BRITISH STANDARD

BS 282, 389:
1963

Specifications for

Lead chromes and zinc chromes for paints

Confirmed
February 2011

Co-operating organizations

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

Admiralty*
Air Ministry
Association of British Chemical Manufacturers*
Board of Trade
British Colour Makers' Association*
British Railways, British Transport Commission
Crown Agents for Oversea Governments and Administrations*
D.S.I.R. — Building Research Station*
D.S.I.R. — Laboratory of the Government Chemist
Incorporated Institute of British Decorators*
Lead Oxide Convention*
London County Council
Ministry of Aviation
Ministry of Public Building and Works*
National Federation of Builders' and Plumbers' Merchants
Oil & Colour Chemists Association*
Paint Manufacturers and Allied Trades Association*
Paint Manufacturers Joint Executive Council*
Post Office
Research Association of British Paint, Colour & Varnish Manufacturers*
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Titanium Pigment Manufacturers' Technical Committee*
War Office*
White Lead Convention*
Zinc Development Association*
Zinc Pigment Development Association

The Government departments 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:

British Electrical & Allied Manufacturers Association
National Federation of Master Painters & Decorators of England & Wales

Amendments issued since publication

Amd. No.	Date	Comments

This British Standard, having been approved by the Pigments, Paints and Varnishes Industry Standards Committee and endorsed by the Chairman of the Chemical Divisional Council, was published under the authority of the General Council on 6 February 1963

BS 282 First published May 1927
BS 389 First published March 1929
First revision combining BS 282 and BS 389 November 1938
Second revision April 1953
Third revision February 1963

The following BSI references relate to the work on this standard:
Committee references PVC/1, PVC/1/8
Draft for comment AA(PVC)667

ISBN 0 580 34468 1

Contents

Foreword

This standard makes reference to the following British Standards:

BS 242, *Refined linseed oil for paints.*

BS 245, *White spirit.*

BS 254, *Zinc oxide (Types 1 and 2).*

BS 410, *Test sieves.*

BS 612, *Nessler cylinders.*

BS 846, *Burettes and bulb burettes.*

BS 950, *Artificial daylight fittings for colour matching.*

BS 1752, *Sintered disk filters for laboratory use.*

BS 1792, *One-mark volumetric flasks.*

BS 1795, *Extenders for paints.*

BS 1851, *Titanium dioxide pigments for paints.*

When the British Standard for lead chromes, BS 282, was first issued, the main criterion of value of a lead chrome was that it should consist entirely of water-insoluble compounds of lead. In 1953, when the specification was last revised, the position was substantially the same, for although newer types of lead chromes had by then appeared their use was still on a small scale. Since then, however, there has been an increasing use of so-called "treated chromes" in which considerably improved properties, particularly greater resistance to darkening under the action of light, have been achieved by surface treatment of chromes under carefully controlled conditions. Such chromes cannot consist wholly of water-insoluble compounds of lead and hence do not conform to the requirements of BS 282:1953. Because of the nature and variety of the treating agents to lay are used (and those which may be used in the future) it is impracticable which down a composition specification which would not be undesirably restrictive. In any case such a specification would be of no assistance to users of these pigments in assessing their value in actual performance.

The specification has, therefore, been rewritten so as to cover, with safeguards, both treated and untreated types of chromes. The method for the determination of aluminium given in BS 282:1953 is no longer required and has been omitted.

The changes in the case of the British Standard for zinc chromes, BS 389, are almost entirely of an editorial character.

A separate specification has been written for each of the three types of zinc chrome, which, it is thought, will be clearer and more convenient than the arrangement followed in BS 389:1953. The test for matter insoluble in ammonia solution in the 1953 specification has been replaced by a test for insolubility in ammoniacal ammonium chloride solution, which gives more consistent results. In the case of the Type 3 material, an upper limit has been imposed on the metallic zinc content.

The test methods in the Appendices have undergone editorial rearrangement to bring them into line with current practice in British Standards.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 15 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.

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Lead chromes

1 Scope and description

This British Standard applies to lead chromes for use in paints, both those consisting wholly of water-insoluble compounds of lead and those containing additions introduced specifically to improve certain pigmentary properties. It is not intended to apply to mixtures of lead chromes with other separately prepared lead compounds other than chromes nor to mixtures with extenders or diluents. The material shall be in the form of a dry soft powder or in such a condition that it may be readily reduced thereto by crushing under a palette knife, without necessitating any grinding action.

2 Composition

The purchaser may require the vendor to declare whether the lead chrome is of an untreated or treated type.

In the former case it shall consist wholly of water-insoluble compounds of lead, except as provided by Clause **7**.

In the case of treated chromes the purchaser may require the vendor to state the nature of the improved properties which are claimed to result from the treatment and may require the vendor to state the minimum percentage content of lead determined in the manner described in Appendix K.

In all cases the material shall be free from organic colours and lakes when tested in the manner described in Appendix A.

3 Sample

For the purpose of examination representative samples shall be taken at random from not less than 10 per cent of the original and previously unopened containers. The material shall be thoroughly mixed and shall then be divided into triplicate samples weighing not less than 0.45 kg (1 lb) each. These shall be packed in clean, dry, air-tight, non-absorbent, opaque or dark glass containers on which the sample has no action. The containers shall be of such a size that they are nearly filled by the sample. Each sample container so filled shall be sealed and shall be marked with full details and date of sampling.

4 Agreed sample

Except where otherwise agreed between the purchaser and the vendor, the agreed sample referred to in Clauses **8**, **9**, **10** and **11** shall be one and the same sample and shall comply in all respects with the other requirements of this specification. The sample shall weigh not less than 0.45 kg (1 lb) and shall be packed in the manner specified in Clause **3**.

5 Residue on sieve

The material shall not leave more than 0.5 per cent by weight of residue when tested in the manner described in Appendix B.

6 Matter volatile at 100 °C

The loss in weight, on heating the material in the manner described in Appendix C, shall not exceed 1.0 per cent.

7 Matter soluble in water

The material shall not yield more than 1.0 per cent by weight of matter soluble in water when tested in the manner described in Appendix D.

The acidity or alkalinity of the aqueous extract, when determined in the manner described in Appendix D, shall not exceed the equivalent of 0.1 per cent by weight of sulphuric acid, H_2SO_4, or sodium carbonate, Na_2CO_2 calculated on the material.

8 Oil absorption value

The oil absorption value of the material, when determined in the manner described in Appendix E, shall be within ± 10 per cent of the oil absorption value of the agreed sample.

9 Colour

The colour of the material shall closely match that of the agreed sample, when compared in the manner described in Appendix F.

10 Stability of colour to light

The colour of the material shall not change to a greater extent than that of the agreed sample, when compared in the manner described in Appendix G.

11 Staining power and colour on reduction

The staining power of the material shall not be inferior to, and the colour on reduction shall closely match, that of the agreed sample, when compared in the manner described in Appendix H.

12 Soluble lead

If it is required to determine the soluble lead content of the material, this shall be done by the method described in Appendix J.

