

Australian Standard™

**Paints and related materials—
Micaceous iron oxide pigment**



This Australian Standard was prepared by Committee CH-003, Paints and Related Materials. It was approved on behalf of the Council of Standards Australia on 16 April 2004 and published on 17 May 2004.

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Australian Paint Manufacturers Federation
Building Research Association of New Zealand
Business New Zealand
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STANDARDS AUSTRALIA

RECONFIRMATION

OF

AS 2855—2004

Paints and related materials—Micaceous iron oxide pigment

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NOTES

Australian Standard™

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PREFACE

This Standard was prepared by the Standards Australia Committee CH-003, Paints and Related Materials, to supersede AS 2855—1986.

The Standard is republished without technical alterations.

Methods of test have been referred to or drawn from relevant Australian Standards where they exist. Otherwise, they consist of adaptations of the ISO methods referred to in ISO 1248—*Iron Oxide Pigments for Paints*, modified to only a minor extent in the technical sense. For purposes of future reference these ISO standards comprised the following:

ISO

787 General Methods of Test for Pigments and Extenders

Part 2: Determination of Matter Volatile at 105°C

Part 3: Determination of Matter Soluble in Water—Hot Extraction Method

Part 4: Determination of Acidity or Alkalinity of the Aqueous Extract

Part 7: Determination of Residue on Sieve—Water Method—Manual Procedure

Part 10: Determination of Density—Pyknometer Method

Part 13: Determination of Water-soluble Sulphates, Chlorides and Nitrates

The terms ‘normative’ and ‘informative’ have been used in this Standard to define the application of the appendix to which they apply. A ‘normative’ appendix is an integral part of a Standard, whereas an ‘informative’ appendix is only for information and guidance.

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FOREWORD

Micaceous iron oxide (MIO), also known as specular haematite, is a refined mineral ore consisting essentially of iron (III) oxide (Fe_2O_3). It is grey with a metallic sheen. The term ‘micaceous’ stems from the similarity between the structure of the particles and the crystalline structure of mica.

Depending on the location of the ore deposits, MIO may differ considerably in purity, size and shape.

The distinguishing feature of MIO is the tabular crystalline structure which can be easily fractured to give very thin platelets or lamellar fragments. When used as a pigment in paint, these thin platelets generally orientate themselves in overlapping layers with their thinnest cross-sections parallel to the coated substrate. This orientation produces an increase in resistance to water permeation through the paint film, reduces ultraviolet degradation of the vehicle and provides mechanical reinforcement to the paint film.

MIO pigmented paints have been widely and successfully used for many years to provide protection to steel from corrosion and long-term resistance to outdoor weathering.

STANDARDS AUSTRALIA

Australian Standard

Paints and related materials—Micaceous iron oxide pigment

1 SCOPE

This Standard specifies requirements for micaceous iron oxide pigment (MIO) used primarily in paints for imparting corrosion resistance and long term durability to steel.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

2162 Verification and use of volumetric apparatus

2162.1 Part 1: General—Volumetric glassware

2831 Thermometers—Solid stem—Long and short—For precision use

AS/NZS

1580 Paints and related materials—Methods of test

1580.101.4 Part 101.4: Conditions of test—Temperature control

3 PROPERTIES

The properties of a representative sample of MIO shall be in accordance with specifications contained in Table 1.

4 FORM

4.1 General

The form (i.e., structure, shape and size) of a representative sample of micaceous iron oxide shall be that of a grey iron oxide with a metallic sheen and of an essentially laminar nature complying with the requirements of Clauses 4.2 and 4.3.

4.2 Structure

When a test portion of the pigment is viewed under a scanning electron microscope using a magnification of about $\times 250$, the structure of the particles shall correspond to the typical lamellar structure shown in Figure 1. It shall not correspond to the amorphous granular structure illustrated in Figure 2.

4.3 Shape, size and colour

When a test portion of the pigment is viewed by means of an optical microscope using a magnification of about $\times 200$ and transmitted light, the particles shall be seen to consist of sharply defined, angular, ruby red crystals with clearly defined fracture planes as shown in Figure 3. The particles shall not correspond to those illustrated in Figure 4.

