4.2.3 *Synthesis of the amide dye-l-lysine* 10

The dye (0·6 g, 1·7 mmol) was dissolved in a mixture of DMF (150 ml) and triethylamine (0·28 ml, 2·0 mmol) and to this was added ethyl chloroformate (0·2 ml, 2·0 mmol) while keeping the mixture at a temperature of −5°C. After stirring for 10 min, a solution of the lysine-copper complex (0·3 g, 0·85 mmol) in cold water (40 ml) was added. The mixture was stirred at room temperature overnight and the yellow solid was filtered off, washed with water and ethanol, and dried.

*Removal of copper.* The crude solid was dissolved in 0·1 M EDTA (50 ml) when a yellow product precipitated on cooling. This was filtered off and washed with water and ethanol and dried (0·48 g, 62%). Part of the solid (0·2 g) was dissolved in DMF with a few drops of 6 M HCl. A 4 M solution of NaOH was added until a precipitate formed. The yellow solid was filtered off, washed with water and ethanol and dried to give the amide 10 (0·18 g, scaled up yield 56%) m.p. 223 224°C; ν max (KBr disc) 3400, 1638 cm⁻¹. Found: C, 59·35; H, 5·7; N, 18·1%. C₂₃H₂₆N₆O₄·H₂O requires C, 58·97; H, 5·98; N, 17·94%.

## 5 CONCLUSIONS

It has been shown that carboxylic acid dyes can be reactively dyed on wool and nylon-6,6 using ethyl chloroformate as activating agent. Reaction of the dyes with the model amines cyclohexylamine and lysine confirms that amide formation can occur via reaction with the free amino groups in the polymers, thus giving covalent linkage of the dye to the polymer. These bonds would be sufficiently strong to withstand the alkaline conditions used in the ISO 105 CO3 test, as indeed was the case.

Dyes with no sulphonate acid groups gave the best results, which suggests that the preferred carboxy dyes would not have too great an affinity for wool and nylon-6,6, which would imply that better levelness and less uneven dyeings could be achieved.

The low pH necessary for fixation is also an advantage since it means that there is less wool damage.

It is noteworthy that the dyes containing sulphonate groups (i.e. 1a–3a) did not fix on wool and nylon-6,6, even though they formed amides with cyclohexylamine. The possibility of extending this reactive system to the practical reactive dyeing of polyamide fibres is the subject of further study, and will be reported in a future publication.