BRITISH STANDARD

BS 3483-C6: 1974

Methods for

Testing pigments for paints —

Part C6: Determination of water-soluble sulphates, chlorides and nitrates

This part should be read in conjunction with the General Introduction to BS 3483 issued separately.

NOTE This part of BS 3483 is technically identical with Part XIII of ISO/R 787, *General methods of test for pigments.*

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 and 2 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.

Amendments issued since publication

Amd. No.	Date	Comments

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Contents

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1 Scope

Part C6 of this British Standard describes a general method of test for determining the content of water-soluble sulphates, chlorides and nitrates in a sample of pigments.

NOTE When this general method is applicable to a given pigment, only a cross-reference to it should be included in the British Standard relating to that pigment, with a note of any detailed modification which may be needed in view of the special properties of the pigment in question. Only when this general method is not applicable to a particular pigment should a special method be specified for determination of water-soluble sulphates, chlorides and nitrates.

2 Reagents

2.1 All reagents used shall be of recognized analytical reagent quality. Distilled water or water of at least equal purity shall be used.[1)]

2.1.1 *Hydrochloric acid,* N solution.

2.1.2 *Silver nitrate*, 0.01 N standard volumetric solution.

2.1.3 *Ammonium chloride solution*, 17.2 mg/l.

2.1.4 *Sodium hydroxide solution*, 200 g/l.

2.1.5 *Barium chloride solution*, 50 g/l.

2.1.6 *Potassium chromate solution*, 50 g/l.

2.1.7 *Devarda's alloy*, powdered.

2.1.8 *Nessler's reagent*. Prepared by either method 1) or method 2) as follows:

1) Dissolve 5 g of potassium iodide in 35 ml of water. Add cold saturated mercury (II) chloride ($HgCl_2$) solution while stirring until a faint red precipitate is formed. Continuing to stir, add 40 ml of potassium hydroxide solution (500 g/l), dilute to 100 ml, mix well, allow to settle, decant the clear supernatant liquor and store it in the dark.

2) Dissolve 3.5 g of potassium iodide and 1.25 g of mercury (II) chloride ($HgCl_2$) in 80 ml of water. Add cold saturated mercury (II) chloride solution while 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 mercury (II) 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.

3 Apparatus

3.1 *Sintered silica crucible*, porosity grade No. 4.[2)]

3.2 *Nessler cylinders*, 50 ml complying with BS 612[3)].

3.3 *Distillation apparatus*

4 Sampling

The sample of pigment used for the test shall be taken in accordance with the provisions of BS 4726[4)].

5 Determination of sulphates

5.1 Procedure. Take 50 ml of the clear aqueous extract obtained in one of the methods, as appropriate, for the determination of matter soluble in water (either the hot extraction method[5)] or the cold extraction method[6)]), acidify with 2.5 ml of the hydrochloric acid (**2.1.1**) and boil the solution vigorously, taking care to avoid loss of solution by splashing. Add the barium chloride solution (**2.1.5**), drop by drop, to the hot solution until in slight excess, and allow the solution to stand overnight. Decant the supernatant liquid through the tared silica crucible (**3.1**). Transfer the precipitate to the crucible and wash it free from chloride, ignite it gently, then heat to red heat, cool it in a desiccator and weigh to the nearest milligram.

5.2 Expression of results

Calculate the water-soluble sulphate content, expressed as SO_4, as a percentage by mass, by means of the formula $206\ m_1/m_0$, where m_1 is the mass in grams of barium sulphate precipitate and m_0 is the mass in grams of pigment used in the determination of matter soluble in water.

Report the result to two decimal places.

6 Determination of chlorides

6.1 Procedure. Take 50 ml of the clear aqueous extract obtained in one of the methods, as appropriate, for the determination of matter soluble in water (either the hot extraction method[5)] or the cold extraction method[6)]) and add 1 ml of the potassium chromate solution (**2.1.6**). Titrate the solution with the 0.01 N silver nitrate solution (**2.1.2**), slowly and with vigorous shaking, until a faint reddish brown colour persists.

[1)] BS 3978, "*Water for laboratory use*".
[2)] BS 1752, "*Laboratory sintered or fritted filters*".
[3)] BS 612, "*Nessler cylinders*".
[4)] BS 4726, "*Sampling raw materials for paints and varnishes*".
[5)] BS 3483-C1, "*Determination of matter soluble in water (hot extraction method)*".
[6)] BS 3483-C2, "*Determination of matter soluble in water (cold extraction method)*".