



A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior to performing a reaction, a thorough risk assessment should be carried out that includes a review of the potential hazards associated with each chemical and experimental operation on the scale that is planned for the procedure. Guidelines for carrying out a risk assessment and for analyzing the hazards associated with chemicals can be found in Chapter 4 of Prudent Practices.

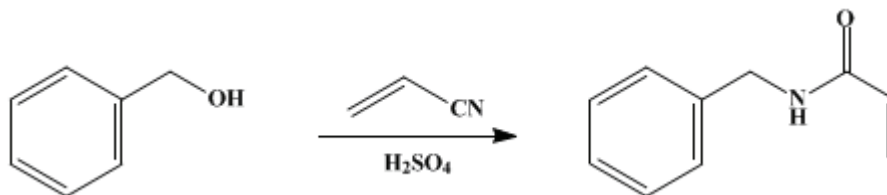
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These paragraphs were added in September 2014. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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N-BENZYLACRYLAMIDE

[Acrylamide, N-benzyl-]



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1. Procedure

In a 1-l., three-necked, round-bottomed flask equipped with a sealed Hershberg stirrer,² a 125-ml. dropping funnel, and a thermometer is placed 200 g. (250 ml., 3.78 moles) of acrylonitrile (Note 1). The flask is immersed in an ice-water bath, and then 75 ml. of concentrated sulfuric acid is added dropwise over a period of about 1 hour while the temperature is maintained at 0–5°. From a clean dropping funnel (Note 2), 108.1 g. (105 ml., 1.0 mole) of benzyl alcohol (Note 3) is added dropwise over about 1 hour at the same temperature. The clear, yellow mixture is held below 5° for about 3 hours longer and is then allowed to warm slowly to room temperature. After 2 days of stirring at room temperature the mixture is poured into a 2-l. separatory funnel containing about 1 l. of water and chopped ice. The mixture is shaken thoroughly and the resulting oil is taken up with 200 ml. of ethyl acetate. The aqueous phase is separated and extracted twice more with 200-ml. portions of solvent. The organic extracts are combined and washed successively with four 250-ml. portions of saturated sodium chloride solution, four 250-ml. portions of saturated sodium bicarbonate solution, and again with four portions of the salt solution. The neutral ethyl acetate extract is dried over 20 g. of anhydrous magnesium sulfate and filtered. The filtrate is concentrated and the residue is distilled under reduced pressure. A fore-run of 1–3 g. of semisolid is obtained up to 120°/0.02 mm. The product is then collected as a light-yellow oil, b.p. 120–130°/0.01–0.02 mm., which solidifies in the chilled receiver. The distillate (97–101 g.) is melted on a steam bath and dissolved in a mixture of 50 ml. of benzene and 50 ml. of hexane. The solution is transferred quantitatively to a 500-ml. Erlenmeyer flask and the solvent evaporated on a steam bath. The oily residue is placed in a refrigerator for at least 1 day to ensure complete crystallization. The white solid is transferred to a Büchner funnel with the aid of a little ice-cold hexane. After drying in air, the yield is 95–100 g. (59–62%) of N-benzylacrylamide, m.p. 65–68° (Note 4).

2. Notes

1. Commercial acrylonitrile from American Cyanamid Company was redistilled, b.p. 77–78°.
2. The dropping funnel which has been wetted with sulfuric acid should be washed and thoroughly dried, or a fresh funnel employed.
3. Benzyl alcohol from Fisher Scientific Company was used without further purification.
4. The product is of suitable purity for further reactions. It may be obtained analytically pure, m.p. 70–72°, by recrystallization from benzene. The reported melting point is 69°.³

3. Discussion

N-Benzylacrylamide has been prepared by dehydrohalogenation of N-benzyl-β-chloropropionamide with aqueous potassium hydroxide,³ and by the reaction of acetylene with carbon monoxide and benzylamine.⁴ The procedure described is the method of Parris and Christenson.⁵

4. Merits of Preparation

The alternative methods of preparation of [N-benzylacrylamide](#) are reported in patents, and no yields are given. One of them requires two steps and costlier intermediates; the other appears to be more suitable for plant than for laboratory preparation. The procedure presented involves a simple, one-step reaction taking place under mild conditions, employing inexpensive reactants and affording satisfactory yields.

The N-alkylation of nitriles with aralkyl alcohols, a special case of the Ritter reaction,⁶ is a novel general reaction. The following compounds were prepared by this procedure in the corresponding yields: [N-benzylacetamide](#) (48%), [N-\(2,4-dimethylbenzyl\)-acetamide](#) (40%), [N-\(4-methoxybenzyl\)-acetamide](#) (60%), [N,N'-diacrylyl-*p*-xylene- \$\alpha\$ - \$\alpha'\$ -diamine](#) (64%), [N,N'-diacetyl-4,6-dimethyl-*m*-xylene- \$\alpha\$, \$\alpha'\$ -diamine](#) (62%). Another example of the Ritter reaction is given elsewhere in this volume.⁷

The title compound is of special interest and utility as a polymerizable monomer.

This preparation is referenced from:

- [Org. Syn. Coll. Vol. 5, 471](#)

References and Notes

1. Pittsburgh Plate Glass Company, Springdale, Pa.
2. E. B. Hershberg, *Ind. Eng. Chem. Anal. Ed.*, **8**, 313 (1936).
3. G. Kranzlein and M. Corell (to I. G. Farbenindustrie, Akt.-Ges.), Ger. pat. 752,481 (Nov. 10, 1952).
4. E. H. Specht, A. Neuman, H. T. Neher (to Rohm and Haas Co.), U. S. pat. 2,773,063 (Dec. 4, 1956).
5. C. L. Parris and R. M. Christenson, *J. Org. Chem.*, **25**, 331, 1888 (1960).
6. L. I. Krinsen and D. J. Cota, *Org. Reactions*, **17**, 213 (1969).
7. [J. J. Ritter and J. Kalish, this volume, p. 471.](#)

Appendix

Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

[sulfuric acid](#) (7664-93-9)

[acetylene](#) (74-86-2)

[Benzene](#) (71-43-2)

[ethyl acetate](#) (141-78-6)

[carbon monoxide](#) (630-08-0)

[sodium bicarbonate](#) (144-55-8)

[sodium chloride](#) (7647-14-5)

[potassium hydroxide](#) (1310-58-3)

[Benzyl alcohol](#) (100-51-6)

magnesium sulfate (7487-88-9)

benzylamine (100-46-9)

acrylonitrile (107-13-1)

hexane (110-54-3)

N-BENZYLACRYLAMIDE,
Acrylamide, N-benzyl- (13304-62-6)

N-benzyl- β -chloropropionamide (501-68-8)

N-benzylacetamide (588-46-5)

N-(2,4-dimethylbenzyl)-acetamide

N-(4-methoxybenzyl)-acetamide

N,N'-diacrylyl-p-xylene- α - α' -diamine

N,N'-diacetyl-4,6-dimethyl-m-xylene- α , α' -diamine