Preparation of *m*-Aminophenyl β-Piperidinoethyl Ketone Mono-hydrochloride

In connection with a problem in this Laboratory, we had occasion to prepare *m*-aminophenyl β-piperidinoethyl ketone mono-hydrochloride. Mannich[1] was not able to obtain this compound by chemical reduction of the nitro base because of the unstable nature of the amine in acid solution and the nitro base was not reduced by catalytic hydrogenation. However, we have obtained the *m*-aminophenyl β-piperidinoethyl ketone mono-hydrochloride by catalytic reduction of *m*-nitrophenyl β-piperidinoethyl ketone mono-hydrochloride in aqueous solution with palladium on charcoal.

Following a procedure of Mannich,[2] the *m*-nitrophenyl β-piperidinoethyl ketone hydrochloride was prepared. The melting point of the crude material was 178–179°, softens at 175°. When recrystallized from alcohol–acetone and from 12A absolute alcohol the m. p. was 180–181°[3] (literature[1] m. p. 171–172°). *Anal.* Calcd. for $C_{14}H_{19}ClN_2O_3$: N, 9.35. Found: N, 9.43.

***m*-Aminophenyl β-Piperidinoethyl Ketone Mono-hydrochloride.**—Twelve grams (0.04 mole) of *m*-nitrophenyl β-piperidinoethyl ketone hydrochloride was dissolved in 100 ml. of water and hydrogenated in a Parr shaker in the presence of 1.2 g. of 5% palladium on activated charcoal (American Platinum Works, Newark, New Jersey) under 20 lb. pressure of hydrogen. The reduction was complete in thirty minutes. The solution was filtered from the catalyst and concentrated under reduced pressure to a sirup. About 50 ml. of absolute alcohol was added and the mass triturated. After a short period the soft mass crystallized. Eight grams of material, m. p. 175–178° (dec.), was obtained on filtering. (A mixed m. p. with starting material showed a depression of 18°.) An additional two grams of lower m. p. was obtained on addition of several volumes of dry ether. The total yield of crude material was 10 g. (92.5%). After treating with Darco and crystallizing three times from absolute alcohol, an analytical sample had m. p. 176–177° (dec.). *Anal.* Calcd. $C_{14}H_{21}ClN_2O$: C, 62.55; H, 7.50; N, 10.42. Found: C, 62.34; H, 7.74; N, 10.42.

We wish to thank E. F. Shelberg, Chief Microanalyst, for the microanalyses here reported.

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(1) Mannich and Dannehl, *Arch. Pharm.*, **276**, 206 (1938).

(2) "Organic Reactions," John Wiley and Sons, Inc., New York, N. Y., 1942, Vol. I, p. 329.

(3) Melting points are uncorrected.

COMMUNICATIONS TO THE EDITOR

THE NUCLEOTIDE COMPOSITION OF RIBONUCLEIC ACIDS[1]

Sir:

In continuation of our nucleic acid studies, procedures have been developed[2] that permit the separation on filter paper and estimation by spectrophotometry of all ribonucleotides encountered in ribonucleic acids. The separations were performed in an ammonia atmosphere with isobutyric acid as solvent. The components were demonstrated on the chromatograms by the previously described indirect method[2] or directly by inspection under the "Mineralight."[3] Although uridylic acid shared its position on the chromatogram with guanylic acid, the complete nucleotide analysis of ribonucleic acids could be effected by either of the following procedures. *Procedure 1:* 6–20 mg. of ribonucleic acid was kept at *p*H 13.5 for twelve to fifteen hours at 30°. The solution was brought to *p*H 3 to 4 (final volume 1 cc.) and submitted to separation in 0.01-cc. portions. Guanylic acid was eluted together with uridylic acid and the extinction at 265 and 245 mμ determined. The concentrations of the two components were calculated by simultaneous equations based on the absorption of the pure nucleotides. *Procedure 2:* 6–14 mg. of ribonucleic acid was suspended in absolute methanol and the purines liberated by gaseous hydrogen chloride.[4] The evaporation residue of the total hydrolysate was brought to *p*H 13.5. The rest of the operations followed Procedure 1. In this manner uridylic and cytidylic acids, guanine, and adenine could be separated and quantitatively determined on one chromatogram without supplementary calculations.

TABLE I

NUCLEOTIDE COMPOSITION OF RIBONUCLEIC ACIDS (EXPRESSED AS PER CENT. OF NUCLEIC ACID PHOSPHORUS)

Source	Procedure (see text)	Guanylic acid	Adenylic acid	Cytidylic acid	Uridylic acid	Total
Yeast Prepn. 1	1	28.0	29.0	17.8	20.3	95.1
	2	28.8	25.8	16.5	19.5	90.6
	3	25.6	26.1	24.4	8.3	84.4
Yeast Prepn. 2	1	27.3	26.2	19.6	15.3	88.4
	2				18.8	
	3	30.6	24.6			
Yeast Prepn. 3	1	26.2	24.8	21.4	20.3	92.7
	3	25.9	24.2			
Pig pancreas	2	40.7	18.9	17.8	8.4	85.8
	3	40.2	16.6	20.5	4.6	81.9
Pig liver	1	31.1	18.5	26.5	12.9	89.0
	3	31	17			

(1) This work was supported by a research grant from the U. S. Public Health Service.

(2) Vischer, Magasanik and Chargaff, *Federation Proc.*, **8**, in press (1949).

(3) We are very grateful to Dr. C. E. Carter, Oak Ridge National Laboratory, for suggesting this instrument.

(4) Vischer and Chargaff, *J. Biol. Chem.*, **176**, 715 (1948).