tone. The purified product weighed 59 g. (84% yield) and melted at 180–182°. (The spectrum showed absorption peaks at 5.79 $\mu$ (carbonyl group), and 11.62 $\mu$ associated with the *trans* configuration.)

*Anal.* Calcd. for $C_4H_2Cl_2O_4$: C, 25.97; H, 1.09; Cl, 38.33; neut. equiv., 92.5. Found: C, 26.06; H, 1.16; Cl, 38.79; neut. equiv., 91.6.

**Dimethyl Dichlorofumarate.**—The dimethyl ester of dichlorofumaric acid was prepared by treating the acid with methanol and sulfuric acid. It boiled at 125° at 20 mm., $n^{20}D$ 1.4820. (The spectrum had absorption peaks at 6.13 $\mu$ (conjugated double bond), 7.9 $\mu$ (C–O–C), 5.75 $\mu$ (carbonyl group), and 11.1 and 11.75 $\mu$ (*trans* configuration).)

*Anal.* Calcd. for $C_6H_6O_4Cl_2$: C, 33.83; H, 2.84; Cl, 33.29. Found: C, 34.28; H, 2.93; Cl, 33.31.

**Dimethyl Dichloromaleate.**—Dichloromaleic anhydride was prepared in 7.5% yield by chlorinating maleic anhydride at 180° using $FeCl_3$–$CaCl_2$ catalyst.[4] The yield can be increased by further chlorination of the recovered intermediate chloromaleic anhydride. The dichloromaleic anhydride, after recrystallization from isopropyl ether and from carbon tetrachloride, was treated with methanolic hydrogen chloride. The dimethyl dichloromaleate obtained boiled at 121° at 20 mm., $n^{20}D$ 1.4830, $d^{20}_4$ 1.3993, $d^{25}_4$ 1.3943. (The spectrum of this ester, like that of the dimethyl dichlorofumarate, showed absorption peaks at 6.13, 7.9 and 5.75 $\mu$. The bands at 11.1 and 11.75 $\mu$ were replaced, however, by a strong band at 14.6 $\mu$, associated with the *cis* configuration. Both esters, dichlorofumaronitrile, and dichloromaleic anhydride (but not dichlorofumaric acid) had an unassigned absorption peak varying between 9.8–9.9 $\mu$.)

*Anal.* Calcd. for $C_6H_6O_4Cl_2$: C, 33.83; H, 2.84; Cl, 33.29. Found: C, 33.94; H, 2.88; Cl, 33.42.

**Acknowledgments.**—The authors wish to thank Mr. Harry Vineyard for technical assistance, Mr. C. M. Lovell for determination and interpretation of the infrared spectra, and Mr. Quentin Quick and his staff for the microanalyses.

[4] C. R. Milone, U. S. Patent 2,391,261; F. Johnston and L. W. Newton, private communication.

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## Long-Chain Alkylurea N-Glycosides[1]

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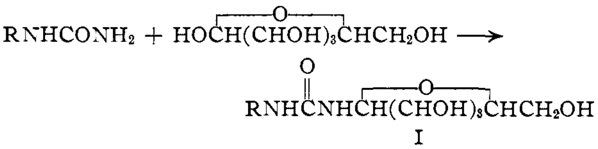
Schoorl[2] found that aldose sugars react with urea, methylurea, dimethylurea, phenylurea, benzylurea, thiourea, biuret and urethan. Dilute aqueous sulfuric acid was used to catalyze the reaction. Helferich and Kosche[3] did closely similar work. No one seems to have used any alkylureas higher than methylurea.

We have found that the long-chain alkylureas react with aldohexoses in warm dilute alcoholic hydrochloric acid. The products presumably have structure I. The reactions fail in the absence of acid.



$$RNHCONH_2 + HO\overset{\text{—O—}}{CH(CHOH)_3CH}CH_2OH \longrightarrow RNH\overset{O}{\overset{\|}{C}}NH\overset{\text{—O—}}{CH(CHOH)_3CH}CH_2OH$$

I

Ketohexoses do not react, even in the presence of acid. Neither the alkylureas nor the sugars used are very soluble in alcohol and rather long reaction times are therefore necessary.

