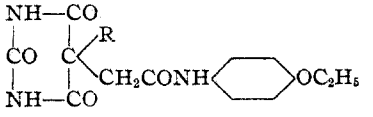
[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, YALE UNIVERSITY]

Alkylacetanilidobarbituric Acids. III. *p*-Ethoxy Derivatives

BY JOHN A. TIMM

The fact that the administration of mixtures or addition products of hypnotic and antipyretic substances produces an analgesic effect is well known.[1] This series of papers reports the preparation of 5-alkylbarbituric acids in which antipyretic and anesthetic groups replace the second hydrogen atom on the 5-carbon atom. Derivatives of acetanilide[2] and *p*-carbethoxyacetanilide[3] have been reported in the previous papers. The series has been extended in this paper to the preparation of 5-alkyl-5-*p*-ethoxyacetanilidobarbituric acids, *i. e.*, derivatives of the antipyretic, phenacetin



These derivatives, together with those reported in the first two papers of this series, are being tested pharmacologically. The results will be published elsewhere.

Experimental Part

Barbituric Acids Containing the *p*-Ethoxyacetanilido Group.—Equimolecular proportions of the appropriate 5-alkylbarbituric acid and *p*-ethoxychloroacetanilide,[4] a one and one-half molecular proportion of sodium acetate and a one-fourth molecular proportion of potassium iodide were dissolved in 70% alcohol by heating on a water-bath in a flask provided with a mechanical stirrer and a reflux condenser. The heating was continued for twenty-four hours. Crystals of the product separated during the course of the reaction. Approximately one-half of the alcohol was distilled off and the mixture cooled in an ice-bath. The products were recrystallized from absolute alcohol. They are white, crystalline solids which melt with decomposition. The usual difficulty was experienced in crystallizing the isopropyl derivative, which comes out of solution at first as a brown oil.

TABLE I

Barbituric acid, 5-*p*-ethoxyacetanilido-	Melting range, °C., with dec.	Yield, %	N Analyses, % Calcd.	Found	
5-Ethyl-	194–205	60	12.6	12.5	12.6
5-Isopropyl-	210–215	20	12.1	12.1	11.8
5-*n*-Butyl-	231–232	40	11.6	11.6	11.7
5-Isobutyl-	217–219	50	11.6	11.6	11.6
5-Isoamyl-	219–220	80	11.2	11.4	11.5
5-Allyl-	215–218	60	12.2	12.0	12.0

The author wishes to express his appreciation to Mr. Paul M. Hauser and to Mr. DeFrance Clarke, Jr., for their help in the analyses and in the preparation of the intermediates of these compounds.

Summary

The following 5-*p*-ethoxyacetanilidobarbituric acids have been prepared: 5-ethyl-, 5-isopropyl-, 5-*n*-butyl-, 5-isobutyl-, 5-isoamyl-, and 5-allyl-.

NEW HAVEN, CONN. RECEIVED JULY 25, 1939

(1) See Hepner and Frenkenberg, *Ber.*, **65B**, 123 (1932).
(2) Timm, THIS JOURNAL, **57**, 1943 (1935).
(3) Timm and Howard, *ibid.*, **58**, 1805 (1936).
(4) Bistrzycki and Ulffers, *Ber.*, **31**, 2790 (1898).

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, THE PENNSYLVANIA STATE COLLEGE]

Fluorocarbons. The Reaction of Fluorine with Carbon

BY J. H. SIMONS AND L. P. BLOCK[1]

Carbon tetrafluoride has been obtained from the reaction of fluorine with carbon.[2] Ruff and Keim isolated hexafluoroethane from the reaction products and obtained small amounts of higher boiling material, which they assumed to be mixtures of higher molecular weight fluorocarbons, but in insufficient quantity to separate and isolate the compounds. Several difficulties are encountered in the study of this reaction. Frequent and sometimes violent explosions occur; and as the higher molecular weight compounds are produced in relatively small quantities, large amounts of reaction products are necessary in order to isolate them. Ruff, Bretschneider and Ebert[3] studied these explosions in detail and found a

(1) The authors gratefully acknowledge a grant from the American Academy of Arts and Sciences which aided in financing the preparation of these compounds.

(2) Moissan, *Compt. rend.*, **110**, 951 (1890); Lebeau and Damiens, *ibid.*, **191**, 939 (1930); Ruff and Keim, *Z. anorg. allgem. Chem.*, **192**, 249 (1930).

(3) Ruff, Bretschneider and Ebert, *ibid.*, **217**, 1 (1934).