Zinc chrome, Type 1

1 Scope and description

This British Standard applies to zinc chromes which include in their composition chromates of alkali metals and are suitable for use in paints. The material shall be in the form of a dry soft powder or in such condition that it may be readily reduced thereto by crushing under a palette knife, without necessitating any grinding action.

2 Composition

The material shall be free from organic colours and lakes when tested by procedures b) and c) as described in Appendix A. After drying in the manner described in Appendix C, it shall consist essentially of basic zinc chromate with chromates of alkali metals.

3 Sample

For the purpose of examination representative samples shall be taken at random from not less than 10 per cent of the original and previously unopened containers. The material shall be thoroughly mixed and shall then be divided into triplicate samples weighing not less than 0.45 kg (1 lb) each. These shall be packed in clean, dry, air-tight, non-absorbent, opaque or dark glass containers on which the sample has no action. The containers shall be of such a size that they are nearly filled by the sample. Each sample container so filled shall be sealed and shall be marked with full details and date of sampling.

4 Agreed sample

Except where otherwise agreed between the purchaser and the vendor, the agreed sample referred to in Clauses **7**, **8** and **9** shall be one and the same sample and shall comply in all other respects with the requirements of this specification. The sample shall weigh not less than 0.45 kg (1 lb) and shall be packed in the manner specified in Clause **3**.

5 Residue on sieve

The material shall not leave more than 0.5 per cent of residue when tested in the manner described in Appendix B.

6 Matter volatile at 100 °C

The loss in weight, on heating the material in the manner described in Appendix C shall not exceed 1.5 per cent.

7 Oil absorption value

The oil absorption value of the material, determined in the manner described in Appendix E, shall be within ± 10 per cent of the oil absorption value of the agreed sample.

8 Colour

The colour of the material shall closely match that of the agreed sample, when compared in the manner described in Appendix F.

9 Staining power and colour on reduction

The staining power of the material shall not be inferior to, and the colour on reduction shall closely match, that of the agreed sample, when compared in the manner described in Appendix H.

10 Freedom from impurities

The material shall not contain more than 0.5 per cent by weight of matter insoluble in ammoniacal ammonium chloride solution, determined by the method described in Appendix L.

Zinc chrome, Type 2

1 Scope and description

This British Standard applies to basic zinc potassium chromate of approximate composition K_2CrO_4, $3ZnCrO_4$, $Zn(OH)_2$, suitable for use in paints. The material shall be in the form of a dry soft powder or in such condition that it may be readily reduced thereto by crushing under a palette knife, without necessitating any grinding action.

2 Composition

The material shall be free from organic colours and lakes when tested by the procedures b) and c) described in Appendix A. After drying in the manner described in Appendix C, it shall consist essentially of basic potassium zinc chromate and shall approximate in composition to the formula K_2CrO_4, $3ZnCrO_4$, $Zn(OH)_2$.

a) *Zinc content*. The zinc content of the material shall be not less than 36.5 per cent[1] and not more than 40 per cent[2] by weight, calculated as zinc oxide, ZnO, and determined by the method described in Appendix O.

b) *Alkali metal content*. The alkali metal content of the material shall be not less than 10 per cent and not more than 12 per cent by weight, calculated as potassium oxide, K_2O, and determined by the method described in Appendix P.

c) *Chromate content*. The chromate content of the material shall be not less than 43 per cent by weight, calculated as chromium trioxide, CrO_3, and determined by the method described in Appendix Q.

3 Sample

For the purpose of examination representative samples shall be taken at random from not less than 10 per cent of the original and previously unopened containers. The material shall be thoroughly mixed and shall then be divided into triplicate samples weighing not less than 0.45 kg (1 lb) each. These shall be packed in clean, dry, air-tight, non-absorbent, opaque or dark glass containers on which the sample has no action. The containers shall be of such a size that they are nearly filled by the sample. Each sample container so filled shall be sealed and shall be marked with full details and date of sampling.

4 Agreed sample

Except where otherwise agreed between the purchaser and the vendor, the agreed sample referred to in Clause **7** shall comply in all other respects with the requirements of this specification. The sample shall weigh not less than 0.45 kg (1 lb) and shall be packed in the manner specified in Clause **3**.

5 Residue on sieve

The material shall not leave more than 0.5 per cent by weight of residue when tested in the manner described in Appendix B.

6 Matter volatile at 100 °C

The loss in weight, on heating the material in the manner described in Appendix C, shall not exceed 1.0 per cent.

7 Oil absorption value

The oil absorption value of the material, determined in the manner described in Appendix E, shall be within ± 10 per cent of the oil absorption value of the agreed sample.

8 Freedom from impurities

a) *Matter insoluble in ammoniacal ammonium chloride solution*. The material shall not contain more than 0.5 per cent by weight of matter insoluble in ammoniacal ammonium chloride solution, determined by the method described in Appendix L.

b) *Water-soluble sulphates, chlorides and nitrates*. The material shall not contain more than the following percentages by weight of each of the following constituents when determined by the method described in Appendix M.

Sulphates, calculated as SO_4, 0.1 per cent

Chlorides, calculated as Cl, 0.1 per cent

Nitrates, calculated as NO_3, 0.1 per cent

9 Water-soluble chromates

The material shall contain not less than 0.25 per cent and not more than 0.50 per cent by weight of water-soluble chromates, calculated as chromium trioxide, CrO_3, and determined by the method described in Appendix N.

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[1] Equivalent to 28.5 per cent metallic zinc.

[2] Equivalent to 32.1 per cent metallic zinc.

Zinc chrome, Type 3

1 Scope and description

This British Standard applies to the basic zinc chromate commonly known as zinc tetroxychromate, suitable for use in paints. The material shall be in the form of a dry soft powder or in such condition that it may be readily reduced thereto by crushing under a palette knife, without necessitating any grinding action.

2 Composition

The material shall be free from organic colours and lakes when tested by the procedures b) and c) described in Appendix A. After drying in the manner described in Appendix C, it shall consist of a basic zinc chromate free from alkali metal and approximating in composition to $ZnCrO_4.4Zn(OH)_2$.

a) *Zinc content*. The total zinc content of the material shall be not less than 68.5 per cent[3] and not more than 72.0 per cent[4] by weight, calculated as zinc oxide, ZnO, and determined by the method described in Appendix O.

b) *Chromate content*. The chromate content of the material shall be not less than 17.0 per cent by weight, calculated as chromium trioxide, CrO_3, and determined by the method described in Appendix Q.

3 Sample

For the purpose of examination representative samples shall be taken at random from not less than 10 per cent of the original and previously unopened containers. The material shall be thoroughly mixed and shall then be divided into triplicate samples weighing not less than 0.45 kg (1 lb) each. These shall be packed in clean, dry, air-tight, non-absorbent, opaque or dark glass containers on which the sample has no action. The containers shall be of such a size that they are nearly filled by the sample. Each sample container so filled shall be sealed and shall be marked with full details and date of sampling.