5 PACKAGING AND LABELLING

5.1 Packaging

The MIO shall be supplied in sound, sealed packages, and shall exhibit no damage by moisture or other agents.

5.2 Labelling

The following information shall be legibly and durably marked on each package or container or on a label securely affixed thereto:

- (a) Name or registered mark of the manufacturer/supplier.
- (b) Identification and/or description of the contents.
- (c) Origin of contents including the name of the Australian agent(s) or distributor(s), if the MIO is imported.
- (d) Mass of the contents, in kilograms.
- (e) Information required by any appropriate statutory authority.

NOTE: Manufacturers who place the number of this Australian standard on MIO containers, on packaging or on literature related thereto should ensure that the product is manufactured to comply with the Standard.

TABLE 1
PROPERTIES OF ACCEPTABLE MIO

Property	Required values	Test method
Iron content, as Fe_2O_3	$\geq 85\%$ (m/m)	Appendix A
Water-soluble matter	$\leq 0.5\%$ (m/m)	Appendix B
Water-soluble chlorides and sulphates, as $(\text{Cl}^- + \text{SO}_4^{2-})$	$\leq 0.1\%$ (m/m)	Appendix C
Matter volatile at 105°C	$\leq 1\%$ (m/m)	Appendix D
pH of aqueous extract	$\geq 6.5, \leq 8.0$	Appendix E
Loss on ignition at 1000°C (see also Paragraph F1)	$\leq 1.5\%$ (m/m)	Appendix F
Residue on $106\ \mu\text{m}$ sieve	$\leq 0.1\%$ (m/m)	Appendix G
Density at $25 \pm 1^\circ\text{C}$	$\geq 4700\ \text{kg/m}^3$	Appendix H*

* Refer also to NOTE of Paragraph H1 of Appendix H.

