TABLE I

PHYSICAL AND ANALYTICAL DATA FOR THE N-SUBSTITUTED 2,4:3,5-DIMETHYLENE-D-GLUCONAMIDES

Amide	Yield, %	M. p., °C.	$[\alpha]_D$	Temp., °C.	c	Formula	Nitrogen, % Calcd.	Nitrogen, % Found
N-Methyl	80	195.6–196.8	+92.7	29.6	1.48	$C_9H_{15}O_6N$	6.0	6.0
N-Ethyl	40	169.0–170.0	+77.5	29.0	1.64	$C_{10}H_{17}O_6N$	5.7	5.6
N-Allyl	69	191.2–192.8	+61.2	32.4	1.43	$C_{11}H_{17}O_6N$	5.4	5.4
N-*n*-Butyl	20	141.6–142.2	+64.2	30.8	1.04	$C_{12}H_{21}O_6N$	5.1	5.0
N-Isobutyl	15	158.6–159.0	+62.2	30.8	0.96	$C_{12}H_{21}O_6N$	5.1	5.0
N-β-Hydroxyethyl	40	176.8–178.0	+75.0	29.6	1.51	$C_{10}H_{17}O_7N$	5.3	5.5
Cyclohexyl	50	217.0–217.6	+34.9	29.6	4.0	$C_{14}H_{23}O_6N$	4.65	4.4

crude amides were recrystallized from methanol or methanol–benzene. All the N-substituted dimethylene gluconamides prepared were found to be soluble in water or methanol and insoluble in ether or benzene. The specific rotations were determined in water as solvent at the concentration and temperature indicated in Table I.

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The System Ammonium Sulfate–Ammonia

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Solvation of ammonium sulfate in liquid ammonia was first observed by Browne and Welsh.[1] Brief mention of this behavior also was made by Friedrichs[2] who stated that $(NH_4)_2SO_4 \cdot 3NH_3$, insoluble in liquid ammonia, was formed, and claimed that in a sealed tube this compound was stable up to 145°. The solubility was found to increase slightly with temperature.

Investigations in this Laboratory have verified the existence of ammonium sulfate 3-ammoniate. A pressure–concentration isotherm at −32.5° and vapor pressure data for this compound were determined. No other ammoniates were formed over the temperature range −33 to +8°.

Experimental

The apparatus employed in this investigation was similar to the equipment described by Howard, Friedrichs and Browne[3] for the study of binary systems having one volatile component. Commercial anhydrous ammonia was dried by passing the gas through a tube containing potassium hydroxide and then through a tube containing fine sodium wire. Ammonium sulfate (J. T. Baker C.P.) was purified further by recrystallization.

The pressure–concentration isotherm shown in Fig. 1 was obtained by adding a measured excess of ammonia to a weighed sample of ammonium sulfate and then withdrawing small increments of the gas. Pressure readings were taken when the system reached equilibrium, usually after twenty-four hours. Vapor pressure data for ammonium sulfate 3-ammoniate which are shown graphically in Fig. 2, were determined in the same apparatus. These data may be summarized by the equation log p (mm. Hg) = 10.2737 − 1934.4/T°K.

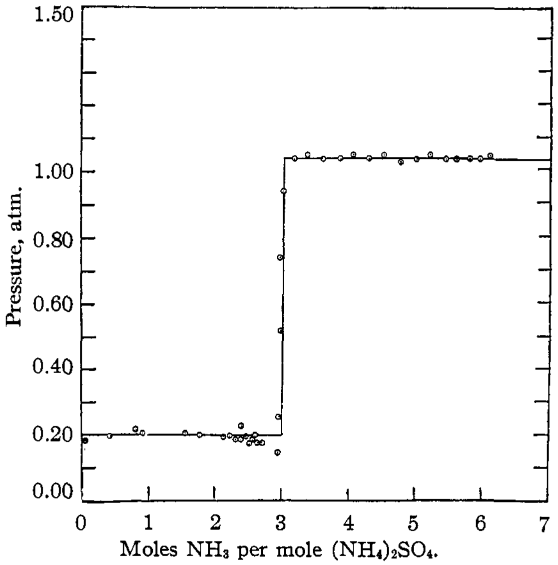

Fig. 1.—Pressure–concentration isotherm (−32.5°) of the system ammonium sulfate–ammonia.

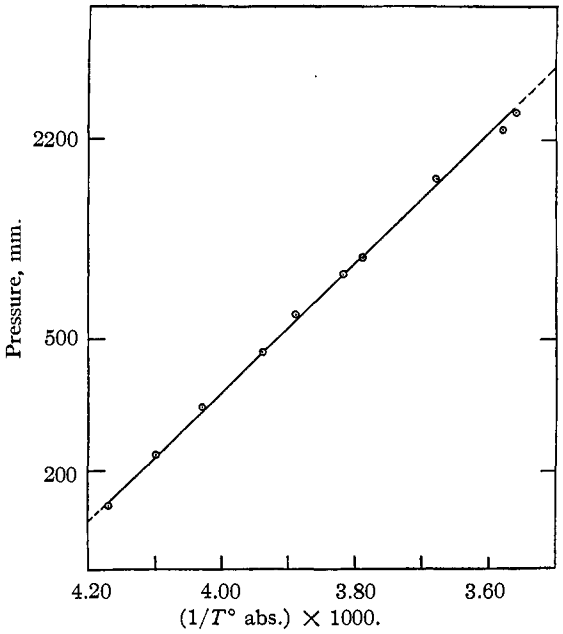

Fig. 2.—Vapor pressure of ammonium sulfate 3-ammoniate.

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[1] Browne and Welsh, THIS JOURNAL, **33**, 1728 (1911).

[2] Friedrichs, *ibid.*, **35**, 1866 (1913); *Z. anorg. allgem. Chem.*, **84**, 373 (1914).

[3] Howard, Friedrichs, and Browne, *ibid.* **56**, 2332 (1934).