

stirrer. The temperatures and the durations of the reaction for three experiments will be found in Table III. The distillation process was the same as that described for Procedure 2. For the first two runs, in which chloronitrile was recovered, corrected yields have been given. In this procedure, however, more polymerization occurred than in Procedure 2, but less than in Procedure 1.

TABLE III
DATA FOR EXPERIMENTS

Run	Approx. temp., °C.	Reaction time, hrs.	Yield, %	Yield (corr.), %
1	30	7-8	41	64.7
2	Refluxing	16	69.4	80.2
3	Refluxing	25	74.7	..

Summary

1. Cyclopropyl cyanide may be obtained in 75-90% yields by the action of sodamide on γ -chlorobutyronitrile in a liquid ammonia-ether solution. The reaction in ether solution appears to be less efficient.

2. Trimethylene chlorobromide may be prepared in 90-94% yields by the action of phosphorus tribromide on trimethylene chlorohydrin.

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NOTES

The Cholesterol Content of Shrimp Waste.—The ethyl ether extraction of the dried shrimp and shrimp waste prepared by Vilbrandt and Abernethy¹ produced a viscous oil, dark red by transmitted light and soluble in the common organic solvents. According to elemental analysis the oil contains no sulfur, halogens, nitrogen or heavy metals.

Treatment of the oil with alcohol at 60° precipitated an insoluble brown resinous material and subsequent cooling of the alcoholic solution to 0° effected a fairly rapid separation of monoclinic crystals from the viscous, red oily residue.

Purification of Crystals.—Decolorization of the hot alcoholic solution of the crystals thus obtained with animal charcoal and subsequent crystallizations from hot alcohol produced a material which melted at 146.5°. Measurements under the microscope showed monoclinic plates with angles of 79.5 and 100.5°. The average yield of these crystals from three separate crystallizations and purifications is 19.08% on the basis of the original oil. The total quantity of sterols in the shrimp waste was not determined.

Identification.—The purified crystals were then treated with digitonin by the method of Windaus² and further identified by the Liebermann³

¹ F. C. Vilbrandt and R. F. Abernethy, "Utilization of Shrimp Waste," United States Fisheries Document No. 1079 (1930).

² A. Windaus, *Chem.-Ztg.*, **37**, 1001 (1913).

³ Oliver Kamm, "Qualitative Organic Analysis," John Wiley and Sons, Inc., New York, 1924, p. 123.

and the Hagersalkowski⁴ tests for cholesterol, obtaining positive tests. Further identification of the crystals as cholesterol was accomplished by the preparation of the benzoate⁵ and acetate⁶ derivatives.

The high cholesterol content of the shrimp oil led to feeding tests for antirachitic potency of the oil by the authors and F. P. Brooks,⁷ giving encouraging results. Since the extract of shrimp waste amounts to 2.25% of the waste, a production of 80,000 pounds of cholesterol could have been accomplished by the extraction of the waste of the 1927 crop of shrimp.

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RECEIVED AUGUST 4, 1930
PUBLISHED JULY 8, 1931

A Simple Method for the Preparation of Glycine.—The preparation of glycine by the classical method,¹ employing the reaction between monochloroacetic acid and ammonia, has proved generally unsatisfactory because of the lengthy procedures involved in the isolation of a product free from ammonia and chloride and because of the low yield of pure glycine resulting. Robertson² found after a careful study of the course of the reaction between monochloroacetic acid and ammonia, that the yield of glycine could be greatly increased by the use of a large excess of ammonia and that there occurred a corresponding decrease in the amount of imino-diacetic acid and trimethylene-amine-tricarboxylic acid, the principal impurities resulting from side reactions. Based upon this observation, he proposed a method in which a 60 to 1 molecular ratio of ammonia to monochloroacetic acid was employed. This increased the actual concentration of glycine formed in solution to 86% of the theoretical. While a 50% final yield of pure glycine was obtained, the isolation of the product was accomplished after the laborious and expensive procedure of removing the ammonium chloride by the use of silver oxide.

Since, when a large excess of ammonia was used, the concentration of glycine formed was large in comparison with the amount of side reaction products, the possibility of *direct* precipitation of glycine seemed worthy of investigation. As was suggested by Robertson, 60 moles of

⁴ D. Holde and E. Mueller, "Saponifiable Hydrocarbons, Fats and Waxes," 1922, p. 519.

⁵ S. P. Mulliken, "Identification of Pure Organic Compounds," John Wiley and Sons, Inc., New York, 1904, Vol. I, p. 172.

⁶ R. J. Anderson, *J. Biol. Chem.*, 71, 707 (1926).

⁷ F. C. Vilbrandt, R. F. Abernethy and F. P. Brooks, "Preliminary Report on the Antirachitic Properties of Shrimp Oil," *THIS JOURNAL*, 52, 4940 (1930).

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¹ Kraut, *Ann.*, 266, 295 (1891).

² G. R. Robertson, *THIS JOURNAL*, 49, 2889 (1927).