

The Detection of α -(Aminoalkyl)acrylates from Hydrolysis of their Phthaloyl Derivatives by a Modified Ninhydrin Test Procedure

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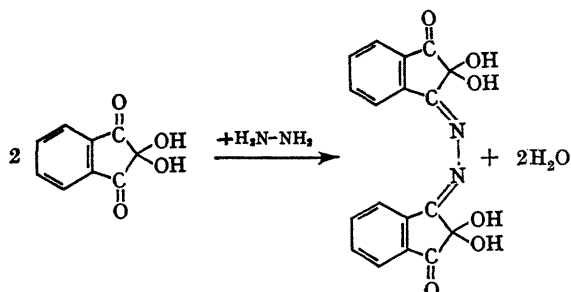
THE removal of various protecting groups such as formamido, ethoxycarbonyl, benzyloxycarbonyl, and acetyl, in efforts to produce α -substituted aminoacrylic acid polymers has hitherto failed.^{1,2} It has now been shown that hydrolysis of the protecting group to yield α -(aminoalkyl)acrylates is possible when the phthalimido-substituent is used. The

effectiveness of the hydrolysis is demonstrated by use of a modified ninhydrin test.

A range of novel α -(phthalimidoalkyl)acrylates were prepared from the respective phthalimidoalkyl halides *via* a conventional malonic ester route and by use of the Mannich reaction. The products were purified by preparative

chromatography. Derivatives of ethyl acrylate with α -phthalimido-, α -phthalimidoethyl, α -phthalimidobutyl, and ethoxy- α -phthalimidoethyl as substituents, were prepared.

The phthalimido-substituent was hydrolysed with hydrazine hydrate and the presence of free amino-compounds was tested with ninhydrin reagent modified since the presence of an excess of hydrazine resulted in a false negative result.



In neutral or acidic solution hydrazine condenses at both amino-groups with *o*-hydroxy-aldehydes and ketones to produce crystalline light yellow, insoluble Schiff bases; it probably undergoes this reaction with ninhydrin.

The interference of hydrazine in the test was eliminated by the dropwise addition of aqueous sodium nitrite to an acidified solution. Care was required since the nitrous acid formed would react with any free amine after all the hydrazine had disappeared. The reagent solution was therefore kept, and the disappearance of hydrazine was tested for by use of the aldazine reaction,³ *i.e.* by spotting the test solution on salicylaldehyde paper. Hydrazine was absent when no yellow fluorescence occurred under u.v. light.

The solutions were then made alkaline and tested with ninhydrin in the usual way.⁴ All the prepared α -substituted acrylates gave a strong positive reaction.

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¹ R. Adams, J. L. Johnson and B. E. Englund, *J. Amer. Chem. Soc.*, 1950, **72**, 5080.

² M. Frankel and M. E. Reichmann, *J. Chem. Soc.*, 1952, 289.

³ F. Feigl, "Spot Tests in Organic Analysis," Elsevier Publishing Co., 1960, 6th edn., p. 410.

⁴ F. Feigl, ref. 3, p. 294.