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Note

Thin-layer chromatographic identification of leather dyes

II. Studies of mixtures of leather dyes

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In our earlier communication[1], the thin-layer chromatographic identification of 34 leather dyes on silica gel G layers (thickness 0.25 mm) using 86 solvent systems

TABLE I

TLC SEPARATION OF MIXTURES OF ANIONIC LEATHER DYES ON PRECOATED SILICA GEL 60 F 254

Grade in decreasing order	*Solvent systems*
I	(1) *n*-Butanol–acetic acid–water (4:1:5, upper phase)
	(2) *n*-Butanol–ethanol–water–acetic acid (6:1:2:0.05)
II	(1) Chloroform–isopropanol–water (1:3:1)
	(2) Isopropanol–ammonia (sp.gr. 0.91)–water (7:1:1)
	(3) *n*-Butanol–ethanol–ammonia (1:9) (9:2:3)
	(4) Isopropanol–ammonia (sp.gr. 0.91) (4:1)
III	(1) *n*-Butanol–acetone–water–ammonia (sp.gr. 0.91) (5:5:1:2)
	(2) Isopropanol–ammonia (sp.gr. 0.91)–water (10:1:1)
	(3) *n*-Butanol–acetic acid–water (2:1:5)
	(4) *n*-Butanol–ethanol–water (9:1:1)
	(5) Isopropanol–chloroform–water–diethylamine (50:25:20:15)
IV	(1) *n*-Propanol–ammonia (sp.gr. 0.91) (6:3)
	(2) Methanol–ethyl acetate–dilute ammonia (3 parts diluted in 7 parts water) (3:15:3)
	(3) *n*-Butanol–ethanol–water (8:1:3)
	(4) Ethyl acetate–acetic acid–water (3:1:2, upper phase)
V	(1) Isoamyl alcohol–pyridine–ammonia (sp.gr. 0.91) (8:4:1)
VI	(1) Chloroform–methanol (8:2)
	(2) Benzene–isopropanol–acetic acid (6:4:0.1)
	(3) Chloroform–ethanol–morpholine (8:1:1)
	(4) Chloroform–methyl ethyl ketone–acetic acid–formic acid (8:6:1:1)
	(5) Chloroform–methyl ethyl ketone–formic acid (6:8:1)

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was described and the solvents which gave good results for each class of dyes were reported. Leather, being a high value commodity in international commerce, is very often dyed with a mixture of dyes to get the appropriate shades and intensities to cater for aesthetic aspects and a competitive market. Earlier studies[2–5] on the thin-layer chromatography (TLC) of leather dyes were on individual dyes only and not on mixtures of dyes. As a continuation of our earlier work, studies have now been made on the TLC separation of mixtures of leather dyes with the different solvent systems which gave encouraging results, and the results are reported here.

EXPERIMENTAL

Precoated TLC plastic sheets of silica gel 60 F254 (25 cm × 20 cm, thickness 0.2 mm; E. Merck, F.R.G.) were used. The solvent systems were prepared with analytical grade reagents as reported earlier[1]. The dyes were dissolved in methanol

