

Stability to Hardness of Dye Solutions

1 Purpose and Scope

- 1.1 By stability to hardness of dye solutions is meant their resistance to precipitation on dilution with hard water.
- 1.2 This method is valid for all water-soluble leather dyes.

2 Principle

- 2.1 A stock solution of the dyestuff (2 g/litre) is diluted with a nineteen-fold amount of distilled water or artificially prepared hard water equivalent to 200 or 400 mg CaO per litre. Precipitation by the hard water is assessed by visual comparison in test tubes with the dye solution diluted with distilled water after 10 min and then again after 1 h.

3 Apparatus and Reagents

- 3.1 Erlenmeyer flask, 300 ml.
- 3.2 Watch glass, diameter about 50 mm, with hole.
- 3.3 Thermometer, 0-100 °C, 1 °C divisions.
- 3.4 Three test tubes, diameter about 18 mm, about 180 mm long.
- 3.5 Pipette, 1 ml.
- 3.6 Three graduated pipettes, 25 ml, 0.1 ml divisions.
- 3.7 Stock solutions of hard water are prepared:
 Stock solution A: 39.05 g $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ /litre \equiv 10 g CaO/litre
 Stock solution B: 43.65 g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ /litre \equiv 10 g CaO/litre
 The standardised hard water, containing permanent hardness with a molar ratio of calcium chloride to magnesium sulphate of 2:1, is prepared from the stock solutions as follows:

Equivalent to	200 ml	Stock solution A
200 mg CaO/litre	100 ml	Stock solution B
		diluted with distilled water to 15 litres
Equivalent to	400 ml	Stock solution A
400 mg CaO/litre	200 ml	Stock solution B
		diluted with distilled water to 15 litres

4 Procedure

- 4.1 Dissolve 0.4 g dye in 200 ml distilled water, according to directions, where these are supplied.
- 4.2 In absence of directions for dissolving, pour 200 ml distilled water at room temperature over the dye contained in the Erlenmeyer flask, cover with watch glass, boil up, and boil gently for 2 min. Cool with constant stirring to 20 °C by placing in cold water.
- 4.3 Place 1 ml (Section 3.5) dye solution at 20 °C into each of three test tubes (Section 3.4) at 20 °C.
- 4.4 To one of the test tubes containing dye solution, add 19 ml (Section 3.6) distilled water, water of 200 mg CaO/litre, and water of 400 mg CaO/litre (Section 3.7), respectively and shake briefly by hand.
- 4.5 Assess visually the three dye solutions 10 min and also 1 h after preparation and, in the event of precipitation, distinguish between

weak or incipient and marked precipitation (see Sections 5.4 and 6.3).

5 Report

This should comprise:

- 5.1 Name of the dye and Colour Index designation (see Section 6.4).
- 5.2 Reference to the present method.
- 5.3 Details of any deviations from the procedure, including special directions followed for dissolving the dye, if any.
- 5.4 The stability to hardness in whole numbers 5 to 1, according to the following scheme:
 - Rating 5 = no precipitation with hard water.
 - Rating 4 = no precipitation with hardness equivalent to 200 mg CaO/litre, incipient precipitation with 400 mg CaO/litre.
 - Rating 3 = no precipitation with hardness equivalent to 200 mg CaO/litre, marked precipitation with 400 mg CaO/litre
 - Rating 2 = incipient precipitation with hardness equivalent to 200 mg CaO/litre
 - Rating 1 = marked precipitation with hardness equivalent to 200 mg CaO/litre.

6 Notes

- 6.1 See: *General Principles of Colour Fastness Testing of Leather, IUF 120.*
- 6.2 There is no relationship between the stability of a dye to hardness and the dyeings which can be obtained with it in hard water. The present method gives information only on the behaviour of a dye dissolved in soft water on dilution with water of different degrees of hardness.
- 6.3 Precipitation can occur after 24 h which is not seen after an hour. This may be important for dyestuff solutions kept as stock solutions over an extended period of time.
- 6.4 If there is no Colour Index designation, state dyeing and chemical class.