4 Agreed sample

Except where otherwise agreed between the purchaser and the vendor, the agreed sample referred to in Clause **7** shall comply in all other respects with the requirements of this specification. The sample shall weigh not less than 0.45 kg (1 lb) and shall be packed in the manner specified in Clause **3**.

5 Residue on sieve

The material shall not leave more than 0.5 per cent of residue when tested in the manner described in Appendix B.

6 Matter volatile at 100 °C

The loss in weight, on heating the material in the manner described in Appendix C, shall not exceed 1.0 per cent.

7 Oil absorption value

The oil absorption value of the material, when determined in the manner described in Appendix E, shall be within ± 10 per cent of the oil absorption value of the agreed sample.

8 Freedom from impurities

a) *Matter insoluble in ammoniacal ammonium chloride solution*. The material shall not contain more than 0.5 per cent by weight of matter insoluble in ammoniacal ammonium chloride solution, determined by the method described in Appendix L.

b) *Water-soluble sulphates, chlorides and nitrates*. The material shall not contain more than the following percentage by weight of each of the following constituents, when determined by the method described in Appendix M.

Sulphates, calculated as SO_4, 0.1 per cent

Chlorides, calculated as Cl, 0.1 per cent

Nitrates, calculated as NO_3, 0.1 per cent

c) *Total water-soluble matter*. The material shall not contain more than 0.5 per cent of matter soluble in water, when determined by the method described in Appendix M.

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[3] Equivalent to 55 per cent metallic zinc.

[4] Equivalent to 57.8 per cent metallic zinc.

Appendix A Method for the detection of the presence of organic colours and lakes

A.1 Reagents

a) *Ethanol*[5] (95 *per cent v/v) and chloroform*, a mixture of equal parts (v/v).

b) *Dioxan*.

A.2 Procedure

a) Examine the water extract obtained as described in Appendix D for colours which bleed in water.

b) Boil 2 g of the test sample for two minutes with 25 ml of the ethanolchloroform mixture. Allow the pigment to settle, decant the liquor through a dry filter paper and examine the filtrate.

c) Boil the pigment residue from b) for two minutes with 25 ml of dioxan. Allow the pigment to settle, decant the liquor through a dry filter paper and examine the filtrate.

A.3 Conclusion

Organic colours and lakes are assumed to be absent if the water extract a) and the filtrates b) and c) above are colourless.

Appendix B Method for the determination of residue on sieve

Method 1. Using the automatic muller

B.1 Apparatus

a) *Automatic muller*.

b) *Steel palette knife*. A suitable knife is one having a tapered blade 140–150 mm long, 20–25 mm wide at its widest point, and not less than 12.5 mm wide at its narrowest point.

c) *Sieve*[6] $1\frac{1}{2}$ inches (3.75 cm) in diameter, previously tared, provided with a 240 BS mesh.

B.2 Materials

a) *Medium:* Linseed stand oil. A suitable stand oil is one[7] having a viscosity of 25 poise at 25 °C and an acid value of 3.5 mg KOH per gramme of oil.

b) *White spirit*, complying with BS 245[8].

B.3 Procedure

Weigh out 5 g of the test sample and mix it with 2 ml of the linseed stand oil on the lower plate of the automatic muller using the palette knife. (The oil may be measured from a graduated hypodermic syringe, or 1.85 g weighed on a flat glass plate and transferred.)

When all the colour particles are wetted with the oil, spread the mixture in a roughly circular patch in the centre of the lower plate. Remove the portion of colour/oil mixture which adheres to the palette knife by drawing both sides of the blade, with pressure, across the surface of the upper plate.

Close the muller plates and apply pressure of 50 lbf.

After 100 revolutions, open the muller and scrape the paste from both plates by means of the palette knife. Mix the paste and again spread it in a roughly circular patch in the centre of the lower plate and reclean the knife by drawing both sides across the surface of the upper plate.

Repeat this procedure so that the paste is ground for a total of 400 revolutions and then scrape the paste together and transfer it to a 600 ml squat form beaker with a lip.

Without cleaning the muller plates or the palette knife, repeat this whole procedure 3 times more, to make 4 quantities of paste in all.

Gradually thin this paste with 2 additions, each of 4 ml of linseed stand oil, and stir to uniformity by means of a glass rod after each addition.

Then slowly add 250 ml of the white spirit, stirring well to ensure uniformity during the whole addition.

Pour the resulting pigment suspension through the tared 240 mesh sieve. Should the sieve fill up, cease the pouring and tap the sides of the sieve to assist the passage of the liquid through the sieve.

When the suspension has passed through the sieve, rinse the beaker into the sieve and wash the residue on the sieve, using a jet of white spirit from a handoperated polythene wash bottle, until the washings are free from pigment (250 ml of white spirit will usually be found sufficient).

Allow the sieve to drain and then dry in an oven at a temperature of 100 ± 2 °C for 1 hour, cool and weigh.

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[5] Ethanol may be replaced by industrial methylated spirits (ethanol containing 5 per cent by volume of approved wood naphtha) of equivalent strength. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations 1952 (S.I. 1952 No. 2230).

[6] BS 410, *"Test sieves"*.

[7] This grade is commonly known in the trade as "Tint" litho varnish.

[8] BS 245, *"White spirit"*.

B.4 Calculation

Residue on sieve, per cent by weight = $\frac{100\ W_1}{W}$

where W_1 = weight, in grammes, of residue

W = weight, in grammes, of sample taken

Method 2. Using the palette knife

NOTE This method is less satisfactory than Method 1 because the amount of work done on the mixture cannot be so accurately controlled. It is only to be used when the automatic muller is not available.

B.5 Apparatus

a) *Ground-glass* or *marble plate*, at least 30 × 40 cm.

b) *Steel palette knife*. A suitable knife is one having a tapered blade 140–150 mm long, 20–25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

c) *Sieve*[9] 1$\frac{1}{2}$ inches (3.75 cm) in diameter, previously tared, provided with a 240 BS mesh.

B.6 Materials

a) *Acid-refined linseed oil*, complying with BS 242[10] having an acid value of 7.5 to 8.5 mg of KOH per gramme.

b) *White spirit*, complying with BS 245[11].

B.7 Procedure

Weigh accurately about 20 g of the test sample on to the ground-glass or marble plate. Add the linseed oil to the sample drop by drop, mixing continuously with the palette knife until the pigment is thoroughly wetted and a smooth thick paste is obtained.

Transfer the paste by means of the palette knife to a 600 ml squat form beaker with lip and wash the last traces of the sample paste from the plate into the beaker, using a stream of white spirit from a hand-operated polythene wash bottle. Thin the paste in the beaker with 250 ml of the white spirit, stirring well to ensure uniformity during the whole addition. Pour the resulting pigment suspension on to the tared 240 mesh sieve.

Wash the residue remaining on the sieve with white spirit (not under pressure) and gently brush with a soft camel-hair pencil until the washings are clear. Dry the residue and sieve in an oven at 100 ± 2 °C for one hour, cool and weigh.

B.8 Calculation

Residue on sieve, per cent by weight = $\frac{100\ W_1}{W}$

where W_1 = weight, in grammes, of residue

and W = weight, in grammes, of sample taken.