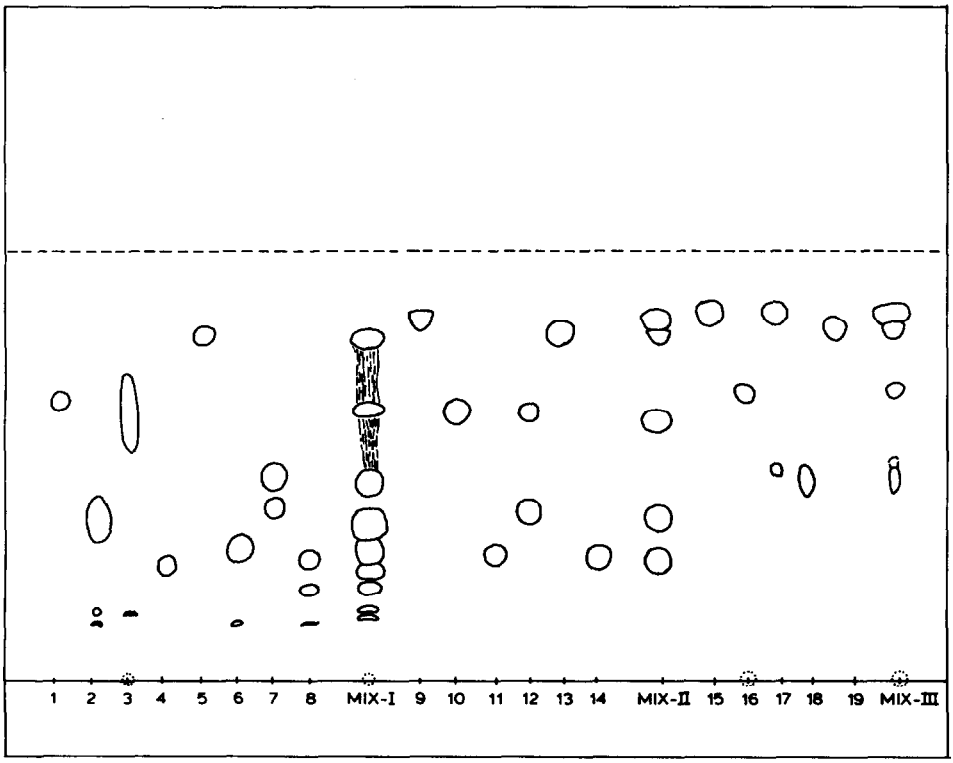

Fig. 1. TLC of anionic leather dye mixtures on precoated silica gel 60 F254 (thickness 0.2 mm) in the solvent system *n*-butanol–acetic acid–water (4:1:5, v/v, upper phase). Dyes: 1 = Polar Brilliant Blue GAW; 2 = Metalan Dark Blue; 3 = Neolan Violet Brown BX; 4 = Xyline Fast Green B; 5 = Metalan Red S-BR; 6 = Metalan Bordeaux S-B; 7 = Polar Red 3B; 8 = Metalan Brown S-GL; 9 = Cibalan Olive 3 BL; 10 = Metalan Yellow 5 RL; 11 = Ranomil Brilliant Red 3 BN; 12 = Sandopal Blue; 13 = Dermalight Yellow GLN; 14 = Derma Brilliant Red 3B; 15 = Dermalight Yellow 2RL; 16 = Chlorozol Orange Brown XS; 17 = Dermalight Orange RLN; 18 = Chlorozol Green BNS; 19 = Dermalight Grey 2 BL. Dyes 1, 4, 7, 11, 12 and 14 are acid, 2, 3, 5, 6, 8, 9, 10, 13, 15, 17 and 19 are premetallized and 16 and 18 are direct. Mixtures: I, dyes 1–8; II, 9–14; III, 15–19.

(0.1%) and each was spotted 2 cm above the edge of the TLC plates and as mixtures, and developed for 10 cm at room temperature.

RESULTS AND DISCUSSION

Generally in the leather industry, as in any other industries, mixtures of dyes are used. The leather dyes can be classified broadly into (i) anionic (acid, direct and premetallized) (ii) cationic (basic) and (iii) reactive dyes, and dyes of the same group with similar penetration power are used as mixtures for dyeing. The earlier studies carried out by Wada *et al.*[2], Sagala and Studniarski[3], Biedermann and Golz[4], Tzicas[5] and by us[1] were on individual dyes and not on mixtures of dyes. In the present studies, the TLC of 19 anionic dyes —6 acid, 2 direct and 11 premetallized— was carried out on precoated silica gel 60 F254, individually and also as mixtures consisting of acid and premetallized, and direct and premetallized, dyes. Twenty-one solvent systems[1] which gave better results for the TLC identification of various leather dyes —acid, direct and premetallized— were used. The separating power of each solvent system as indicated by its resolution of the dye mixture was studied and the solvent systems are classified in decreasing order of their resolving power for the dye components present in the mixtures (Table I). The system *n*-butanol–acetic acid–

