

1,1-Dimethylbutyl-N-Phenyl Urethan

JERRY W. RAMSEY¹

The Pennsylvania State University, Pottsville, Pa.

THE PHENYLURETHAN of 2-methyl-2-pentanol has been prepared. The original investigator (1) of 2-methyl-2-pentanol erroneously reported having prepared this urethan. In that method, one which is not applicable to tertiary alcohols, the carbinol was boiled with the phenyl isocyanate for a few minutes and the product was recrystallized from alcohol and acetone. The resulting product was the diphenyl urea.

Since the Tables for Identification of Organic Compounds (4) list only three derivatives for the alcohol: the 3,5-dinitrobenzoate; the benzoate; and allophanate; the phenylurethan should be a useful addition to the list. A slight modification of the method of McElvain (3) for preparing the phenylurethan of a tertiary alcohol was used.

EXPERIMENTAL

Into a 100 ml. round bottom flask, connected to a reflux condenser by a ground glass joint, was placed 1.5 grams of 2-methyl-2-pentanol which is commercially available (2). Two grams of phenyl isocyanate and 0.1 gram of sodium acetate were added. A calcium chloride drying tube was fitted to the top of the condenser. The flask was placed in a boiling water bath, where it was kept for four hours. A white precipitate appeared shortly after the beginning of the reaction and slowly increased throughout

¹Present address: U. S. Department of the Interior, Bureau of Mines, Schuylkill Haven, Pa.

the heating period. Octane, boiling 124° C., was added to the reaction mixture to dissolve the phenylurethan and separate it from the insoluble diphenyl urea formed by dehydration of some of the tertiary alcohol. The octane solution was allowed to come to a boil and filtered. The solution was evaporated down to 5 ml., and allowed to cool. The phenylurethan separated as needles. These were filtered, washed carefully with a small portion of ice cold octane, and recrystallized three times from octane. The final product was filtered with suction and air dried for two days until a constant melting point of 52° C. was obtained. The final yield of a dense silvery mass of very fine needles was 0.73 gram (22.4% of theoretical). *Anal.* Calcd. for C₁₃H₁₉O₂N: C, 70.55%; H, 8.65%. Found: C, 70.49%; H, 8.62%.

LITERATURE CITED

- (1) Deschamps, A., *J. Am. Chem. Soc.* **42**, 2670 (1920).
- (2) "Fisher Chemical Index 62-C", p. 202, Fisher Scientific Co., New York, 1961.
- (3) McElvain, Samuel M., "The Characterization of Organic Compounds," 2nd ed., pp. 199-200, MacMillan Co., New York, 1953.
- (4) Tables for Identification of Organic Compounds, p. 29, Chemical Rubber Publishing Co., Cleveland, Ohio, 1960.

RECEIVED for review October 23, 1963. Accepted February 4, 1964. This work was supported by the Central Research Fund. The Pennsylvania State University.

Some Butanediol Derivatives

N. SHARGHI, I. LALEZARI and A. SHAFII

University of Tehran, Tehren-Iran

A number of butanediol derivatives such as 2-(*p*-chlorophenyl)-3-methyl-2,3-butanediol (phenaglycodol) were found to possess anticonvulsant effects and, as a further investigation five new analogs of phenaglycodol have been prepared. These analogs are:

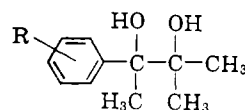
2-(*p*-xenyl)-3-methyl-2,3-butanediol,
2-(*p*-methylmercaptophenyl)-3-methyl-2,3-butanediol,
2-(*p*-phenylmercaptophenyl)-3-methyl-2,3-butanediol,
2-(*m*-trifluoromethylphenyl)-3-methyl-2,3-butanediol,

and

2-(*p*-trifluoromethylphenyl)-3-methyl-2,3-butanediol.

IN VIEW OF the fact that some butanediol derivatives have an anticonvulsant effect, we have prepared a number of phenaglycodol (I) analogs, the medicinal effects of which will be the subject of another publication.

In five new synthesized analogs (Table I), we have replaced the *p*-chlorophenyl group in I by xenyl (II), *p*-methylmercaptophenyl (III), *p*-phenylmercaptophenyl (IV), *m*-trifluoromethylphenyl (V) and *p*-trifluoromethylphenyl (VI).



I, R = *p*-Cl
II, R = *p*-C₆H₅
III, R = *p*-SCH₃
IV, R = *p*-SC₆H₅
V, R = *m*-CF₃
VI, R = *p*-CF₃

The syntheses of II, III, and IV commenced with Friedel-Crafts reaction of the appropriate aromatic hydrocarbon