

(10 g.)<sup>1</sup> in 100 cc. of methanol at 200° (330 atm.) with 3 g. of W-3 Raney nickel. The reduction was complete in four hours. The product remaining after removal of catalyst and solvent weighed 10.04 g. and melted at 123–138°.<sup>2</sup> It consisted of a mixture of isomers.

*Anal.* Calcd. for  $C_{14}H_{26}O_2$ : C, 74.29; H, 11.58. Found: C, 74.59; H, 12.01.<sup>3</sup>

The mixture of isomers (1.0 g.) was adsorbed on aluminum oxide (General Chemical Co., Reagent) from benzene solution. Elution with benzene gave 0.13 g. of crude material (m. p. 102–115°) which was obtained pure after one crystallization from Skellysolve B and melted at 123.5–124.5°.

*Anal.* Calcd. for  $C_{14}H_{26}O_2$ : C, 74.29; H, 11.58. Found: C, 74.19; H, 11.69.

Fractional crystallization of the original mixture (1.0 g.) from benzene yielded 0.41 g. of material melting at 139–145°. Further crystallizations from benzene and ethyl acetate raised the melting point to 144–145°. A mixture of the pure isomers melted at 133–143°.

*Anal.* Calcd. for  $C_{14}H_{26}O_2$ : C, 74.29; H, 11.58. Found: C, 74.05; H, 11.56.

1,2-Di-(4-ketocyclohexane)-ethane was prepared by oxidation of the crude mixture of isomers (1.0 g.) dissolved in 20 cc. of benzene and 3 cc. of acetic acid, with a solution of 4.4 g. of sodium dichromate and 5.9 cc. of concentrated sulfuric acid in 20 cc. of water at room temperature. The benzene extract of the reaction mixture yielded 0.65 g. of crude diketone melting at 89–93.5°. Several crystallizations from Skellysolve C gave a constant melting product, m. p. 94.5–96°, yield 0.45 g.

*Anal.* Calcd. for  $C_{14}H_{22}O_2$ : C, 75.64; H, 9.97. Found: C, 75.55; H, 10.19.

(1) The author is indebted to Dr. W. M. Hoehn, formerly with the George Breon Co., Kansas City, Mo., for the gift of this material.

(2) All melting points uncorrected.

(3) Analyses by K. T. Zilch and E. W. Crandall.

CHEMISTRY DEPARTMENT  
UNIVERSITY OF MISSOURI  
COLUMBIA, MO.

HERBERT H. UNGNADE

RECEIVED APRIL 12, 1948

#### Preparation of 2-Methyl-4-methoxy-5-isopropylpropiophenone and 2-Methyl-4-methoxy-5-isopropylphenylethylcarbinol<sup>1</sup>

**2-Methyl-4-methoxy-5-isopropylpropiophenone.**—Twenty-six and eight-tenths grams (0.13 mole) of 2-methyl-4-hydroxy-5-isopropylpropiophenone, which was prepared by the Fries rearrangement<sup>2</sup> of 2-isopropyl-5-methylphenylpropionate, was treated with 32.8 g. (0.26 mole) of dimethyl sulfate in aqueous sodium hydroxide to yield 21.0 g. of the desired methyl ether, b. p. 169–171° (20 mm.).

(1) Author's present address: Wallace and Tiernan Products, Inc., Belleville, New Jersey.

(2) K. W. Rosenmund and W. Schnurr, *Ann.*, **460**, 56 (1938).

*Anal.* Calcd. for  $C_{14}H_{20}O_2$ : C, 76.36; H, 9.09. Found: C, 76.20; H, 9.21.

**2-Methyl-4-methoxy-5-isopropylphenylethylcarbinol.**—To a refluxing solution containing 19.8 g. (0.09 mole) of 2-methyl-4-methoxy-5-isopropylpropiophenone in 150 ml. of absolute ethanol was added in small pieces through the top of the condenser a total of 14.5 g. (0.63 mole) of sodium. The reaction mixture was set aside at room temperature for twenty-four hours, and then 150 ml. of water were cautiously added. The alkaline solution was extracted with ether, and the ether extracts were washed with water until the ether layer was neutral to litmus. After removing the ether by distillation, the yellow oily residue was distilled under reduced pressure to yield 13.2 grams of the carbinol; b. p. 156–159° (8 mm.).

*Anal.* Calcd. for  $C_{14}H_{22}O_2$ : C, 75.67; H, 9.90. Found: C, 75.73; H, 9.89.

DEPARTMENT OF CHEMISTRY  
NEW YORK UNIVERSITY  
NEW YORK, N. Y.

PHILIP WEISS

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#### Benzyl Chloromethyl Selenide

A new compound, benzyl chloromethyl selenide, was prepared by the authors<sup>1</sup> as an intermediate in the preparation of  $\beta,\beta'$ -diseleno-dialanine. The purpose of the present report is to present some of the properties of this new compound.

**Preparation.**—Seventy-three grams of benzyl seleno was mixed with 25 g. of polyoxymethylene in a flask equipped with a mechanical stirrer. The mixture was cooled in a water-bath and saturated with dry hydrogen chloride with occasional stirring. Thirty grams of calcium chloride was added and the mixture was allowed to stand at room temperature for twenty-four hours. The solid material was removed by filtration and the orange-colored filtrate was distilled *in vacuo* collecting the fraction that boiled at 121° at 5 mm.; yield, 53.3 g., 57%.

*Anal.* Calcd. for  $C_8H_9ClSe$ : C, 43.7; Cl, 16.2, Se, 36.0. Found: C, 43.4; Cl,<sup>2</sup> 16.0; Se,<sup>3</sup> 36.1.

**Physical Properties.**—It forms long pale-yellow needles below 37° and a yellow liquid with disagreeable odor above 38°; b. p. 121° at 5 mm.; sp. gr. 1.034<sup>4</sup>; refractive index, 1.5587<sup>5</sup>. It is soluble in acetone, benzene, ethyl ether, petroleum ether and ethyl alcohol but insoluble in water.

**Chemical Properties.**—Cleavage with hydriodic acid produces benzyl iodide, m. p. 24.1°; chloromethyl iodide, b. p. 109° and selenols (qualitative identification with mercuric chloride).

DEPARTMENT OF CHEMISTRY  
UNIVERSITY OF NEVADA  
RENO, NEVADA

LORING R. WILLIAMS  
ABRAHAM RAVVE

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(1) L. R. Williams and A. Ravve, *THIS JOURNAL*, **70**, 1244 (1948).

(2) O. Kamm, "Qualitative Organic Analysis," 2nd Ed., John Wiley and Sons, Inc., New York, N. Y., 1932, p. 200.

(3) W. O. Robinson, H. C. Dudley, K. T. Williams and H. G. Byers, *Ind. Eng. Chem., Anal. Ed.*, **6**, 274 (1934).