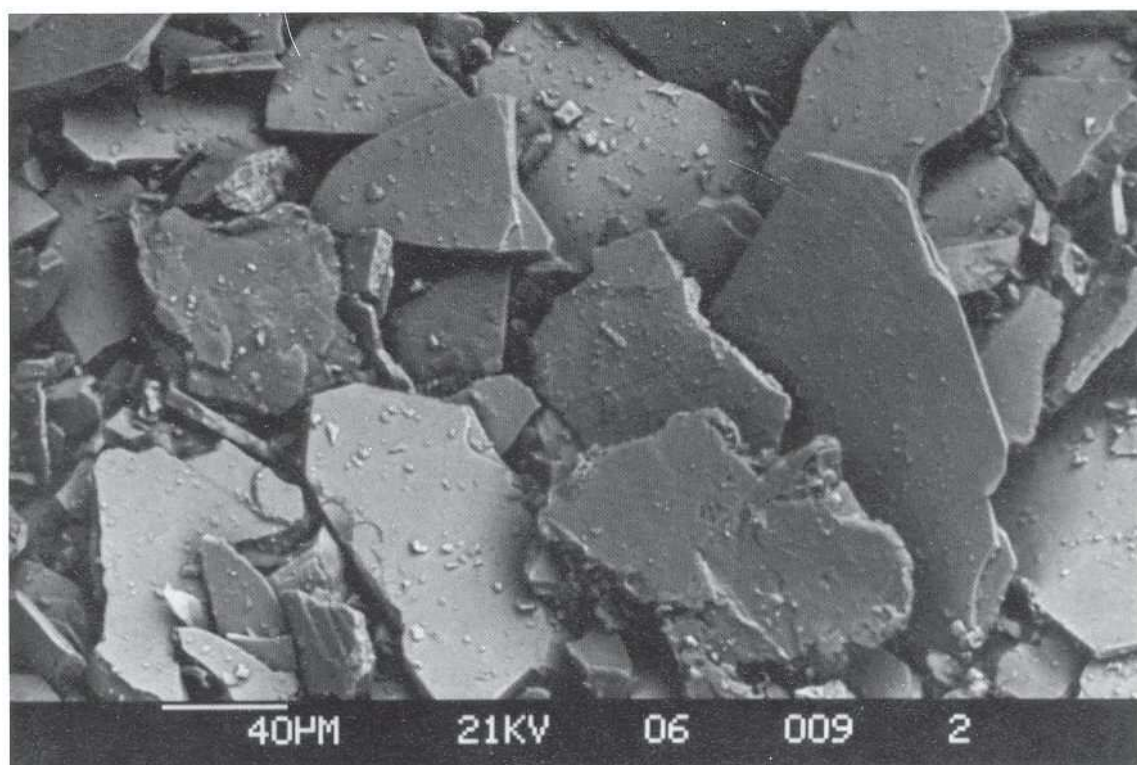


FIGURE 1 TYPICAL LAMELLAR STRUCTURE

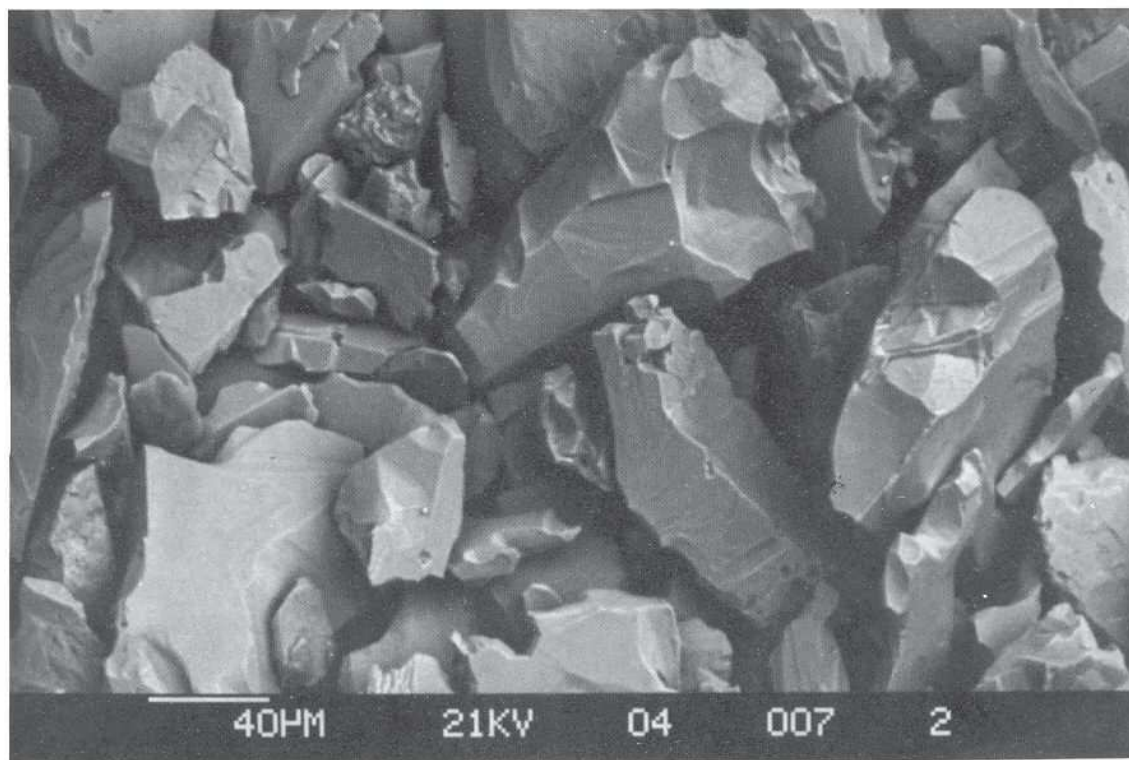


FIGURE 2 TYPICAL GRANULAR OR AMORPHOUS STRUCTURE

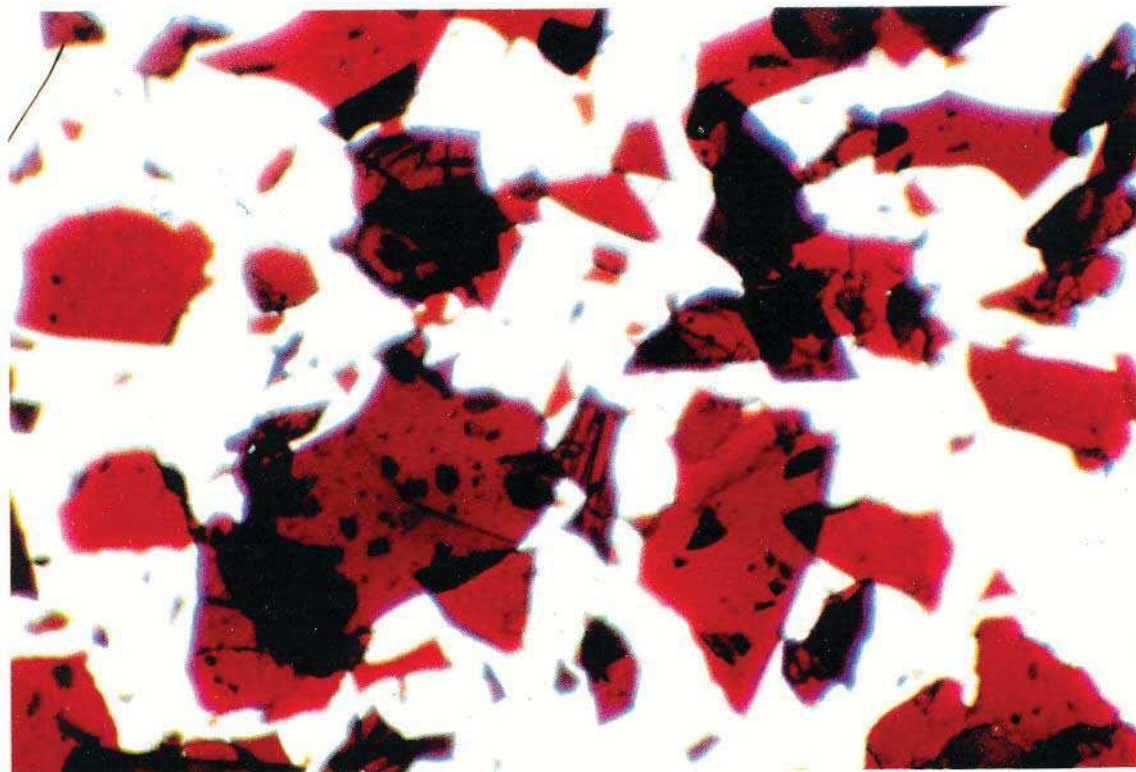


FIGURE 3 SHAPE AND SIZE—ACCEPTABLE CHARACTERISTICS



FIGURE 4 SHAPE AND SIZE—UNACCEPTABLE CHARACTERISTICS

APPENDIX A

DETERMINATION OF TOTAL IRON CONTENT EXPRESSED AS IRON(III)
OXIDE

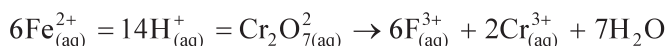
(Normative)

A1 SCOPE

This Appendix describes a titrimetric method for the determination of total iron content in natural iron oxide pigments, expressed as iron(III) oxide.

A2 PRINCIPLE

The test sample is solubilized with hot hydrochloric acid solution aided by the controlled addition of small amounts of tin(II) chloride solution. The solution is then diluted, heated until simmering and the soluble iron completely reduced to the Fe^{2+} state with more tin(II) chloride. After removal of excess tin(II) reagent with saturated mercury(II) chloride, the iron(II) content is determined titrimetrically with potassium dichromate solution using diphenylamine as an end-point enhancer in the presence of a sulphuric/phosphoric acid mixture. The titration reaction, used for stoichiometric purposes, is as follows:

**A3 REAGENTS**

NOTE: All reagents shall be of analytical grade, and distilled or demineralized water shall be used throughout the analysis.

A3.1 Ammonium thiocyanate solution (170 g/L)**A3.2 Diphenylamine, sulphuric acid solution**

Carefully dissolve 1 g of barium diphenylaminesulphonate in 100 mL of sulphuric acid ($\rho_{20} 1840 \text{ kg/m}^3$).