Appendix C Method for the determination of matter volatile at 100 °C

C.1 Procedure

Weigh, to the nearest milligramme, about 10 g of the test sample and spread it as a uniform layer over the bottom of a dry, tared, wide-mouthed, short weighing bottle provided with a ground-glass stopper. Heat the bottle and contents, with the stopper removed, in an oven at 100 ± 2 °C for one hour. Cool the bottle in a desiccator, insert the stopper and weigh to the nearest milligramme, momentarily releasing the stopper immediately before weighing.

C.2 Calculation

Matter volatile at 100 °C, per cent

by weight = $\frac{100(W - W_1)}{W}$

where W = original weight, in grammes, of the sample

and W_1 = final weight, in grammes, of the sample.

Appendix D Method for the determination of matter soluble in water, and acidity or alkalinity of the water extract

D.1 Apparatus

a) *One-mark volumetric flask*,[12] 250 ml.

b) *Evaporating dish*, flat bottomed, of glass, glazed porcelain or silica.

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[9] BS 410, *"Test sieves"*.

[10] BS 242, *"Refined linseed oil for paints"*.

[11] BS 245, *"White spirit"*.

[12] BS 1792, *"One-mark volumetric flasks"*.

D.2 Reagents

All reagents used shall be of recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.

a) *Ethanol*[13]. 95 per cent (v/v).

b) *Sulphuric acid*. 0.01N solution, accurately standardized.

c) *Sodium carbonate*. 0.01N solution, accurately standardized.

d) *Bromothymol blue indicator*. 0.05 per cent aqueous solution.

D.3 Procedure

a) Weigh, to the nearest milligramme, 10 g of the test sample in powder form, into a beaker and thoroughly wet with ethanol[13] (5 ml is usually sufficient). Add 200 ml of neutral distilled water, stir and boil for five minutes. Cool rapidly to room temperature, transfer to the 250 ml one-mark volumetric flask and dilute to the mark with neutral distilled water. Mix thoroughly by shaking and inversion and filter through a fine-textured filter paper. Reject the first 25 ml of the filtrate and evaporate 100 ml of the succeeding, perfectly clear, filtrate to dryness in the tared, flat-bottomed, glass, glazed porcelain or silica dish on a water bath or in an oven at 100 °C.

Dry the residue to constant weight in an oven at 100 ± 2 °C.

b) To 50 ml of the perfectly clear filtrate obtained in procedure a) above, add 5 ml of the bromothymol blue indicator.

If the solution is blue (alkaline), titrate it with the acid until the blue colour disappears.

If the solution is yellow (acid), titrate it with the alkali until the first permanent blue colour appears.

D.4 Calculation

Water-soluble matter, per cent,

$$\text{by weight} = \frac{250\,W_1}{W}$$

where W_1 = weight, in grammes, of residue

and W = weight, in grammes, of sample taken.

Alkalinity, per cent, expressed

$$\text{as } Na_2CO_3 = \frac{0.265\,T_1}{W}$$

Acidity, per cent, expressed

$$\text{as } H_2SO_4 = \frac{0.245\,T_2}{W}$$

where W = weight, in grammes, of sample taken.

T_1 = volume, in millilitres, of 0.01N sulphuric acid solution required.

T_2 = volume, in millilitres, of 0.01N sodium carbonate solution required.

Appendix E Method for the determination of oil absorption value

E.1 Apparatus

a) *Ground-glass* or *marble plate*, at least 30 × 40 cm.

b) *Steel palette knife*. A suitable knife is one having a tapered blade 140–150 mm long, 20–25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

c) *Burette*, 25 ml, sub-divided at each 0.1 ml, complying with the requirements of BS 846[14].

E.2 Material required

Acid-refined linseed oil, complying with BS 242[15], having an acid value of 7.5 to 8.5 mg of KOH per gramme.

E.3 Procedure

Weigh out 20 g of the test sample and place the material on the ground glass or marble plate. Then add the linseed oil slowly, 4–5 drops at a time, from the burette. After each addition rub the oil into the pigment with the palette knife, and continue the addition of oil at this rate until conglomerates of oil and pigments are formed. From this point, add the oil one drop at a time and follow each addition of oil by thorough rubbing with the palette knife. Cease the addition of oil when a stiff paste of smooth consistency has been formed. This paste should just spread without cracking or crumbling. Read the burette and note the quantity of oil used. The time taken for the complete operation must be between twenty and twenty-five minutes and during this time, the whole pigment mass should be manipulated with maximum effort by the operator[16].

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[13] Ethanol may be replaced by industrial methylated spirits (ethanol containing 5 per cent by volume of approved wood naphtha) of equivalent strength. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1952 (S.I. 1952 No. 2230).

[14] BS 846, *"Burettes and bulb burettes"*.

[15] BS 242, *"Refined linseed oil for paints"*.

[16] In order to determine the rate of addition of oil which complies with the above conditions, make a preliminary trial run.

The oil absorption of the agreed sample shall be determined in the same manner by the same operator immediately afterwards.

E.4 Calculation

a) on a volume/weight basis oil absorption = $\frac{100T}{W}$

b) on a weight/weight basis oil absorption = $\frac{93T}{W}$

where T = volume, in millilitres, of oil required

and W = weight, in grammes, of sample taken.

Appendix F Method for the comparison of colour

F.1 Apparatus

a) *Ground-glass* or *marble plate*, at least 30 × 40 cm.

b) *Steel palette knife or glass muller*. A suitable knife is one having a tapered blade 140–150 mm long, 20–25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point; a suitable muller is one 70–75 mm in diameter.

c) *Glass slide*, clear and colourless, 150 × 50 mm, or other suitable size.

d) *Burette* with a delivery so that 35 drops = 1 ml of refined linseed oil.

F.2 Material required

Acid-refined linseed oil, complying with BS 242[17] having an acid value of 7.5–8.5 mg of KOH per gramme, or other agreed medium.

F.3 Method

Weigh to the nearest milligramme 1 g of the test sample on a suitable balance and transfer to the glass plate. Collect 15 drops of oil on the end of the palette knife, or sufficient to obtain a suitable grinding consistency, and mix with the pigment on the glass plate. When the pigment has become uniformly wetted with oil commence rubbing with the palette knife or muller using a backwards and forwards motion. Rubbing should spread the paint over an area approximately 200 × 75 mm.

After 100 rubs (one rub consists of one forward plus one backward stroke of the knife), scrape the pigment/oil mixture into a heap at the centre of the plate, making sure that any unground pigment is removed from the knife blade outside the area which has been in contact with the plate. Repeat the rubbing out operation using a further 100 rubs after which add a further 5 drops of oil. Mix well until uniform and transfer to one corner of the plate until the agreed sample is rubbed out.

Prepare a rub-out of the agreed sample in an exactly similar manner using the same grinding consistency as that used in treating the test sample even though this may involve adding a larger or smaller number of drops of oil than were used with the test sample.

