

3. No evidence that use of an optically active peroxide will cause an asymmetric synthe-

sis in vinyl polymerization has been obtained.
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NOTES

Trimolecular Acetone Peroxide in Isopropyl Ether

BY FRED ACREE, JR., AND H. L. HALLER

During the past few years several notes have appeared in the literature citing explosions of isopropyl ether.¹ In all cases the ether had stood for several years and the explosion hazard was attributed to the formation of peroxides. So far as we have been able to learn, the responsible peroxide has not been isolated or described.

Recently, while an organic compound was being crystallized from isopropyl ether,² the mother liquor was evaporated on the steam-bath with the aid of a stream of air to about one-third of its original volume. On standing at room temperature, a crop of crystals, resembling common salt, separated. They melted at 94°. On recrystallization from methyl alcohol the melting point was 98°. The compound sublimed readily at room temperature, liberated iodine from potassium iodide solution, and exploded on percussion or when heated on a platinum spatula. A molecular weight determination by the Signer method as modified by Clark³ gave a value of 221. The compound possessed all the properties of trimolecular acetone peroxide,⁴ and a mixture melting point determination of the unknown peroxide with an authentic sample of trimolecular acetone peroxide showed them to be identical.

Trimolecular acetone peroxide was readily obtained in crystalline form from four out of five lots of isopropyl ether, all of which gave qualitative tests for peroxides, when the ether was evaporated to one-third of its original volume on the steam-bath with a stream of air. The fifth

(1) (a) E. F. Degering, *J. Chem. Ed.*, **13**, 494 (1936); (b) B. L. Herrington, *Ind. Eng. Chem., News Ed.*, **20**, 1458 (1942); (c) G. T. Morgan and R. H. Pickard, *Chem. and Ind.*, **55**, 421 (1936); (d) E. C. Williams, *J. Soc. Chem. Ind.*, **55**, 580 (1936).

(2) The ether was a commercial grade that had stood in the laboratory in clear glass bottles for at least five years. It was not purified before use.

(3) E. P. Clark, *Ind. Eng. Chem., Anal. Ed.*, **13**, 820 (1941).

(4) R. Wolfenstein, *Ber.*, **28**, 2265 (1895).

lot gave a viscous sirup from which no crystals of the trimolecular acetone peroxide were obtained.

It is of interest to note that Radulesco⁵ found that the photochemical oxidation of isopropyl ether was slightly autocatalytic and produced acetone, water, peroxides, acids, and gum; and that Brown⁶ has shown the formation of acetone when the ether mixed with oxygen is passed over a silver catalyst. Whether other peroxides are also present in isopropyl ether has not been determined. Trimolecular acetone peroxide is possibly responsible for the peroxide test found by Redemann⁷ with isopropanol.

(5) G. Radulesco, *Ann. combustibile liquides*, **13**, 1071 (1938).

(6) R. L. Brown, U. S. Patent 2,246,569, June 24, 1941.

(7) C. E. Redemann, *THIS JOURNAL*, **64**, 3049 (1942).

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Some Fluorinated Compounds of Possible Chemotherapeutical Interest

BY E. BOGRACHOV

In the course of experiments on lipophilic chemotherapeuticals¹ an investigation of fluorinated compounds was undertaken. As these experiments cannot be continued at present, a brief description of three of the new substances prepared may be permitted. They were obtained in well-crystallized and undoubtedly pure form. While the hydrogen and nitrogen analyses (Dumas) were satisfactory, the carbon values were throughout too low, most probably due to the formation of the volatile, stable carbon tetrafluoride.²

N-(*o*-Fluorobenzoyl)-1-amino-4-(benzene-azo)-naphthalene.—4-(Benzeneazo)-1-naphthylamine (12.3 g.) was dissolved in dry chloroform (100 cc.), and the solution, after addition of pyridine (4 g.), cooled to 0°. At this tempera-

(1) Bergmann, *et al.*, *THIS JOURNAL*, **63**, 2243, 2245 (1941).

(2) Satisfactory values can be obtained only by combustion with lead chromate and silver, not with the method usually applied in microcombustion analysis. Wallach and Heusler, *Ann.*, **248**, 243 (1888); Schiemann and Bolstad, *Ber.*, **61**, 1407 (1928).