A3.3 Hydrochloric acid ($\rho_{20} 1180 \text{ kg/m}^3$)**A3.4 Mercury(II) chloride solution, saturated****A3.5 Potassium chlorate, solid****A3.6 Potassium dichromate solution (0.01667 mol/L)**

Dry about 10 g $\text{K}_2\text{Cr}_2\text{O}_7$ to constant mass at 150°C . Weigh out between 4.903 g and 4.905 g and dissolve quantitatively in a minimum of water in a 1 L volumetric flask. Make up to the mark with water and mix thoroughly.

A3.7 Potassium permanganate solution (approx. 0.02 mol/L)

Weigh approximately 0.3 g of KMnO_4 into a clean 100 mL beaker and dissolve in 50–60 mL water. Filter through a sintered glass funnel or simply, through a glass funnel plugged with glass wool, into a 100 mL brown, covered or dark-coloured glass stock bottle and make up to capacity with distilled water, mixing thoroughly.

A3.8 Sulphuric acid/phosphoric acid mixture

Carefully add, with stirring, 150 mL sulphuric acid ($\rho_{20} 1840 \text{ kg/m}^3$) to 150 mL phosphoric acid ($\rho_{20} 1700 \text{ kg/m}^3$). Add the mixture cautiously in small amounts to 600–700 mL of water in a 2 L beaker with stirring. After allowing to cool, dilute to 1 L.

CAUTION: Great care must always be taken when diluting concentrated acids, as the heat generated by improper mixing procedures may be sufficient to cause local boiling, resulting in the ‘spitting’ of hot acid droplets. The recommended safe procedure is to always add the acid, in small amounts, to the water with stirring preferably using a magnetic stirrer. Safety goggles or the equivalent must always be worn.

A3.9 Tin(II) chloride solution (100 g/L)

Carefully dissolve 50 g of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 300 mL of hydrochloric acid (Paragraph A3.3) and dilute to 500 mL with water (see CAUTION to Paragraph A3.8). Store solution in a hermetically sealed flask containing a few grams of metallic tin.

A4 APPARATUS

AS Grade A laboratory glassware used in accordance with AS 2162.1.

A5 TEST SAMPLE

A5.1 Pretreatment

Unless otherwise specified, dry approximately 10 g of bulk material, to constant mass at 105°C and subsequently store in a desiccator until required.

If the sample is known or suspected to contain organic matter, heat it in a porcelain crucible until it reaches a dark red colour, before commencing the test procedure (Paragraph A6).

A5.2 Sample size

The mass of test sample used per titration depends upon the quantity of total iron present in the sample. As a guide, it is recommended that 0.3 g of sample be used if the total iron oxide content is above 95% m/m. For expected iron oxide contents below this amount, the sample size should be proportionately increased relative to this value.

A6 PROCEDURE

The procedure for analysis shall be as follows:

- (a) Weigh out the estimated amount of test sample (see Paragraph A5.2) to the nearest 0.0001 g and transfer quantitatively to a 400 mL beaker.
- (b) Add 60 mL of hydrochloric acid (Paragraph A3.3) and 0.5 g potassium chlorate (Paragraph A3.5). Cover the beaker with a watch-glass and heat at 80–90°C until all dark particles in the insoluble residue disappear.
- (c) Add in, dropwise fashion, with stirring, some of the tin(II) chloride solution (Paragraph A3.9) during the heating process but avoid adding amounts which result in the complete removal of colour from the solution.

NOTE: If too much tin(II) chloride solution has been added in error, add some potassium permanganate to the solution until a yellow colour appears. Then add tin(II) chloride solution, drop by drop, until the yellow colour disappears, subsequently adding one or two drops in excess.

- (d) Proceed to Step (e) when the residue has become almost colourless.
NOTE: If a black insoluble residue remains, filter this residue and fuse it with sodium or potassium carbonate in a platinum crucible. Then extract with hydrochloric acid (Paragraph A3.3) and add the extract to the filtrate.
- (e) Add 25 to 50 mL of water to the solution and heat until gently boiling (avoid intense and prolonged boiling).
- (f) Add slowly, with stirring, tin(II) chloride solution (Paragraph A3.9) in dropwise fashion, until the last drop makes the solution colourless or free from yellow colour, then add one or two drops in excess. See also Note to Step (c).