Compare the colour of the test sample with the agreed sample by spreading the two mixtures so prepared in the same direction on the glass slide in opaque strips of not less than 25 mm wide with touching edges not less than 40 mm long. Make the colour comparison by examining the strips in diffused daylight through the glass, and on the surface, immediately after application. Where good daylight is not available make the comparison in artificial daylight from a source complying with BS 950[18].

Appendix G Method for the comparison of stability to light

(by reference to agreed sample.)

G.1 Apparatus

Carbon arc[19] or *tungsten filament lamps*[20] unless it has been agreed that daylight is to be used.

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[17] BS 242, *"Refined linseed oil for paints"*.

[18] BS 950, *"Artificial daylight fittings for colour matching"*.

[19] A satisfactory carbon arc is the vertical type burning solid pure carbons of 13 mm diameter and consuming approximately 13 amps with a voltage drop across the carbons of 90–100 volts. The arc is enclosed in a cylindrical globe of clear, colourless glass not less than 180 mm in diameter. The painted specimens are fixed vertically at a distance of 280 mm from the arc, so that a line drawn from the centre of any paint film to the mean centre of the arc makes an angle of not more than 15° with the horizontal. The lower carbon is raised before 38 mm has been consumed. The globe is cleaned during the exposure at intervals of not more than twenty four hours.

[20] If tungsten filament lamps are used, a convenient arrangement consists of four 150 watt lamps, mounted at the corners of a 300 mm square with their filaments in a plane parallel to and 185 mm from a base board on which the test portions are placed. A sufficiently uniform intensity of illumination is produced within the 300 mm square.

G.2 Material required

Either a 1 per cent aqueous solution of gum arabic or a 0.5 per cent aqueous solution of methylcellulose or ethylcellulose.

G.3 Procedure

Rub the test sample down to a smooth mixture with the medium; approximately equal proportions by weight of pigment and medium will be found satisfactory. Thoroughly incorporate sufficient additional medium in the mixture so as to bring it to such a condition that it can be evenly spread on a smooth surface.

Treat the agreed sample similarly and at the same time so that the final mixtures so prepared shall each contain the same proportion of pigment to medium. By means of a brush apply an even coating on non-absorbent paper of each mixture separately and allow them to dry in the dark, then cover a part of each painting by a closely fitting strip of material opaque to light and expose the two painted portions side by side to a suitable light source in an atmosphere which is free from hydrogen sulphide and sulphur dioxide and under conditions such that each painting is evenly illuminated with the same intensity of light.

From time to time remove the paintings from the light source and, after removing the opaque covering strip, assess the amount of change which has occurred in the exposed portion of each painting compared with the covered portion. If there is little change in both paintings, replace the covering strips and re-expose both paintings simultaneously until it is found that at least one of them shows a significant change of shade of the exposed portion against its own unexposed control portion. The comparison of the relative degree of change of one painting against the other can then be made.

By agreement between purchaser and vendor the paintings may be exposed to daylight or a carbon arc or a battery of tungsten filament lamps.

Appendix H Method for the comparison of staining power and colour on reduction

Method 1. Using the palette knife or hand muller

H.1 Apparatus

a) *Ground-glass* or *marble plate* at least 30 × 40 cm.

b) *Steel palette knife or glass muller*. A suitable knife is one having a tapered blade 140–150 mm long, 20–25 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point; a suitable muller is one 70–75 mm in diameter.

c) *Glass slide*, clear and colourless, 150 × 50 mm or other suitable size.

d) *Burette* with a delivery so that 35 drops = 1 ml of refined linseed oil.

H.2 Material required

a) *Acid-refined linseed oil*, complying with BS 242[21], having an acid value of 7.5–8.5 mg of KOH per gramme, or other agreed medium.

b) *Reducing pigment*, either zinc oxide, type 1, complying with BS 254[22]; or a 1 : 3 mixture of titanium dioxide, Type R, complying with BS 1851[23] and barytes Grade 1 complying with BS 1795[24]; or other agreed pigment.

NOTE It is recommended that when titanium dioxide is used as the reducing pigment, a glass muller should be employed for dispersing.

H.3 Procedure

Weigh to the nearest milligramme 0.1 g of the test sample and transfer to the glass plate.
Collect 3 drops of oil from the burette on the end of the palette knife and mix with the pigment on the plate. Spread to a thin film and commence rubbing with the palette knife or muller using a backwards and forwards motion. Rubbing should spread the paint over an area approximately 200 × 75 mm.
After 100 rubs (one rub consists of one forward plus one backward stroke of the knife) scrape the pigment up on the palette knife and spread out to a thin film. Complete the initial dispersion by giving the mixture another 100 rubs on the plate.

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[21] BS 242, *"Refined linseed oil for paints"*.
[22] BS 254, *"Zinc oxide (Types 1 and 2) for paints"*.
[23] BS 1851, *"Titanium dioxide pigments for paints"*.
[24] BS 1795, *"Extenders for paints"*.

Weigh out 2.0 g of zinc oxide (or 2.0 g of the titanium dioxide mixture or the appropriate amount of agreed pigment), place on the plate, add 20 drops of oil from the burette, using the same palette knife or muller (without cleaning it) and mix to uniformity. Mix the dispersed pigment with the reducing pigment/linseed oil paste by the application of a further 100 rubs over the same portion of the plate which was used for rubbing the colour. Apply a small test portion (area about 1 cm^2) to the glass slide. Apply a further 100 rubs to the mixture and apply a second small test portion side by side with the previous test portion. It no increase in colour intensity is shown, grinding is complete; if a change is shown apply a further 100 rubs, test again and repeat until no further colour development takes place.

Scrape the mixture so prepared to one corner of the plate and clean the rest of the plate thoroughly. Rub out the agreed sample in an exactly similar way using precisely the same quantity of oil.

Compare the test sample with the agreed sample by spreading the two mixtures in the same direction on the glass slide in opaque strips not less than 25 mm wide with touching edges not less than 40 mm long. Examine for colour and strength by examining the two strips in diffused daylight through the glass, and on the surface, immediately after application. Where good daylight is not available, use artificial daylight from a source complying with BS 950[25].

Method 2. Using the automatic muller

H.4 Apparatus

a) *Automatic muller*.

b) *Steel palette knife*. A suitable knife is one having a tapered blade 140–150 mm long, 120–125 mm wide at its widest point and not less than 12.5 mm wide at its narrowest point.

c) *Glass plate*, clear and colourless, 150 × 50 mm, or other suitable size.

H.5 Materials required

a) *Acid-refined linseed oil*, complying with BS 242[26] having an acid value of 7.5–8.5 mg of KOH per gramme; or other agreed medium.

b) *Reducing pigment*, either zinc oxide, type 1, complying with BS 254[27]; or a 1 : 3 mixture of titanium dioxide, Type R, complying with BS 1851[28] and barytes Grade 1 complying with BS 1795[29]; or other agreed pigment.

