

Stability to Acid of Dye Solutions

1 Purpose and Scope

- 1.1 By stability to acid of dye solutions is meant their resistance to precipitation by the action of dilute formic and sulphuric acid.
- 1.2 This method is valid for all water-soluble leather dyes.

2 Principle

- 2.1 To the dye in aqueous solution of normal dyebath concentration (5 g dye/litre) is added severally an amount of formic acid and sulphuric, in the form of a dilute solution and calculated to equal the amount of dye present (see Sections 3.11 and 3.12). The precipitation is assessed by comparison with the unacidified solution.
- 2.2 For this purpose, a drop of the dye solution is placed on to filter paper and, after drying, the spots are assessed visually (see Section 5.4).

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 Measuring cylinder, 250 ml, 2 ml divisions.
- 3.5 Three test tubes, diameter about 18 mm, about 180 mm long.
- 3.6 Pipette, 10 ml.
- 3.7 Three graduated pipettes, 1 ml, 0.05 ml divisions.
- 3.8 Three graduated pipettes or droppers to measure 0.1 ml (3 drops).
- 3.9 Filter paper, equivalent in quality and porosity to Schleicher and Schüll No 604 or Whatman No 4.
- 3.10 Frame over which filter paper can be stretched (see Section 6.1).
- 3.11 Formic acid solution, 100 g 100% HCOOH/litre.
- 3.12 Sulphuric acid solution, 100 g 100% H₂SO₄/litre.

4 Procedure

- 4.1 Dissolve 1.0 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 minutes. Cool to 60 ± 2 °C (see also Section 6.2).
- 4.3 Place 10 ml (Section 3.6) dye solution at 60 °C into each of three test tubes (Section 3.5), prewarmed to 60 °C.
- 4.4 To one of the test tubes containing dye solution, add 0.5 ml (Section 3.7), formic acid solution (Section 3.11), sulphuric acid solution (Section 3.12), and distilled water, respectively, and shake briefly by hand.

- 4.5 Immediately after the addition of acid, allow 0.1 ml (Section 3.8) of each of the three dye solutions to drop from about 1 cm to the stretched filter paper (Sections 3.9/3.10), so that three separate, but adjacent coloured areas are obtained.
- 4.6 After drying, but not before two hours after spotting, assess visually the two spots from the acidified solutions in comparison with that from the unacidified solution and in the event of precipitation distinguish between weak or incipient and marked precipitation (see Section 5.4).

5 Report

This should comprise:

- 5.1 Name of the dye and Colour Index designation (see Section 6.5).
- 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 acid in whole numbers 5 to 1, according to the following scheme:
- Rating 5 = no precipitation with either acid.
Rating 4 = no precipitation with formic acid, incipient precipitation with sulphuric acid.
Rating 3 = no precipitation with formic acid, marked precipitation with sulphuric acid.
Rating 2 = incipient precipitation with formic acid (see Section 6.3).
Rating 1 = marked precipitation with formic acid (see Section 6.3).

6 Notes

- 6.1 Suitable is, for example, a wooden frame about 5 cm high, with an area of about 33 cm x 24 cm, which allows the filter paper to be cut to format A4 after removal from the frame.
- 6.2 Dyes which gelatinise or have poor solubility are to be tested at that temperature at which they are just in solution.
- 6.3 Dyes which are precipitated by formic acid are generally also not resistant to precipitation by sulphuric acid. In the very rare cases where this does not obtain, a note added to rating 1 or 2 should state that the dye tested precipitated on addition of formic acid, but not on addition of sulphuric acid.
- 6.4 The filter papers spotted according to this procedure may be used simultaneously for determining the fastness to acid of dye solutions (see IUF 202).
- 6.5 If there is no Colour Index designation, state dyeing and chemical class.