

Separation of *trans*-cinnamic, α -truxillic, and β -truxinic acids by paper chromatography

α -Truxillic acid and β -truxinic acid are the two reaction products obtained from the solid-state irradiation of cinnamic acid¹⁻⁴. The dimerization reaction which leads to their formation is subject to topochemical control⁵. While irradiation of *trans*-cinnamic acid in its α crystal form invariably affords α -truxillic acid as the sole reaction product, irradiation of its β crystal form generally gives a mixture of both the β -truxinic* and α -truxillic acids⁶. The separation of the dimeric acids and the residual unchanged cinnamic acid can be effected by paper chromatography.

Sheets, 50 × 52 cm, of Whatman No. 1 paper were used. A 1% solution of the acids in ethanol or acetic acid was applied to the paper, 1 μ l at a time, until the quantity deposited reached 30 to 50 μ g. The solvent system used for elution was ethanol-water-ammonia (80:10:5)⁷. The usual elution time was 6 h corresponding to a solvent front migration of 30 cm. The sheets were dried in a circulating-air oven at 100° for 1 h and were developed by being sprayed with an indicator solution containing 300 mg indicator and 0.25 ml (30%) aqueous sodium hydroxide in 500 ml ethanol. A "Paasche" airbrush** was used for spraying the solution. It was operated at a pressure of 40 p.s.i., and kept at a distance of 15-20 cm from the surface of the paper. Of the acid-base indicators tried, bromothymol blue was found to be more sensitive than bromophenol blue and bromocresol green. It permitted the detection of the acids in amounts as small as 10 μ g. The acids showed up as yellow spots in a blue-green environment. The fluorescent indicator 4-methylumbelliferone⁸ also worked adequately. Under ultraviolet illumination acidic regions appeared as nonfluorescent spots in a fluorescent environment.

The following R_F values were obtained: cinnamic acid, 0.56; α -truxillic acid, 0.36; β -truxinic acid, 0.27. When the acids were applied in the form of their sodium salts slightly lower R_F values were obtained and the cations appeared as pronounced blue-colored spots close to the origin. Chloride ion, having an R_F value close to that of α -truxillic acid, had to be rigorously excluded from the acid mixture.

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* β -Truxinic acid is obtained exclusively if the thermal β - to α -phase transformation of the monomer is inhibited by cooling (see ref. 6).

** Manufactured by Paasche Airbrush Co., Chicago, Ill.