

One of us (P. A. G.) desires to express his thanks to the Institute of Applied Scientific Research (I.R.S.I.A.), Brussels, for a fellowship, which has enabled him to do this work.

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<sup>1</sup> Cf. Djerassi, C., *Chem. Rev.*, **43**, 271 (1948).

<sup>2</sup> Hebbelynck, M., and Martin, R. H., *Bull. Soc. Chim. Belg.*, **59**, 193 (1950).

<sup>3</sup> Cf. Noyes, jun., W. A., and Leighton, Ph. A., "The Photochemistry of Gases", 84 (A.C.S. Monograph 86, N.Y., 1941).

<sup>4</sup> Cf., for example, the recent results of Ritchie, M., and Winning, W. I. H., *J. Chem. Soc.*, 3579 (1950).

<sup>5</sup> Cf. the experiments on prepyrolysis of acetaldehyde (Niclauss, M., Goldfinger, P., and Letort, M., *C.R. Acad. Sci., Paris*, **229**, 437 (1949), based on the same idea.

<sup>6</sup> Semenov, N., "Kinetics of Chain Reactions" (Oxford, 1935).

<sup>7</sup> Buu-Hoi, Ng., *Lieb. Ann.*, **556**, 1 (1944).

<sup>8</sup> Schmid, H., and Karrer, P., *Helv. Chim. Acta*, **29**, 573 (1946).

<sup>9</sup> The detailed results of this investigation will be published in the *Bull. Soc. Belg.* and in a thesis to be presented by one of us (P. A. G.) at the University of Brussels.

#### Dangers Attendant on the Use of N-Halogenimides in Organic Reactions

It is our duty, we believe, to direct the attention of research chemists to a tragic accident which should stand as a warning to all users of N-halogenimides. Our collaborator and friend, Dr. H. De Graef, who was a man of great experience and skill, was carrying out the chlorination of xylene by means of 1,3-dichloro-5,5-dimethylhydantoin, when a violent explosion occurred. Wounded and badly burned, De Graef survived only three days.

It is essential to remember that some of the reactions of N-halogenimides can be extremely violent even in the absence of peroxide. Several such reactions have been observed in this laboratory, namely:

- N-chlorosuccinimide + benzylamine
- N-chlorosuccinimide + aliphatic alcohols
- 3-nitro-N-bromophthalimide + tetrahydrofurfuryl alcohol.

Among cases reported in the literature the following may be mentioned here:

- N-bromosuccinimide + hydrazine hydrate<sup>1</sup>.
- N-bromosuccinimide + diallyl sulphide<sup>2</sup>.
- N-bromosuccinimide + aniline<sup>3</sup>.

In some cases we have noticed a vigorous evolution of chlorine or bromine in reactions carried out with 1,3-dichloro-5,5-dimethylhydantoin and 3-nitro-N-bromophthalimide respectively.

Peroxides must, of course, be handled with great care, and attention is directed, once more, to the danger of using chloroform, as recommended in the first edition of "Organic Syntheses"<sup>4</sup>, for the recrystallization of benzoyl peroxide.

Every possible precaution should therefore be taken when using these reagents, and reactions of this type should be done on a small scale only.

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<sup>1</sup> Hirst, E. L., and Macbeth, A. K., *J. Chem. Soc.*, 2169 (1922).

<sup>2</sup> Backer, H. J., Stevens, W., and Dost, N., *Rec. Tr. Chim. Pays-Bas*, **67**, 451 (1948).

<sup>3</sup> Mousseron, M., Winternitz, F., and Manon, G., *Bull. Soc. Chim. Fr.*, 953 (1947).

<sup>4</sup> "Organic Syntheses", **1**, 432 (1941).

#### A Colour Reaction distinguishing between Adrenaline and nor-Adrenaline on Paper Chromatograms

IN recent studies we were concerned with the detection of traces of nor-adrenaline in preparations of adrenaline derived from natural sources. Colorimetric methods<sup>1,2</sup> proved to be of limited value with low concentrations of nor-adrenaline; but excellent results were obtained with paper chromatography, using modifications of the methods described by James and others<sup>3,4</sup>. The adrenaline samples are dissolved in methanol containing a minimum quantity of hydrochloric acid, and then transferred to paper strips for descending chromatography, or to circular disks of filter paper for chromatography by Rutter's technique<sup>5</sup>. Water-saturated phenol is used as the developing solvent in an atmosphere of hydrochloric acid<sup>4</sup>. The paper chromatograms are then dried and sprayed with an appropriate colour reagent.

The commonly-used ferricyanide colour reagent<sup>3</sup> produces browns and pinks which are not very sharply defined, especially when microgram quantities of nor-adrenaline are being sought in the presence of milligram quantities of adrenaline. More satisfactory results can be obtained by spraying the chromatograms with a solution of 0.5 gm. sodium β-naphthoquinone-4-sulphonate dissolved in 100 ml. of 0.2 M borate buffer at pH 8.9, the reagent having a final pH of 8.6. The solution should be freshly prepared just before use. With this reagent nor-adrenaline develops an intense blue colour slowly over a period of 15–30 min. Adrenaline produces a pink colour, while dihydroxyphenylalanine gives a yellow colour which gradually changes to a blue-grey. This colour difference should be of value in distinguishing dihydroxyphenylalanine from nor-adrenaline, since their  $R_F$  values are quite close<sup>1</sup>. At lower pH values (8.3–7.5), the paper background becomes badly discoloured, although the nor-adrenaline colour develops more rapidly. Exposure of the paper chromatogram to ammonia may be necessary before spraying if excessive amounts of acid are present.

This colour reaction is highly sensitive, being capable of detecting less than one microgram of nor-adrenaline per square centimetre of paper.

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<sup>1</sup> Von Euler, U. S., and Hamburg, U., *Acta Physiol. Scand.*, **19**, 74, (1949).

<sup>2</sup> Auerbach, M. E., and Angell, E., *Science*, **109**, 537 (1949).

<sup>3</sup> James, W. O., *Nature*, **161**, 851 (1948).

<sup>4</sup> Goldenberg, M., Faber, M., Alston, E. J., and Chargaff, E. C., *Science*, **109**, 534 (1949).

<sup>5</sup> Rutter, L., *Analyst*, **75**, 37 (1950).

#### Lamellar Compounds of Graphite with Chromyl Chloride and Chromyl Fluoride

THE ability of graphite to form lamellar compounds by occluding certain other substances (fluorine, boron, oxygen, potassium, rubidium, caesium and ferric chloride) between its layer planes has been known for a considerable time. No new compounds of this type have, however, been reported for the past fifteen years, though attempts have been made to intercalate other volatile compounds in graphite. Thus Rüedorff and Schulz<sup>1</sup>, who studied the ferric