H.6 Procedure

Weigh 0.1 g of the test sample and 2.0 g of zinc oxide (or 2.0 of titanium dioxide mixture or the appropriate amount of the agreed pigment) into separate suitable receptacles. Transfer the reducing pigment completely to the clean, lower muller plate and add 20 drops of the oil via the knife blade so that, when mixed, a smooth pigment-oil paste is obtained. Transfer the sample completely on to the white paste and incorporate it by gently working with the knife, avoiding any grinding action. Add a further 3 drops of oil.

When the pigments have been wetted as described, spread the paste in a circle of approximately two inches diameter around the centre of the lower muller plate and clean the palette knife by drawing it across the top plate. Close the muller plates, apply a pressure of 150 lbf and grind the paste in four stages of 50 revolutions each stage, picking up the paste with the same palette knife and transferring it to the centre of the plate after each stage.

When the grinding is complete, remove the paint from the plate and store it on a palette.

Prepare a paint immediately afterwards, with the agreed sample, using the same procedure and compare the colours as in Method 1.

Appendix J Method for the determination of "soluble lead" in lead chromes

J.1 Apparatus

a) *Beaker*, 1 500 ml.

b) *Mechanical stirrer*.

c) *Water bath*, thermostatically controlled.

d) *Sintered glass crucible*[30], porosity No. 4.

e) *Sintered silica crucible*[30], porosity No. 4.

J.2 Reagents

All reagents used shall be of recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.

a) *Hydrochloric acid*, dilute solution containing 0.25 per cent by weight of hydrogen chloride.

b) *Hydrogen sulphide*.

c) *Nitric acid/bromine reagent*. Saturate nitric acid ($d = 1.42$) with bromine.

d) *Sulphuric acid*, ($d = 1.84$).

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[25] BS 950, *"Artificial daylight fittings for colour matching"*.
[26] BS 242, *"Refined linseed oil for paints"*.
[27] BS 254, *"Zinc oxide (Types 1 and 2) for paints"*.
[28] BS 1851, *"Titanium dioxide pigments for paints"*.
[29] BS 1795, *"Extenders for paints"*.
[30] BS 1752 *"Sintered disk filters for laboratory use"*.

e) *Sulphuric acid*, dilute solution. Dilute 5 ml of sulphuric acid, (d = 1.84) by adding to water, with cooling, and making up to 100 ml with water.

f) *Ethanol*[31] 95 per cent (v/v).

J.3 Procedure

Place 1 000 g of the dilute hydrochloric acid solution in the 1 500 ml beaker which is fitted with a mechanical stirrer and immersed in a water bath, the temperature of the water having been adjusted to 60 °F. Allow the diluted acid to take up the temperature of the bath. Add exactly 1.0 g of the finely powdered sample (previously dried at 98–102 °C) and stir the whole continuously for exactly one hour at a rate sufficient to keep the whole of the pigment in continuous suspension. Stop the agitation and allow the beaker to remain in the constant temperature bath for one hour to allow the pigment to settle. Filter the mixture as rapidly as possible, preferably by suction, rejecting the first 50 ml of filtrate. Do not wash the filter.

Treat 800 ml of the perfectly clear filtrate with a rapid stream of hydrogen sulphide to saturation, allow the precipitated lead sulphide to settle and pour off the clear supernatant liquor through a sintered glass crucible. Wash the precipitate once by decantation with saturated hydrogen sulphide water and transfer to the filter with a jet of hydrogen sulphide water removing the last traces from the beaker with a "policeman". Wash the precipitate on the filter five times with hydrogen sulphide water and reject the filtrate. Dissolve the lead sulphide precipitate from the filter by treatment with the nitric acid/bromine reagent and wash well five times with hot water. Add to the filtrate and washings 15 ml of the sulphuric acid (d = 1.84) and evaporate the mixture carefully to dense fumes of sulphur trioxide. Cool the beaker and contents, wash the sides down and re-evaporate the contents to fumes. Cool the beaker again and add 250 ml of water and 100 ml of ethanol and allow the whole to stand overnight.

Filter the contents of the beaker through a tared sintered silica crucible, transfer the precipitate to the filter by a jet of a mixture of equal parts of ethanol and the dilute sulphuric acid solution, the last traces being removed with a "policeman". Wash the precipitate with ethanol until neutral and then dry in a steam oven, heat to dull red heat and weigh as lead sulphate.

J.4 Calculation

Percentage "soluble lead" as PbO — $92W$ where W is the weight of precipitate in grammes.

NOTE The method of ascertaining whether any compound is a "lead compound" (commonly known as the determination of "soluble lead") as defined in S.R. and O. 1926, No. 1621, specifies that the dried material shall be continuously shaken for one hour at the common temperature with 1 000 times its weight of an aqueous solution of hydrochloric acid containing 0.25 per cent by weight of hydrogen chloride. Since the "soluble lead" content of the material depends significantly upon the temperature at which the extraction is carried out, a definite temperature must be fixed for the estimation and in order to facilitate the maintenance of a constant temperature during the extraction period, stirring has here been substituted for the shaking required by the order.

For the purpose of this specification the "common temperature" has been interpreted as 60 °F but it should be noted that the interpretation of the words "the common temperature" is a matter for the Courts to decide. Pending such a decision, however, the Factory Department of the Ministry of Labour has stated that it would regard a temperature of 60 °F as falling within the meaning of the words and consequently would accept a test carried out at this temperature.

Appendix K Method for the estimation of total lead in lead chromes

K.1 Apparatus

a) *Sintered glass crucible*[32], No. 4 porosity.

b) *Sintered silica crucible*[32], No. 4 porosity.

c) *Beaker*, 600 ml.

d) *Silica crucible*, suitable size for holding b).

K.2 Reagents

The reagents used shall be of recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.

a) *Hydrochloric acid* (d = 1.18).

b) *Hydrogen sulphide*.

c) *Tartaric acid*.

d) *Nitric acid/bromine reagent*. Saturate nitric acid (d = 1.42) with bromine.

e) *Sulphuric acid* (d = 1.84).

f) *Sulphuric acid*, 5 per cent (w/v) aqueous solution.

g) *Ethanol*[33], 95 per cent (v/v) solution.

h) *Sodium sulphide*, 5 per cent (w/v) aqueous solution.

j) *Ammonium acetate*, saturated solution.

k) *Ammonia solution* (d = 0.880).

l) *Ammonium acetate*, crystals.

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[31] Ethanol may be replaced by industrial methylated spirits (ethanol containing 5 per cent by volume of approved wood naphtha) of equivalent strength. It should be noted that the use of industrial spirits is governed by the Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230).

[32] BS 1752, *"Sintered disk filters for laboratory use"*.

[33] Ethanol may be replaced by industrial methylated spirits (ethanol containing 5 per cent by volume of approved wood naphtha) of equivalent strength. It should be noted that the use of industrial spirits is governed by the Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230).

K.3 Procedure

Weigh accurately by difference about 0.5 g of the sample, previously dried at 98–102 °C into the 600 ml beaker. Add 100 ml of water, 15 ml of the hydrochloric acid and 2 ml of the ethanol, cover and raise to boiling point. Boil gently until all odour of aldehyde has been removed and the colour is clear bluish green. Dilute to 200 ml. If necessary, filter and wash the filter and residue well with hot water until a few drops of the filtrate give no coloration with sodium sulphide.

