

7-[α -(4-Pyridylamino)acetamido]cephalosporanic Acid (9). (a) *N*-(4-Pyridyl)glycyl Chloride Dihydrochloride. A stirred suspension of 10 g (0.065 mol) of *N*-(4-pyridyl)glycine¹⁵ in 200 ml of CH₂Cl₂ was cooled to -5° while being saturated with dry HCl gas. To this suspension was added 17.85 g (0.086 mol) of PCl₅ and the mixture stirred 1 hr at -5° and 2 hr at 0°. The solids were collected by filtration, washed well with dry CH₂Cl₂, and dried under vacuum over P₂O₅. The yield was 9.3 g whose ir spectra had a carbonyl (acid chloride) at 1785 cm⁻¹ as opposed to the carbonyl on the starting acid hydrochloride of 1710 cm⁻¹. The crude acid chloride was used for the acylation.

(b) **Coupling.** To a suspension of 8.16 g (0.03 mol) of 7-ACA (2) in 150 ml of dry CH₂Cl₂ was added 8.1 ml (0.058 mol) of TEA and 5.3 ml of *N,N*-dimethylaniline. The resulting solution was cooled to 0° and 7.6 ml (0.06 mol) of trimethylchlorosilane in 30 ml of CH₂Cl₂ was added dropwise. After 5 min at 0° the solution was refluxed for 30 min and cooled to -5°, and the crude *N*-(4-pyridyl)glycyl chloride hydrochloride added in portions over a 30-min period. The cooling bath was then removed and the mixture allowed to come to room temperature over a 2-hr period. To this mixture was added 150 ml of water and the pH adjusted to 1.8 with 20% NaOH. The slurry was then filtered and the aqueous layer separated from the filtrate. The aqueous solution was stirred 15 min with 2 g of decolorizing carbon (Darko-KB) and filtered and the pH adjusted to 3 with 20% NaOH under a layer of 150 ml of ether. The product crystallized and after 10 min stirring was cooled at 0° for 30 min. The product was collected by filtration, washed with water and then acetone, and air-dried. After drying 18 hr over P₂O₅ the yield was 7.01 g (59%), mp 192°. *Anal.* (C₁₇H₁₈N₄O₆S·H₂O) H, N; C: calcd 48.11; found, C, 48.56.

7-[α -(1,3-Diethylformamidino-2-thio)acetamido]cephalosporanic Acid (10).¹¹ To a stirred solution of 3.93 g (0.01 mol) of 3 and 1.4 ml (0.01 mol) of TEA in 50 ml of CH₂Cl₂ and 10 ml of acetone was added 1.32 g (0.01 mol) of *N,N'*-diethylthiourea (Eastman). The slightly turbid solution was filtered and after stirring for 30 min the crystalline precipitate was collected by filtration, washed well with CH₂Cl₂, air-dried, and vacuum-dried over P₂O₅. The yield was 3.05 g (68%), mp 130°. *Anal.* (C₁₇H₂₄N₄O₆S₂) C, H, N.

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Additions and Corrections

1972, Volume 15

Raymond D. Kimbrough, Jr.: Synthesis and Oral Hypoglycemic Activity of *N*-(*p*-Deuteriomethylbenzenesulfonyl)-*N'*-*n*-butylurea, Deuterium-Substituted Tolbutamide.

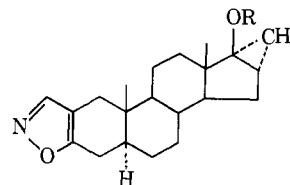
Page 409. Reference to prior work on this subject by R. U. Lemieux, K. F. Sporek, I. O'Reilly, and E. Nelson, *Biochem. Pharmacol.*, **7**, 31 (1961), was omitted. The results published are in agreement with the prior definitive work of Lemieux, *et al.*

T. Kametani, M. Ihara, T. Suzuki, T. Takahashi, R. Iwaki, H. Takei, N. Miyake, M. Yoshida, Y. Hasegawa, and H. Kitagawa: Studies on the Syntheses of Heterocyclic Compounds. 459. Synthesis of Rescinnamine-Like Compounds as Antihypertensive Agents.

Page 686. In Table I, R₂ of compound 12 and R₃ of compound 13 should be OCO₂C₂H₅.

Kenneth E. Fahrenholtz, Kenneth P. Meyers, and R. W. Kierstead: Cycloprop[16 α ,17 α]androstanes.

Page 1057. Structure 23 should be corrected to read



Page 1058. Footnote *a* in Table II should be changed from $p < 0.0001$ to $p < 0.001$.