

radicals are not used in initiating polymerization chains (unpublished results). The efficiency of initiation is independent of the concentration of monomer if its concentration is not small. The failure of the scavenger and the monomer to capture all the radicals can be ascribed to a cage effect. The radicals are generated in pairs and therefore their direct interaction is favoured.

The actual mechanism of the reaction between  $\alpha, \alpha$ -diphenyl- $\beta$ -picrylhydrazyl and radicals is not certain<sup>5</sup>; but it is hoped to obtain evidence using the carbon-14 compound. This material has been synthesized and will be used for seeking the various possible products.

Details of these and other experiments designed to study the action of radical scavengers will be published later. I thank Prof. H. W. Melville for his interest in this work.

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<sup>2</sup> Walling, C., *J. Polymer Sci.*, **14**, 214 (1954).

<sup>3</sup> Meeting at New York in September 1954 of the Division of Polymer Chemistry of the American Chemical Society.

<sup>4</sup> Bevington, J. C., *J. Chem. Soc.*, 3707 (1954).

<sup>5</sup> Wild, W., *Disc. Farad. Soc.*, **12**, 127 (1952).

### Use of Triethylammonium Buffers in Ion-Exchange Chromatography and Electrophoresis

In preparative ion-exchange chromatography or electrophoresis of mixtures of low-molecular non-volatile compounds, it is essential to use buffer salts that are easily removed. Many methods have been employed or proposed for desalting. Of these, only evaporation of the buffer can, in our opinion, be considered as entirely satisfactory, since it introduces no other operation than the one required for the removal of the solvent. The number of volatile buffers in use so far is much limited, and those buffers do not meet all demands.

For some time, buffers made from triethylamine have been used in ion-exchange chromatography and electrophoresis at this Institute. Trimethylammonium salts may serve the same purpose, but we have found the triethylammonium salts preferable since triethylamine, due to its higher boiling point, is more convenient to handle and is easier to purify than is trimethylamine. Triethylammonium formate, acetate, bicarbonate and carbonate are all volatile and cover the approximate pH ranges 3-6 and 7-12. In many respects these buffers are superior to all volatile buffers used so far (pyridinium<sup>1</sup>, collidinium<sup>2</sup> and ammonium<sup>3</sup> acetates and formates). They have the following advantages: (1) They can be removed by lyophilization. (2) Ultra-violet absorption can be measured without prior removal of the buffer. (3) A number of reactions proceed without complications in the presence of these buffers. Particularly noteworthy is the fact that little or no interference takes place in the ninhydrin reaction according to Moore and Stein<sup>4</sup> when aliquots of 0.1-0.5 ml. of column fractions are taken if the concentration of the amine is 0.1 N or less. Much

larger amounts of the amine may cause too high blank values (possibly due to traces of impurities). Disturbing background colour also results on paper containing triethylammonium buffer when the paper is strongly heated. (4) They are stable towards oxidation (collidinium salts are anodically oxidized).

It is obvious that much time and labour may be saved when these buffers can be used for fractionation of mixtures containing both ninhydrin-positive and ultra-violet absorbing material. Excellent fractionations of mixtures of amino-acids, in free and bound form, and purine and pyrimidine compounds have been obtained on 'Dowex 50' by the use of triethylammonium formate and acetate.

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### Preparation of Hexachlorocyclohexane with 18-20 per cent Gamma-Isomer Content

In Greece, hexachlorocyclohexane was first synthesized during 1948, by the chlorination of benzene in white demijohns in the presence of sunlight. This method was convenient due to the clear sky of the country.

The hexachlorocyclohexane so prepared was examined by La Clair's method, namely, rate of dehydrochlorination at 0°C., and it was found that it contained 8-10 per cent  $\gamma$ -isomer.

It is well known that the insecticidal activity of hexachlorocyclohexane depends upon its content of  $\gamma$ -isomer, and for this reason we investigated the possibility of increasing the  $\gamma$ -isomer content of our product. Chlorinations under different conditions were carried out, and it was found that when the chlorination took place in yellow or red demijohns, instead of white ones, the product contained 18-20 per cent  $\gamma$ -isomer. This increase in  $\gamma$ -isomer content is attributed to selective chlorination favouring the formation of the  $\gamma$ -isomer.

The chlorination of benzene in monochromatic light must be further investigated in order to find out the real reason for the formation of hexachlorocyclohexane with higher  $\gamma$ -isomer content. Aiming at the commercial production of hexachlorocyclohexane on a higher scale, we carried out the chlorination of benzene inside closed Pfaudler vessels, using fluorescent lamps and different temperatures. From this work it was found that if the chlorination of benzene was carried out at a temperature of 35-45°C. and mercury radiation 2537 Å. with a fluorescent substance (the lamp contains argon under 8 mm. mercury pressure), zinc orthosilicate (for green light) and calcium tungstate (for blue light), then the product formed contains 18-20 per cent  $\gamma$ -isomer.

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