

Stability of Purified Hydrazine. Borosilicate glass ampules were attached to the vacuum line and outgassed at 300° C. and a pressure of 10^{-5} mm. of mercury. Freshly distilled hydrazine in the storage bulb was transferred under its vapor pressure at room temperature to the ampules, which were cooled to liquid-nitrogen temperature. The ullages were approximately 50% (approx. 2 ml. of sample). The ampules were isolated from the hydrazine vapors, sealed, and removed from the vacuum line. The hydrazine stored in this manner deteriorated to what appeared to be an equilibrium composition. Similar samples of hydrazine from the ammonolysis of hydrazine sulfate were used for comparison. Table I shows the change in composition of these samples with time at room temperature.

Refractionation of samples 3 or more days old yielded 99.9% hydrazine and traces of ammonia. The ammonia was identified on the basis of vapor pressure (5) and molecular weight. The values in the literature for the vapor pressure of ammonia are 10 and 1 mm. of mercury at -91.9° and -109.1° C., respectively; the calculated molecular weight is 17.03. The values obtained in this experiment are 11 and 1.1 mm. of mercury at -91° and -110° C. and molecular weights of 17.01 and 17.04.

No attempts was made to search for or identify products which could not be trapped with liquid nitrogen.

Experience in this laboratory has shown that decomposition rates for hydrazine are meaningless unless the history of the material decomposed is defined, and the material is decomposed in equipment where hydrazine is exposed to a unique surface. For example, for a given sample of hydrazine, the rate of decomposition in a mixed glass-metal system will differ from that of a system of one particular metal.

Table I. The Effect of Two Procedures on the Purity of Hydrazine

Sample	Time After Fractionation, Days	N ₂ H ₄ , %	
		Calcium hydride	Ammonolysis
1	0	99.99 ± 0.03	99.99 ± 0.03
2	1	99.95	99.93
3	2	99.92	99.91
4	3	99.89	99.90
5	10	99.90	99.90
6	30	...	99.90
	184	99.90	...
7	300	99.90	99.90

LITERATURE CITED

- (1) Audrieth, L.F., Ogg, B.A., "The Chemistry of Hydrazine," Chap. 3, Wiley, New York, 1951.
- (2) Browne, A.N., Houlehan, A.E., *J. Am. Chem. Soc.* **33**, 1734 (1911).
- (3) Browne, A.W., Welsh, T.W.B., *Ibid.*, 1728 (1911).
- (4) Class, J.B., Aston, J.G., Oakwood, T.S., *J. Am. Chem. Soc.* **75**, 2937 (1953).
- (5) Overstreet, Roy, Giauque, W.F., *J. Am. Chem. Soc.* **59**, 254 (1937).
- (6) Penneman, R.A., Audrieth, L.F., *Anal. Chem.* **20**, 1058 (1948).
- (7) Rice, F.O., Sherber, F., *J. Am. Chem. Soc.* **77**, 291 (1955).
- (8) Scott, A., *J. Chem. Soc.* **85**, 913 (1904).
- (9) Stolle, P., *J. Prakt. Chem.* **83**, 200 (1911); *Ber.* **37**, 454 (1904).

RECEIVED for review October 3, 1961. Accepted March 23, 1962.

1-Benzyl-4,5-bis(chloromethyl)-1H-1,2,3-triazole

JAMES MOFFAT and JERRY L. RUTTER

Department of Chemistry, University of Kansas City, Kansas City, Mo.

WE HAVE prepared the title compound by the reaction of benzyl azide and 1,4-dichloro-2-butyne.

EXPERIMENTAL

To 100 ml. of toluene was added 5.6 grams (0.045 mole) of 1,4-dichloro-2-butyne (commercial sample) and 6.0 grams of benzylazide (1). The solution was refluxed 8 hours and the product was precipitated by addition of petroleum ether. After two recrystallizations from a mixture of petroleum ether and ethyl acetate, 5.7 grams (49%) of product was obtained, m.p. 54.5°-55°.

Anal. Calcd. for C₁₁H₁₁Cl₂N₃: C, 51.58; H, 4.33; Cl, 27.68; N, 16.40. Found: C, 51.69; H, 4.20; Cl, 27.96; N, 16.57.

ACKNOWLEDGMENTS

The authors are grateful to the Spencer Chemical Co. for the analyses and for laboratory privileges.

LITERATURE CITED

- (1) Curtius, T., Erhart, G., *Ber.* **55**, 1565 (1922).

RECEIVED for review July 5, 1962. Accepted July 12, 1962.