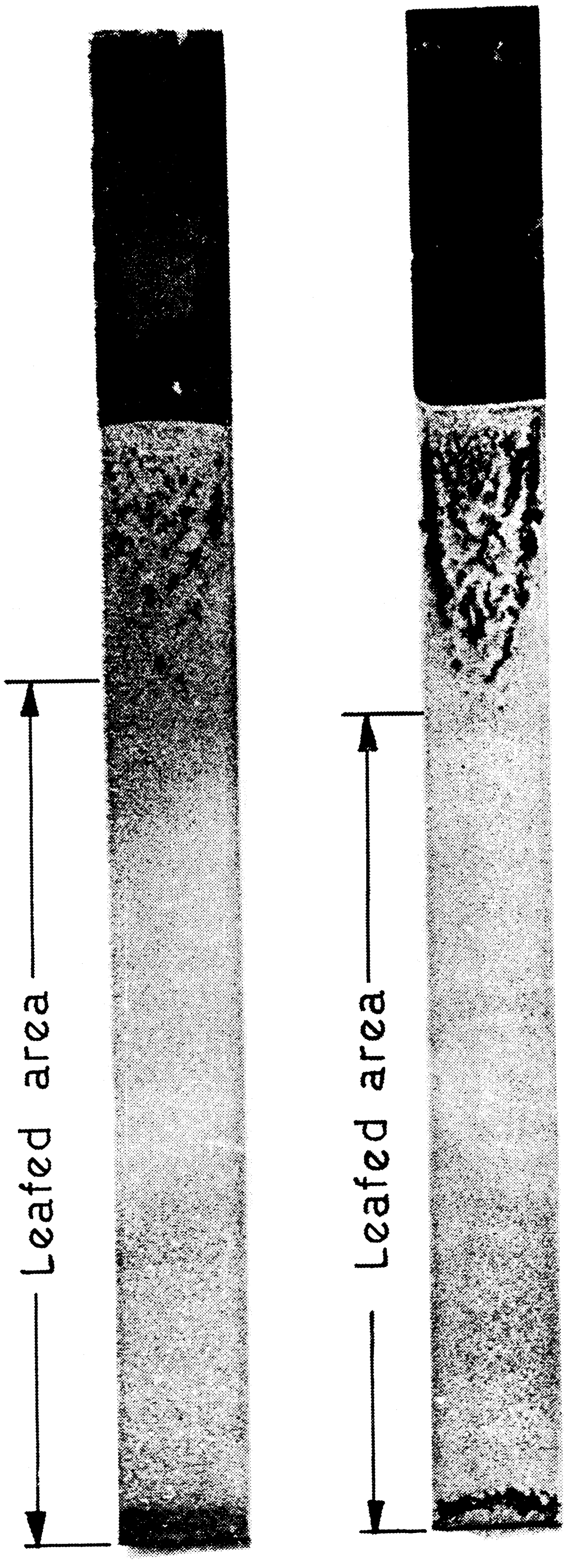


Figure 3 — Examples of measurement of leafed area

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