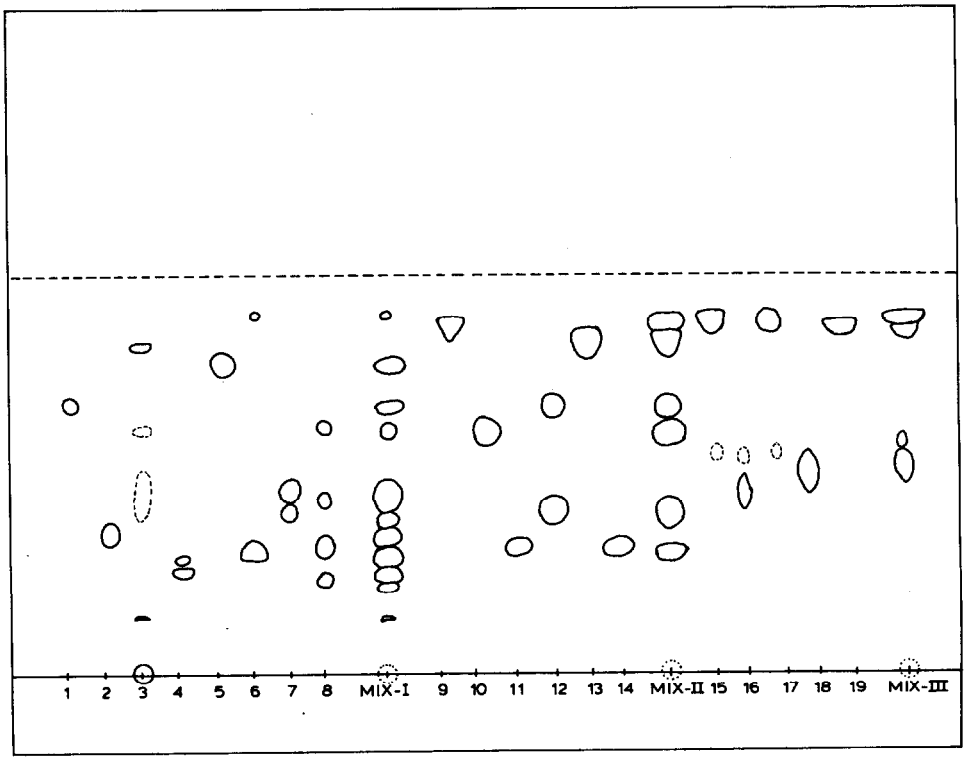

Fig. 2. TLC of mixtures of anionic leather dyes on precoated silica gel 60 F254 in the solvent system *n*-butanol–ethanol–water–acetic acid (6:1:2:0.05, v/v). Other details as in Fig. 1.

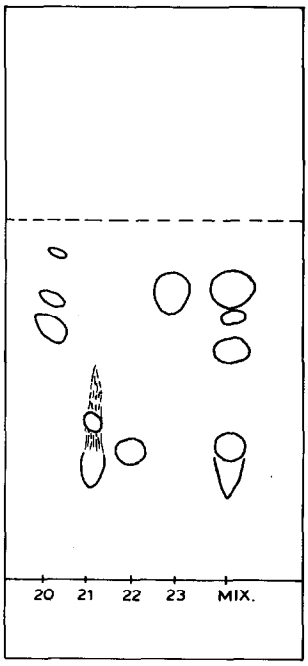

Fig. 3. TLC of a mixture of basic leather dyes on precoated silica gel 60 F254 in the solvent system *n*-butanol–acetic acid–water (4:1:5, v/v, upper phase). Dyes: 20 = Malachite Green; 21 = Methylene Blue; 22 = Fast Rubine 4 BN; 23 = Auromine O.

water (4:1:5, v/v) (upper phase) and *n*-butanol–ethanol–water–acetic acid (6:1:2:0.05, v/v) gave the best resolution of mixtures of anionic leather dyes (Figs. 1 and 2).

TLC of a mixture of four basic dyes was carried out using *n*-butanol–acetic acid–water (4:1:5, v/v, upper phase), ethyl acetate–acetic acid–water (3:1:2, v/v) and *n*-butanol–acetone–water–ammonia (sp.gr. 0.91) (5:5:1:2, v/v). Good separation of the dye components was obtained with the first of these systems (Fig. 3).

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