water giving $[\alpha]^{28}D$ +95.4°. The melting point of the initial sample was 157–158° (dec.) and of the sample obtained from the 95.5-hour solution was 160–161° (dec.).

By the isolated rabbit uterus method,[1] the physiologic activity of the base obtained from the 95.5-hour sample was about 90% of that of the initial sample.

Initial rotations of the maleate salt of the new base were as follows.

TABLE III

Solvent	Wt. and vol. of solution	$\alpha^{28}D$	$[\alpha]^{28}D$
Distilled water	0.1042 g. in 10 cc.	+0.48°	+46.2°
Abs. methyl alcohol	.1017 g. in 25 cc.	+ .308°	+37.9°

Rotations were again made on these same solutions after standing at room temperature for forty-eight hours, and were found to be as follows: on the water solution $\alpha^{28}D$ +0.56°, $[\alpha]^{28}D$ +53.7°; on the methyl alcohol $\alpha^{28}D$ +0.20°, $[\alpha]^{28}D$ 24.6°. The forty-eight-hour methyl alcohol solution was evaporated *in vacuo* to dryness at room temperature, water was added to bring the solution up to the original methyl alcohol volume. The rotation was $\alpha^{28}D$ 0.214°, $[\alpha]^{28}D$ 52.9°. The physiologic activity of these forty-eight-hour samples as determined by the isolated rabbit uterus method was approximately the same as that of an initial sample.

The explanation of these results is not clear at present; evidently some change is occurring in the molecule which affects the optical rotation, but which does not greatly affect the physiologic activity. Changes of rotation have been noted in the cases of ergotinine and ergotamine which have been ascribed to a change into ergotoxine in the former case and into ergotaminine in the latter case. This explanation does not appear to be a logical one for the changes occurring here, since the physiologic activity seems to be practically unchanged and since the product obtained from a solution of the salt of the new base in methyl alcohol and the product obtained from a solution in water appear to be the same.

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An Improved Method of Extraction

BY CHARLES A. MARLIES AND VICTOR K. LA MER

In an investigation on the acid and salt catalysis of nitramide,[1] NH_2NO_2, a novel method of extraction was employed in the final stage of the preparation of this interesting compound. In the customary method[2] the compound is extracted from its aqueous solution, some forty extractions with ether being necessary on account of the exceedingly unfavorable distribution ratio. The improvement consists of immersing the flask containing the nitramide solution and supernatant ether layer into a "dry ice" freezing mixture and swirling until the water layer solidifies completely. The nitramide passes into the ether layer which is decanted through a filter. Complete extraction was achieved by repeating the process three times. The yield obtained on evaporation of the four combined ether extracts was 80%, whereas the maximum yield by the previous method was but 25%, in agreement with the experience of Brönsted's laboratory.[3]

The low yields by the previous[2] method are probably due to decomposition during the prolonged evaporation of the large volume of ether. Nitramide is an extremely unstable substance and the catalytic action resulting from the concentration of the ever-present impurities (including water) during the evaporation probably causes considerable loss by decomposition.

This method of freezing the solvent during extraction should prove generally useful not only in cases where the distribution ratio is unfavorable but also to remove small amounts of material from large volumes of solution, provided, of course, that solid solution is not an important complication.

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