



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.

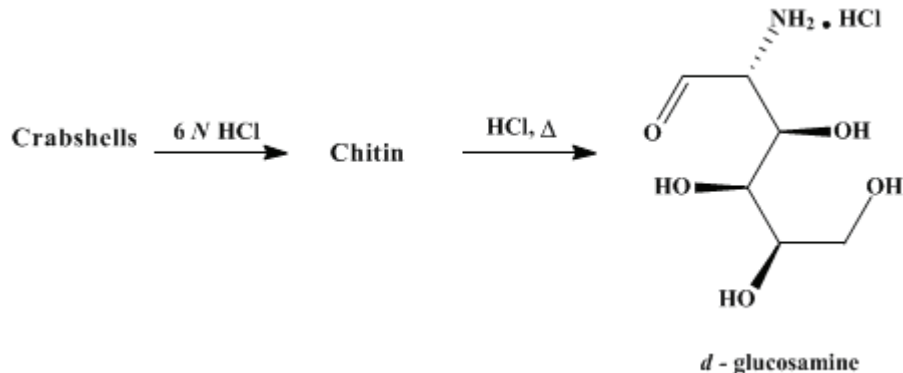
In some articles in *Organic Syntheses*, chemical-specific hazards are highlighted in red "Caution Notes" within a procedure. It is important to recognize that the absence of a caution note does not imply that no significant hazards are associated with the chemicals involved in that procedure. Prior 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.

The procedures described in *Organic Syntheses* are provided as published and are conducted at one's own risk. *Organic Syntheses, Inc.*, its Editors, and its Board of Directors do not warrant or guarantee the safety of individuals using these procedures and hereby disclaim any liability for any injuries or damages claimed to have resulted from or related in any way to the procedures herein.

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.

Organic Syntheses, Coll. Vol. 3, p.430 (1955); Vol. 26, p.36 (1946).

***d*-GLUCOSAMINE HYDROCHLORIDE**



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1. Procedure

Two hundred grams of cleaned and dried crab shells ([Note 1](#)) ground to a fine powder is placed in a 2-l. beaker, and an excess of dilute (approximately 6 *N*) commercial [hydrochloric acid](#) is added slowly to the powdered material until no further action is evident. Much frothing occurs during the addition of the acid, and care must be exercised to avoid loss of material due to foaming over the sides of the beaker. After the reaction has subsided, the reaction mixture is allowed to stand from 4 to 6 hours to ensure complete removal of [calcium carbonate](#). The residue is then filtered, washed with water until neutral to litmus, and dried in an oven at 50–60°. The weight of dried chitin is usually about 70 g., but with some lots of crab shells it may be as low as 40 g.

To 40 g. of dry chitin in a 500-ml. beaker is added 200 ml. of concentrated [hydrochloric acid](#) (C.P., sp. gr. 1.18), and the mixture is heated on a boiling water bath for 2.5 hours with continuous mechanical agitation. At the end of this time solution is complete, and 200 ml. of water and 4 g. of Norit are added. The beaker is transferred to a hot plate, and the solution is maintained at a temperature of about 60° and is stirred continuously during the process of decolorization. After an hour the solution is filtered through a layer of a filter aid such as Filter-Cel. The filtrate is usually a pale straw color; however, if an excessive color persists, the decolorization may be repeated until the solution becomes almost colorless. The filtrate is concentrated under diminished pressure at 50° until the volume of the solution is 10–15 ml. The white crystals of [glucosamine hydrochloride](#) are washed onto a sintered-glass filter with 95% [ethanol](#). The white crystalline product, after being washed with 95% [ethanol](#) and dried, weighs 24–28 g. (60–70% of the weight of the chitin used). The optical rotation of a stable solution of the product containing the α - and β -isomers at equilibrium, $[\alpha]_{D}^{25}$, varies from +68.8° to +70.1° ($c = 4.75$ in water).

The product is pure enough for most uses. If a purer product is desired, the crystals may be dissolved in the minimum amount of boiling water and treated with Norit. The resulting solution is filtered and added to a large excess of 95% [ethanol](#) and stirred vigorously for several hours. The product is collected after 4–6 hours. An appreciable amount of the β -form of the amino sugar remains dissolved in the alcohol and may be precipitated by adding [ether](#).

2. Notes

1. The cleaned and dried crab shells were obtained from Carter and Lanhardt Company, Eleventh and Maine Avenue, S.W., Washington, D. C.

3. Discussion

The preparation of [glucosamine hydrochloride](#) from lobster shells and crab shells by essentially this

method has been reported by Irvine, McNicoll, and Hynd¹ and Hudson and Dale.² Other methods involving the use of cicad larvae shells³ and shrimp shells^{4,5} also have been reported.

References and Notes

1. Irvine, McNicoll, and Hynd, *J. Chem. Soc.*, **99**, 256 (1911).
 2. Hudson and Dale, *J. Am. Chem. Soc.*, **38**, 1434 (1916).
 3. Komori, *J. Biochem. Japan*, **6**, 1–20 (1926) [*C. A.*, **21**, 372 (1927)].
 4. van Alphen, *Chem. Weekblad*, **26**, 602 (1929) [*C. A.*, **24**, 2113 (1930)].
 5. Rigby, U. S. pat. 2,040,879 [*C. A.*, **30**, 4598 (1936)].
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Appendix Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

ethanol (64-17-5)

hydrochloric acid (7647-01-0)

ether (60-29-7)

calcium carbonate (471-34-1)

glucosamine hydrochloride,
D-Glucosamine hydrochloride (66-84-2)