EXPERIMENT Y5
TLC OF AROMATIC COMPOUNDS

This experiment is an introduction to the use of TLC, a powerful and convenient technique for analysis of organic compounds. This exercise is an artificial one, but you will see typical applications of TLC later. Read 'Notes on Thin Layer Chromatography' (next page) for full details of the experimental procedure. The experiment takes approximately one lab. period.

The following solutions are provided:

(i) *ca.* 5% *p*-nitroaniline in acetone
(ii) *ca.* 5% *o*-nitroaniline in acetone
(iii) *ca.* 5% 1-naphthol in acetone
(iv) *ca.* 20% naphthalene in acetone

Plastic-backed TLC plates, cut to the appropriate size, are also provided. The plates (Polygram Sil G/UV_{254}) are coated with silica gel which is impregnated with a fluorescent indicator. The four solutions will be analysed on the silica plates using two different solvents.

Procedure

1 Make at least four capillary applicators by drawing out melting point tubes. Mark the plates with a pencil, and apply a spot of each of the four solutions to one plate.

2 Prepare a solvent tank using a beaker, a strip of filter paper, and a clock-glass. Add dichloromethane to a depth of about 2 mm. Place the plate in the tank and allow the solvent to run almost to the top of the plate. Remove the plate, mark the position of the solvent front with a pencil, and allow the solvent to evaporate.

3 Apply the four solutions to a second plate, and elute the plate in a tank containing a 1:9 mixture of ethyl acetate and dichloromethane.

4 Examine the two plates under a UV lamp (**do not look directly at the lamp**):
(i) using a low-pressure mercury-vapour lamp, emission mainly at 254 nm
(ii) using a 'black-light' UV lamp, emission at *ca.* 350 nm
Mark lightly in pencil the positions of any spots seen.

5 Place the two plates in a beaker containing a few crystals of iodine, cover the beaker with a clock glass, and leave until visible spots are formed (faint with naphthalene). If necessary, the beaker may be warmed gently on a water bath (**in the fume-cupboard**) to increase the concentration of iodine vapour in the beaker.

6 Make a full-scale sketch of the developed chromatograms, identify the spots, and note the colour of the spots, if any,
(i) before development (*i.e.* by visual inspection)
(ii) under both UV lamps, and
(iii) after development with iodine.

7 Determine the R_f value for each of the four compounds after elution in dichloromethane, and in ethyl acetate/dichloromethane, and tabulate the results.

Questions

1 Comment on the difference in R_f values for *o*- and *p*-nitroaniline.

2 Why does naphthalene have a much higher R_f value than 1-naphthol?

3 Why is the R_f value of 1-naphthol greater when ethyl acetate is present in the eluting solvent?

4 Why are naphthalene and 1-naphthol invisible under the 350 nm 'black-light' UV lamp?