Combine filtrate and washings.

Slowly add ammonia solution, while stirring, until a faint permanent precipitate forms and at once add 0.5 g of the tartaric acid and 15 ml of the hydrochloric acid. Pass hydrogen sulphide to saturation, dilute to 400 ml and again saturate with hydrogen sulphide. Allow the precipitate to settle and pour off the clear supernatant liquor through the sintered glass crucible. Wash the precipitate once by decantation with saturated hydrogen sulphide solution and transfer it to the filter with a jet of hydrogen sulphide water. Wash the precipitate on the filter five times with hydrogen sulphide water and reject the filtrate and washings.

Transfer the precipitate to the precipitation beaker, and wash through the crucible with the nitric acid/bromine reagent, collecting the washings in the same beaker. Cover the beaker, heat the contents using a fume hood, filter through a sintered glass crucible and wash the beaker, cover and filter five times with hot water, transferring the filtrate and washings to a second 600 ml beaker. Cool and add 15 ml of the sulphuric acid, (d = 1.84), and evaporate the mixture carefully until dense fumes of sulphur trioxide are evolved. Cool the beaker and contents, wash down the sides and re-evaporate the contents to fumes. Cool the beaker again, add 250 ml water and 100 ml of the ethanol and allow it to stand overnight.

Pour off the clear liquor through the tared No. 4 porosity sintered silica crucible, wash the precipitate once by decantation with a mixture of equal parts of ethanol and the diluted sulphuric acid, and transfer to the filter by a jet of the same wash fluid, removing the last traces with the aid of a “policeman”. Wash the precipitate with ethanol until the washings are neutral, dry the crucible and precipitate in a steam oven, place in the outer silica crucible and heat to dull red heat. Cool in a desiccator and weigh.

Fill the crucible with ammonium acetate crystals and slowly pour 50 ml of boiling ammonium acetate solution through it. Wash very thoroughly with hot water until a few drops of the filtrate give no coloration with sodium sulphide solution, dry, ignite, cool and re-weigh as before.

K.4 Calculation

$$\text{Percentage total lead, as PbO} = \frac{73.6\ (W_1 - W_2)}{W}$$

where W_1 is the weight, in grammes, of the crucible plus lead sulphate

W_2 is the weight, in grammes, of the crucible after extraction

and W is the weight, in grammes, of the sample taken.

Appendix L Method for the determination of matter insoluble in ammoniacal ammonium chloride solution

L.1 Apparatus

a) *Sintered glass crucible*, porosity No. 4[34].

b) *Mechanical shaker*.

L.2 Reagent

The reagent used shall be of recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.

Ammoniacal ammonium chloride solution. Dissolve 30 g ammonium chloride in a mixture of 500 ml of ammonia solution (d = 0.880) and 600 ml of water.

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[34] BS 1752, *“Sintered disk filters for laboratory use”*.

L.3 Procedure

Shake approximately 5 g of the sample, weighed accurately, with 100 ml of the reagent for one hour at room temperature. Allow to stand for 30 minutes and decant the supernatant liquor through the tared sintered glass crucible. To the residue add a further 100 ml of the reagent, shake for 5 minutes and filter the whole suspension through the same filter crucible, any adherent residue being transferred to the filter by means of a "policeman" and the minimum additional reagent. Wash the sides of the filter crucible and the residue with two successive 25 ml portions of water, and finally dry the crucible and residue to constant weight in an oven at 98–102 °C.

L.4 Calculation

Percentage matter insoluble in ammoniacal ammonium chloride = $\frac{100W_1}{W}$

where W is the weight, in grammes, of sample taken

and W_1 is the weight, in grammes, of residue.

Appendix M Method for the determination of water soluble sulphates, chlorides and nitrates and total water soluble matter

M.1 Apparatus

a) *Sintered glass crucible*[35], porosity No. 4.

b) *Sintered silica crucible*[35], porosity No. 4.

c) *Nessler cylinders*, 50 ml, complying with BS 612[36].

d) *Distillation apparatus.*

e) *Mechanical shaker*.

M.2 Reagents

All reagents used shall be of recognized analytical reagent quality. Neutral distilled water or water of at least equal purity shall be used throughout.

NOTE Special ammonia-free water is not required for these determinations.

a) *Hydrochloric acid*, (d = 1.18).

b) *Ethanol*[37] 95 per cent (v/v).

c) *Nitric acid*, (d = 1.42).

d) *Nitric acid*, 1 per cent (v/v) solution.

e) *Ammonium chloride* 0.00172 per cent (w/v) solution.

f) *Sodium hydroxide* 20 per (w/v) solution.

g) *Devarda's alloy*, powdered.

h) *Barium chloride*, 10 per cent (w/v) solution.

j) *Silver nitrate*, 5 per cent (w/v) solution.

k) *Nessler's reagent*. Prepared by either method i) or method ii) as follows.

i) Dissolve 5 g of potassium iodide in 3.5 ml of water. Add cold saturated mercuric chloride solution with stirring until a faint red precipitate is formed. With continued stirring add 40 ml of 50 per cent potassium hydroxide solution, dilute to 100 ml, mix well, allow to settle, decant the clear supernatant liquor and store in the dark.

ii) Dissolve 3.5 g of potassium iodide and 1.25 g of mercuric chloride in 80 ml of water. Add cold saturated mercuric chloride solution with shaking until a slight red precipitate remains; then add 12 g of sodium hydroxide, shake until dissolved, and finally add a little more of the saturated mercuric chloride solution and dilute to 100 ml with water. Shake occasionally during several days, allow to stand, and use the clear supernatant liquid for the test.

M.3 Procedure

Shake 30 g of the sample with 300 ml of water in a chemically resistant glass flask for one hour on the mechanical shaker at room temperature. Filter the mixture, and reserve the perfectly clear filtrate for determinations M4 to M7 below.

M.4 Determination of sulphates

Acidify 50 ml of the clear aqueous extract with 3 ml of the hydrochloric acid and add a few ml of the ethanol. Warm the solution to reduce the chromate as indicated by a change of colour to green. Boil the solution vigorously to drive off organic compounds, taking care to avoid loss of solution by splashing. Add the barium chloride solution dropwise until in slight excess to the hot solution and allow to stand overnight. Decant the supernatant liquid through the tared silica crucible, transfer the precipitate and wash it with hot water until free from chloride, ignite gently, then at red heat, cool in a desiccator and weigh.

Calculation. Percentage of sulphates (SO_4) = 8.23W where W is the weight, in grammes, of barium sulphate precipitate.

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[35] BS 1752, *"Sintered disk filters for laboratory use"*.

[36] BS 612, *"Nessler cylinders"*.

[37] Ethanol may be replaced by industrial methylated spirits (ethanol containing 5 per cent by volume of approved wood naphtha) of equivalent strength. It should be noted that the use of industrial spirits is governed by the Methylated Spirits Regulations, 1952 (S.I. 1952, No. 2230).

