ethylbarbituric acid and related compounds described by Bousquet and Adams [THIS JOURNAL, **52,** 227 (1930)]. Through the Bart reaction, 5-aminophenyl- and 5-nitroaminophenyl-5-ethylbarbituric acids have been converted into the arsonic acids, 5-arsonophenyl- and 5-nitroarsonophenyl-5-ethylbarbituric acids. By means of hypophosphorous acid the latter were reduced to the corresponding arseno compounds.

During the research it became necessary to establish the position of the nitro group in the starting product and it was shown to be meta. In view of a recent article in this field [Rising, Shroyer and Stieglitz, *ibid.*, **55,** 2819, footnote (1933)] in which mention is made of a forthcoming communication by Rising and Pierce concerning the structure of 5-nitrophenyl-5-ethylbarbituric acid, a brief outline is given here of the method of proof used in our work. 5-Nitrophenyl-5-ethylbarbituric acid (m. p. 279–280°) [all melting points are corrected] ⟶ (nitrophenylethylmalonic acid) ⟶ α-nitrophenylbutyric acid (80% yield, m. p. 115.5–116.3°; neutral equivalent 210, calcd. 209.1) ⟶ *m*-nitrobenzoic acid (60% yield, m. p. 141–142°; identity confirmed by mixed m. p.).

5-*m*-Aminophenyl-5-ethylbarbituric acid was converted to a 5-(nitro-*m*-aminophenyl)-5-ethylbarbituric acid (dec. 330–340°) by the method of Bousquet and Adams, and by elimination of the amino group a new isomeric 5-nitrophenyl-5-ethylbarbituric acid (dec. 327–330°) was obtained. In the latter the nitro group may occupy the ortho or para position; the difficulty of hydrolyzing this compound has prevented us from establishing its structure.

BAKER LABORATORY OF CHEMISTRY
CORNELL UNIVERSITY
ITHACA, NEW YORK

MILTON T. BUSH
JOHN R. JOHNSON

RECEIVED AUGUST 2, 1933 PUBLISHED SEPTEMBER 5, 1933

THE NITRATION OF PHENOBARBITAL

Sir:

As stated in a previous publication [THIS JOURNAL, **55,** 2817 (1933)] the authors have proved that the nitration of phenobarbital

$$(C_6H_5)(C_2H_5)\underbrace{CCONHCONHC}_{}=O$$

leads to the formation of 5-*m*-nitrophenyl-5-ethylbarbituric acid.[1,2] That the product of nitration of melting point 279–280° (corr.) is the meta, and not the para, isomer was proved as follows: 8.4 g. of the nitro-ureide yielded on oxidation 1.0 g. of *m*-nitrobenzoic acid, which was identified by

---

(1) In confirmation of the findings of the present authors, Bush and Johnson have recently offered evidence for the formation of the meta rather than the para derivative.

(2) It is of interest to note that the sulfonation of phenobarbital leads to a meta derivative. (Stieglitz and Yoran, unpublished dissertation for the doctorate degree, University of Chicago, June, 1933).

means of comparing its properties and those of the corresponding amino acid with the behavior of authentic samples of these substances. The nitrophenobarbital of melting point 279–280° must therefore be a *m*-nitro compound.

Residues obtained by evaporation of the alcohol used for purification of the crude nitration product will be examined for ortho and para nitro-derivatives.

GEORGE HERBERT JONES CHEMICAL LABORATORY
THE UNIVERSITY OF CHICAGO
CHICAGO, ILLINOIS
MARY M. RISING
ALAN PIERCE

RECEIVED AUGUST 7, 1933 PUBLISHED SEPTEMBER 5, 1933

THE ACTION OF ELECTROLYTES ON WOOL FIBER

Sir:

During an x-ray investigation of the mordant dyeing of wool, an interesting phenomenon was observed. Using monochromatic radiation (Cu K_α) and mounting the fiber in a box type Laue camera in place of the usual crystal, pure wool fiber gives a very indistinct fiber diagram as contrasted with that obtained from the coarser keratin fibers [W. T. Astbury, *Trans. Faraday Soc.*, **29**, 193 (1933)] such as hair, quills and feathers. However, when this wool has been treated with certain electrolytes, two quite sharp and distinct rings appear. The inner ring is the more intense of the two, corresponding to a spacing of 4.08 Å., while the outer and lighter ring corresponds to a spacing of 3.72 Å. In the case of some of the wool samples, a broad ring quite close to the primary beam was observed corresponding to a spacing of 12.9 Å. Table I is a summary of the results obtained with all of the electrolytes that have been used. The third column indicates in a very approximate manner the visibility of the two lines (4.08 and 3.72 Å.) as observed on diagrams obtained after various treatments, relative to the type of treatment. It is interesting to note that wool which has been dyed as in a regular industrial process shows these two rings. The dye used throughout this work was commercial Orange II furnished through the courtesy of the Dupont Dye Company. NaX signifies the sodium salt of this dye. The wool fiber used was also furnished by the Dupont Dye Company.

It is suggested that the action of electrolytes, particularly those of an acid character, causes the wool protein to become crystalline to some extent and that these crystallites so formed are unoriented along the fiber axis as evidenced by the character of the rings. Table II shows the results of a calculation that would lead one to believe that these three rings are but high orders of a period of 77.8 Å. in length. The data in Table II are averages of all the observations made on many films. The rings on the