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Separation of acrylate, methacrylate, and tiglate ions by paper chromatography

A solution of phenol and water is an extremely useful solvent for both paper and thin-layer chromatography and has been used as such for years. The effectiveness of this solvent can be enhanced, in certain instances, by the addition of a third component.

Although phenol and water will not begin to separate sodium acrylate from sodium methacrylate, the addition of methyl acetate to the system changes the results significantly. This modified solvent will also effect the separation of sodium methacrylate from sodium tiglate, the free acids of which are often found together in the defensive scent fluid of certain ground beetles[1].

A convenient preparation for this solvent is to mix 30 ml of liquefied phenol (88%), 10 ml of methyl acetate free of any acid, and 3 ml of distilled water. At ordinary room temperatures only one layer should be obtained. A chamber equilibration time of 1 h is adequate but a minimum solvent flow on the paper of 15 cm, requiring about 3 h, is needed to achieve a clean separation.

After the run the paper should be dried for 1 h in a stream of warm air before being dipped in 0.015% Methyl Red in 2-propanol or 95% ethanol. Within 1 min, yellow spots will appear on the pink background and should be marked immediately as they tend to fade in time. Alternatively a dip of 0.03% dichlorofluorescein in 95% ethanol followed by viewing under UV light can be used for detection. This second method is more sensitive, being able to detect spots applied from 0.03 *M* solutions of any of the three salts. However, this sensitivity also reveals streaking below each spot and in addition causes the spot to appear somewhat larger than those disclosed by Methyl Red.

Even though less sensitive, Methyl Red picks out just the core of each spot and none of the streaking, thus improving the apparent separation of the individual ions. With the phenol–methyl acetate–water solvent already described, R_F values of 0.22 for sodium acrylate, 0.32 for sodium methacrylate, and 0.41 for sodium tiglate are commonly observed. Both detecting agents give about the same values while potassium salts will yield slightly higher R_F values. Whatman No. 1 paper and ascending flow were used throughout this investigation.

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1 H. SCHILDKNECHT, K. HOLOUBEK, K. H. WEIS AND H. KRAMER, *Angew. Chem., Intern. Ed. Engl.*, 3 (1964) 80.

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