

Anal. Calcd. for $C_{15}H_{10}BrNO$: C, 56.5; H, 3.6. Found: C, 56.3; H, 3.9.

A solution of 0.5 g. of the amino ketone in 10 ml. of dilute hydrochloric acid was treated at 10° with 0.2 g. of sodium nitrite, allowed to stand for ten minutes, and then heated to boiling. The resulting 3-bromofluorenone separated from alcohol in the form of yellow plates (0.3 g.), m. p. 161–162°.

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The Stereochemistry of Coördination Number Eight. Corrections

By LOUIS E. MARCHI

Attention has been called to several serious errors which appeared in "The Stereochemistry of Coördination Number Eight."¹

On page 331, in the right hand column, the sixth line from the bottom which reads 3AB CD EF should be deleted, since it is an obvious repetition of the line above it; the seventh line from the bottom should read 2AB CD EF instead of 3AB CD EF, since the latter is not a member of a class of coördination number eight.

In Table IV, page 332, the isomer numbers for Class AB, 6c, Configuration C, should be: Optically active, 0; Optically inactive, 1; Total, 1, instead of Optically active, 2; Optically inactive, 0; Total, 2. In the same table, on page 333, the isomer numbers for Class 3AB 2c, Configuration C, should be: Optically active, 14; Optically inactive, 2; Total, 16, instead of Optically active, 20; Optically inactive, 0; Total, 20.

(1) Marchi, *THIS JOURNAL*, **65**, 329–333 (1943).

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The Reaction of Alkyl Halides with Potassium Iodide

By H. J. McDONALD, L. R. ROTHSTEIN AND H. E. ROBISON

Conant and Hussey¹ have previously described the reaction between several types of alkyl chlorides and potassium iodide. We have checked some of the results of these investigators and have extended their procedure to certain other alkyl halides.

We have found that there is no measurable reaction between either *t*-amyl chloride or *i*-butyl chloride and potassium iodide after contact times of twenty-four hours at temperatures up to 60°. The result obtained in the former case is not surprising in view of the fact that tertiary alkyl halides in general are extremely unreactive. In such second order substitution reactions, Conant¹ has shown the ease of substitution to be primary > secondary > tertiary.

On this basis it is difficult to see why the primary alkyl halide, *i*-butyl chloride, should prove

(1) Conant and Hussey, *THIS JOURNAL*, **47**, 476 (1925).

so unreactive. For although the above-mentioned workers have also shown that branching of the chain reduces reactivity by as much as 100%, their data indicate that *i*-amyl chloride would have reacted with the iodide to an extent of 50% under the same conditions of time and temperature (twenty-four hours and 60°).

The possibility that the *i*-butyl compound might have rearranged during distillation to the more unreactive *t*-butyl chloride was eliminated by refractive index measurements; found, n_D^{25} 1.4000; known, n_D^{15} 1.40096.²

(2) Timmermans and Martin, *J. chim. phys.*, **23**, 747 (1926).

DEPARTMENT OF CHEMISTRY
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3,5-Diiodo-4-(4'-hydroxyphenoxy)-hippuric Acid and 3,5-Diiodo-4-(3',5'-diiodo-4'-hydroxyphenoxy)-hippuric Acid

By CARL NIEMANN AND G. E. McCASLAND

In the course of testing a series of reactions in order to determine their usefulness for the synthesis of thyroxine containing peptides 3,5-diiodo-4-(4'-hydroxyphenoxy)-hippuric acid and 3,5-diiodo-4-(3',5'-diiodo-4'-hydroxyphenoxy)-hippuric acid have been prepared and characterized.

3,5-Diiodo-4-(4'-acetoxyphenoxy)-benzoic Acid (I).—To 10 g. of recrystallized 3,5-diiodo-4-(4'-hydroxyphenoxy)-benzoic acid,¹ m. p. 259–60°, was added 40 ml. of acetic anhydride and 2–3 drops of concentrated sulfuric acid and the mixture heated at 100° for thirty minutes. The solution was cooled, poured into ice water and, after decomposition of the acetic anhydride, the colorless solid, which had separated, was collected, washed and dried *in vacuo*. The crude product (10.8 g.) was twice recrystallized from absolute ethanol to give 9.6 g. (89%) of I, colorless needles, m. p. 231–232°.

Anal. Calcd. for $C_{15}H_{10}O_5I_2$ (524): C, 34.4; H, 1.9; I, 48.4. Found: C, 34.7; H, 2.1; I, 48.8.

Ethyl 3,5-Diiodo-4-(4'-acetoxyphenoxy)-hippurate (II). To 3.1 g. of I suspended in 20 ml. of dry benzene was added 1.35 g. of phosphorus pentachloride and the mixture gradually heated to 70°. After cooling to 10° the crystalline acid chloride was collected, at 10°, and washed with 30–60° ligroin. The vacuum dried product weighed 2.7 g. A dry ethereal solution of glycine ethyl ester prepared by the method of Fischer² was standardized, by titration with standard acid, and the requisite amount of this solution added to the dry acid chloride and the mixture allowed to stand for sixteen hours at 5°. The solid was collected, washed several times with water and dried *in vacuo* to give 1.7 g. of II, colorless powder, m. p. 144–145°. The ethereal filtrate was washed with water, dilute hydrochloric acid, and again with water and dried first over anhydrous potassium carbonate and finally over Drierite. Evaporation of the dried solution gave an additional 0.7 g. of II for a total yield of 2.4 g. (67%).

Anal. Calcd. for $C_{19}H_{17}O_5NI_2$ (609): C, 37.5; H, 2.8; N, 2.3. Found: C, 37.5; H, 3.0; N, 2.4.

3,5-Diiodo-4-(4'-hydroxyphenoxy)-hippuric Acid Monohydrate (III).—To 2 g. of II was added 40 ml. of 1 *N* sodium hydroxide in 50% ethanol and the suspension

(1) (a) C. R. Harington and G. Barger, *Biochem. J.*, **21**, 169 (1927); (b) C. Niemann and C. E. Redemann, *THIS JOURNAL*, **63**, 1549 (1941).

(2) E. Fischer, *Ber.*, **34**, 433 (1901).