Lord, Jr., and D. H. R. Barton gave valuable advice for the theoretical part.

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Organic Salts of Benzylpenicillin. I. Aliphatic Amine Vasoconstrictors

BY H. W. RHODEHAMEL, JR.

During the preparation of a series of organic amine salts of benzylpenicillin, a number of crystalline combinations have been found. This note deals with the relatively water soluble crystalline benzylpenicillin salts of three useful aliphatic amine vasoconstrictors which have been of sufficient interest to suggest possible clinical application. Each is prepared in the same manner as follows: The free amine base in a convenient organic solvent, generally amyl acetate or diethyl ether, is added to an organic solution of free benzylpenicillin acid. The salt combination precipitates out as crystals immediately or after short standing with scratching or seeding.

The 2-Aminoheptane Salt of Benzylpenicillin.—White, needle-like crystals with a theoretical penicillin potency of 1270 Oxford units per mg.; m.p. (hot stage) 102–105° dec.; $[\alpha]^{25}\text{D} + 225 \pm 3°$ (c, 1 in H_2O). *Anal.* Calcd. for $C_{23}H_{35}O_4N_3S \cdot H_2O$: C, 59.09; H, 7.98; N, 8.99. Found: C, 59.99; H, 8.56; N, 8.89.

The 2-Amino-4-methylhexane Salt of Benzylpenicillin.—White, small, needle-like crystals with a theoretical penicillin potency of 1270 Oxford units per mg., m.p. (hot stage) 103–106° dec.; $[\alpha]^{25}\text{D} + 225 \pm 3°$ (c, 1 in H_2O). *Anal.* Calcd. for $C_{23}H_{35}O_4N_3S \cdot H_2O$: C, 59.09; H, 7.98; N, 8.99. Found C, 59.00; H, 7.87; N, 8.94.

The 1-Cyclopentyl-2-methylaminopropane Salt of Benzylpenicillin.—White, needle-like crystals with a theoretical penicillin potency of 1200 Oxford units per mg., m.p. (hot stage) 202–204° dec. *Anal.* Calcd. for $C_{25}H_{37}N_3O_4S \cdot H_2O$: C, 60.70; H, 8.15; N, 8.50. Found: (ash free) C, 61.21; H, 6.36; N, 8.86.

Another commerically available aliphatic amine vasoconstrictor, 1-cyclohexyl-2-methylaminopropane, has given a water soluble, amorphous salt with benzylpenicillin, but efforts to induce its crystallization have to date been unsuccessful.

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The Trimer of *o*-Phthalonitrile[1]

BY SIDNEY D. ROSS AND MORTON FINEMAN

In the course of their work on magnesium phthalocyanine, Linstead and Lowe[2] isolated an acetic acid soluble by-product melting at 296° and analyzing correctly for a polymer of phthalonitrile, $(C_8H_4N_2)_x$. In a later publication Dent and Linstead[3] showed by a molecular weight determination that this substance was a trimer, and that on hydrolysis with nitric acid it yielded phthalimide. On the basis of this evidence they assigned to the trimer a 2,4,6-tri-(*o*-cyanophenyl)-1,3,5-triazene structure. It is the purpose of the present communication to report a preparation of this trimer, some of its properties, and evidence confirming Linstead's structure assignment.

In connection with other work in these laboratories, we have observed that when *o*-phthalonitrile is heated at high temperature in the presence of a trace of acid or water, the trimer, reported by Linstead and Lowe, is formed. The trimer gives a yellow solution in sulfuric acid, from which phthalimide can be isolated after dilution with water.

The Linstead structure was confirmed by comparing the infrared spectrogram[4] of the trimer, mulled in nujol, with that of 2,4,6-triphenyl-1,3,5-triazene prepared by the method of Cook and Jones.[5] The *o*-phthalonitrile trimer (Figs. 1 and 2) shows the expected nitrile absorption at 4.53 microns, and the other absorptions can be attributed to the benzene ring, the triazene ring or the nujol vehicle. The spectrum of 2,4,6-triphenyl-1,3,5-triazene (Fig. 3) is shown for comparison.

All of the *o*-phthalonitrile trimer samples which we prepared were crystallized from acetic acid. Nevertheless, we observed that we obtained the product in two crystalline modifications, fine needles (Form A) and poorly defined clusters (Form B). Both crystalline forms melted at the same point and gave similar infrared spectrograms (Figs. 1 and 2). However, the two forms

TABLE I

X-RAY DIFFRACTION LINES FOR 2,4,6-TRI-(*o*-CYANOPHENYL)-1,3,5-TRIAZENE (FORM A)

#[a]	θ[b]	d[c]	I[d]
1	4.35	10.1	V. S.
2	5.90	7.49	M
3	7.10	6.25	S
4	8.96	4.94	M
5	11.30	3.92	S
6	12.7	3.50	S
7	13.5	3.30	V. S.
8	15.0	2.96	W
9	16.0	2.79	W
10	17.5	2.56	W
11	18.9	2.38	W
12	20.9	2.16	W
13	22.0	2.05	W
14	22.8	1.98	W
15	23.5	1.93	W
16	26.6	1.72	W
17	27.8	1.65	W

TABLE II

X-RAY DIFFRACTION LINES FOR 2,4,6-TRI-(*o*-CYANOPHENYL)-1,3,5-TRIAZENE (FORM B)

#	θ	d	I
1	4.14	10.7	V. S.
2	6.38	6.92	W
3	7.68	5.75	M
4	9.12	4.85	W
5	9.87	4.49	W
6	10.6	4.19	W
7	11.4	3.89	V. W.
8	12.0	3.70	W
9	13.6	3.27	V. S.
10	14.6	3.05	W
11	21.6	2.09	W
12	27.7	1.65	W

[a] #, order in which lines found. [b] θ, Bragg angl. [c] d, interplanar spacing. [d] I, qualitative intensity.

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[1] This work was performed under contract no. W36-039-sc-38142 with the Signal Corps of the United States Army.

[2] R. P. Linstead and A. R. Lowe, *J. Chem. Soc.*, 1022 (1934).

[3] C. E. Dent and R. P. Linstead, *ibid.*, 715 (1938).

[4] The spectra were determined by Philip Sadtler of Samuel P. Sadtler and Son, Inc.

[5] A. H. Cook and D. G. Jones, *J. Chem. Soc.*, 278 (1941).