M.5 Determination of chlorides

Acidify 50 ml of the clear aqueous extract with 10 ml of the nitric acid and add an excess of the silver nitrate solution. Heat the solution until the precipitate of silver chloride coagulates. Filter off the precipitate in a weighed sintered glass crucible, wash with the dilute nitric acid till free from silver nitrate, and then once with hot water. Dry at 100° ± 5 °C and weigh.

Calculation. Percentage of chlorides (Cl) = $4.96\,W$

where W is the weight in grammes of silver chloride precipitate.

M.6 Determination of nitrates

Place 50 ml of the clear aqueous extract in the distillation flask and dilute to 150 ml with water. Add 3 g of Devarda's alloy and 30 ml of the sodium hydroxide solution and close the apparatus at once. Place 2 ml of the hydrochloric acid and 30 ml of water in the receiver.

Warm the flask gently until the reaction starts and then allow the reaction to proceed gently for about half an hour. Then allow about 70 ml of water to distil over, the receiver being kept cool with running water.

Make up the distillate to 250 ml and transfer 5 ml to a Nessler cylinder. Add 1 ml of Nessler's reagent and match the colour against that of a similar standard solution prepared by adding ammonium chloride solution from a burette.

Calculation. Percentage of nitrate (NO_3) = $0.02\,T$

where T is the volume, in millilitres, of ammonium chloride solution required.

M.7 Determination of total water-soluble matter

Place 100 ml of the clear aqueous extract in a tared flat-bottomed, glass, glazed porcelain or silica dish and evaporate to dryness on a water bath or in an oven at 100 °C.

Calculation. Water-soluble matter, per cent by weight = $\dfrac{300\,W_1}{W}$

where W_1 = weight, in grammes, of residue

and W = weight, in grammes, of material taken.

Appendix N Method for the determination of water soluble chromate

N.1 Apparatus

Mechanical shaker.

N.2 Reagents

All reagents used shall be of recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.

a) *Sulphuric acid*, dilute solution. Dilute 25 ml sulphuric acid ($d = 1.84$) by adding to water, with cooling, and making up to 100 ml with water.

b) *Ammonium ferrous sulphate*, 0.1N solution.

c) *Potassium permanganate*, 0.1N solution.

N.3 Procedure

Shake 30 g of the sample with 100 ml of water for one hour on a mechanical shaker, at a temperature between 18 and 25 °C. Filter, transfer 50 ml of the clear filtrate to a conical flask and acidify with the sulphuric acid. Add a measured excess volume (50 ml is usually sufficient) of the ammonium ferrous sulphate solution and titrate the excess with the potassium permanganate solution.

N.4 Calculation

Percentage of water soluble chromates (CrO_3) = $0.02223\,(T - T_1)$.

where T is the volume, in millilitres, of 0.1N ammonium ferrous sulphate added and T_1 is the volume, in millilitres, of 0.1N potassium permangante solution required.

Appendix O Method for the determination of zinc content

O.1 Reagent

All reagents used shall be of recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.

a) *Diphenylbenzidine* solution, 0.25 g in 25 ml of sulphuric acid, ($d = 1.84$).

b) *Potassium ferrocyanide-potassium ferricyanide solution.* Dissolve 22.0 g of potassium ferrocyanide and 0.5 g of potassium ferricyanide in water and make up to 1 litre.

c) *Sulphuric acid* ($d = 1.84$).

d) *Sulphuric acid*, dilute solution. Dilute 25 ml of sulphuric acid ($d = 1.84$), by adding to water, with cooling and making up to 100 ml with water.

e) *Sulphur dioxide solution.*

f) *Ammonium sulphate*.

g) *Zinc solution*, dissolve 12.5 g of pure, dry, ignited zinc oxide in 1 litre of the dilute sulphuric acid.

O.2 Procedure

Dissolve 0.5 g of the sample in a mixture of 50 ml of water and 20 ml of the sulphuric acid. Reduce the solution with the sulphur dioxide solution and boil off the excess sulphur dioxide. If necessary make up the total volume to the original volume by the addition of water. Cool the solution and add approximately 10 g of the ammonium sulphate and 4 drops of the diphenylbenzidine solution. Titrate the solution slowly with the potassium ferrocyanide-potassium ferricyanide solution until a full purple colour develops, indicating the approach of the end point. Titrate to the end point which is characterized by a change in colour from purple to light green.

Standardize the titrant against 20 ml of the zinc solution diluted with 50 ml of water. Add 10 g of the ammonium sulphate and 4 drops of the diphenylbenzidine solution as before and titrate to the same colour change.

O.3 Calculation

Percentage of zinc as zinc oxide = $\frac{50\,T}{T_1}$

where T is the volume, in millilitres, of solution required by the sample and T_1 is the volume, in millilitres, of solution required by the standard zinc solution.

NOTE For a freshly prepared titrant solution T_1 = 39.5 ml.

Appendix P Method for the determination of alkali metals

P.1 Reagents

All reagents used shall be of recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.

a) *Sulphuric acid*, dilute solution. Dilute 25 ml of sulphuric acid, (d = 1.84) by adding to water, with cooling and making up to 100 ml with water.

b) *Ammonium ferrous sulphate*, 0.1N solution.

c) *Potassium permanganate*, 0.1N solution.

P.2 Procedure

Heat 2 g of the sample to approximately 600 °C for 1 hour. The material is thereby decomposed into zinc oxide, chromic oxide (Cr_3O_3) and alkali metal chromate. Cool and extract the alkali metal chromate from the residue with hot water, filter and wash the residue free from soluble chromate. Acidify the total filtrate with the sulphuric acid.

Add a measured excess volume (100 ml is usually sufficient) of the ammonium ferrous solution and titrate the excess with the potassium permanganate solution.

P.3 Calculation

Percentage of alkali metal (as K_2O) = 0.1565 ($T - T_1$)
where T is the volume, in millilitres, of 0.1N ammonium ferrous sulphate solution added and T_1 is the volume, in millilitres, of 0.1N potassium permanganate solution required.

Appendix Q Method for the determination of chromate content

Q.1 Reagents

All reagents used shall be of recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used throughout.

a) *Sulphuric acid*, dilute solution. Dilute 25 ml of sulphuric acid (d = 1.84), to 100 ml with water.

b) *Ammonium ferrous sulphate*, 0.1N solution.

c) *Potassium permanganate*, 0.1N solution.

Q.2 Procedure

Dissolve 1 g of the sample in 10 ml of the sulphuric acid and make up to exactly 250 ml with water. To 50 ml of this solution, add 5 ml of the sulphuric acid, then add a measured excess volume (50 ml is usually sufficient) of the ammonium ferrous sulphate solution and titrate the excess with the potassium permanganate solution.

Q.3 Calculation

Percentage of chromate (CrO_3) = 1.667 ($T - T_1$)
where T is the volume, in millilitres, of 0.1N ammonium ferrous sulphate solution and T_1 is the volume, in millilitres, of 0.1N potassium permanganate solution required.

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