### Experimental

**Dodecylurea N-Galactoside.**—A mixture of dodecylurea (22.8 g., 0.10 mole), galactose (19.8 g., 0.11 mole), concd. hydrochloric acid (2.5 g.), and 95% ethanol (200 ml.) was stirred and heated at 50° for 100 hours, then cooled and filtered. Washing with water and hot benzene gave 15.6 g. (40%) of white solid, m.p. 165–188° (dec.).

*Anal.* Calcd. for $C_{19}H_{38}N_2O_6$: C, 58.44; H, 9.81; N, 7.18. Found: C, 58.31; H, 9.64; N, 7.05.

**Octadecylurea N-Glucoside.**—A mixture of octadecylurea (6.2 g., 0.02 mole), glucose (4.0 g., 0.022 mole), concd. hydrochloric acid (0.5 g.) and 95% ethanol (100 ml.) was stirred and heated at 50° for 147.5 hours, cooled and filtered. Washing with hot benzene left 1.5 g. (27%), m.p. 175–188° (dec.); m.p., after further washing with water and hot benzene, 181–189° (dec.).

*Anal.* Calcd. for $C_{25}H_{50}N_2O_6$: C, 63.26; H, 10.62; N, 5.90. Found: C, 63.20; H, 10.42; N, 6.16.

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[1] Paper No. 147, Journal Series, General Mills, Inc., Research Dept.

[2] M. N. Schoorl, *Rec. trav. chim.*, **22**, 31 (1903).

[3] S. B. Helferich and W. Kosche, *Ber.*, **59**, 69 (1926).

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## A Note on the Heat of Vaporization of Chlorotrifluoroethylene ($C_2F_3Cl$)[1]

By George T. Furukawa, Robert E. McCoskey and Martin L. Reilly

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Oliver, *et al.*,[2] recently reported the entropy of chlorotrifluoroethylene in the ideal gaseous state at 244.80°K. (normal boiling point) and 1 atm. as 73.18 cal. deg.$^{-1}$ mole$^{-1}$ from the results of their low temperature calorimetric measurements. These workers computed the entropy of vaporization from their vapor pressure data and the Clapeyron equation. In many calculations of this sort large uncertainties arise on account of the inaccuracies in the density data and the derivative of the vapor pressure. In connection with the program at the National Bureau of Standards involving the spectral assignments for a series of compounds related to chlorotrifluoroethylene, it was felt desirable to determine the heat of vaporization of chlorotrifluoroethylene directly to eliminate any uncertainty that may exist in this quantity. As this molecule is considered to be a "key" member of the series, an accurate value for the entropy was especially desirable to verify the spectral assignment.[3] This note deals with the measurement of the heat of vaporization of chlorotrifluoroethylene.

### Experimental

**Apparatus.**—The vaporization experiments were made in an adiabatic calorimeter similar in design to that described by Osborne and Ginnings[4a] and by Aston, *et al.*,[4b] in which a throttle valve is contained within the space enclosed by the adiabatic shield. Briefly, as electrical energy was sup-

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[1] This investigation was conducted under research sponsored by the Ordnance Corps, Department of the Army.

[2] G. D. Oliver, J. W. Grisard and C. W. Cunningham, This Journal, **73**, 5719 (1951).

[3] D. E. Mann, N. Acquista and E. K. Plyler, *J. Chem. Phys.*, submitted for publication.

[4] (a) N. S. Osborne and D. C. Ginnings, *J. Research Natl. Bur. Standards*, **39**, 453 (1947); (b) J. G. Aston, H. L. Fink, G. J. Janz and K. E. Russell, This Journal, **73**, 1939 (1951).