Organic Syntheses

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.

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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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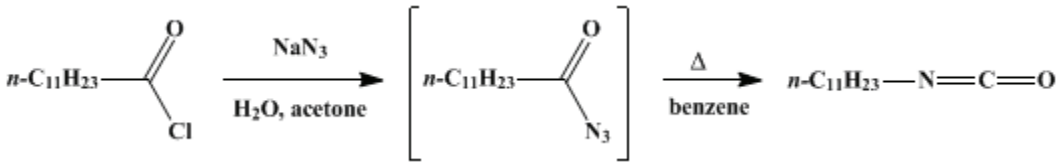
UNDECYL ISOCYANATE



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1. Procedure

In a 1-l. three-necked flask, equipped with a stirrer and a thermometer and immersed in an ice bath, is placed 46 g. (0.7 mole) of sodium azide (Note 1) in 150 ml. of water. A mixture of 109 g. (0.5 mole) of lauroyl chloride (b.p. 134–137°/11 mm.) and 150 ml. of acetone is then added from a separatory funnel to the well-stirred solution of the azide at such a rate that the temperature remains at 10–15°. After the mixture has been stirred at this temperature for an hour, the stirrer is stopped and, when the layers have separated, the lower water layer is removed carefully by suction through a glass capillary tube (Note 2). The upper layer is then added slowly to 500 ml. of benzene which has been warmed to 60° (Note 3). A rather rapid evolution of gas results, and the mixture is kept at 60–70° (Note 4) until no more nitrogen is evolved; the conversion of azide to isocyanate requires about an hour. The solution is filtered to remove any insoluble matter, and the benzene is removed by distillation from a modified Claisen flask. Distillation of the residue yields 80–85 g. of ester (81–86%) (Note 5) and (Note 6).

2. Notes

1. A practical grade of sodium azide such as that obtained from the Eastman Kodak Company is satisfactory.
2. It is important that the water be removed as completely as possible before the azide is added to the warm benzene. Failure to remove the water causes formation of the *sym*-disubstituted urea during decomposition of the azide. If the water is separated carefully, there will be no need to filter the benzene solution before the final distillation.
3. If the azide is added too rapidly, the solution may froth over; it is best to carry out this reaction in a 1-l. beaker.
4. The heat of reaction is usually sufficient to maintain the temperature at 60–70°.
5. On redistillation all the product boils at 103°/3 mm. A second distillation is unnecessary; the original ester is pure enough for all practical purposes.
6. This method is a general one for the preparation of isocyanates.[1]

3. Discussion

This procedure is one used by Schröter for preparing alkyl isocyanates.[1]

References and Notes

1. Schröter, *Ber.*, **42**, 3356 (1909).

Appendix
Chemical Abstracts Nomenclature (Collective Index Number);

(Registry Number)

sym-disubstituted urea

Benzene (71-43-2)

nitrogen (7727-37-9)

acetone (67-64-1)

sodium azide (26628-22-8)

UNDECYL ISOCYANATE (2411-58-7)

lauroyl chloride (112-16-3)