Mitchell A. Avery, Michael S. Verlander, and Murray Goodman.* Synthesis of 6-Aminoisoproterenol.

Page 2750. The synthesis of 6-aminoisoproterenol 1 and 6acetamidoisoproterenol 3 as previously described¹ are incorrect. We actually isolated and characterized the ethyl ethers 2 and 4 which had formed when 1 and 3 were suspended in ethanol in the presence of acid.² After crystals had been collected from the ethanol it was thought that the appearance of the ethyl group in the NMR and by elemental analysis was due to cocrystallization of 1 equiv of ethanol.



The synthesis of 1 and 3 from the benzyl ether precursors 5 and 6 has been repeated under conditions that avoid ether formation. An examination of the NMR spectra of 1 and 3 show clearly that the earlier compounds were in fact the ethyl ethers 2 and 4.

Both 6-aminoisoproterenol 1 and 6-acetamidoisoproterenol 3 have been tested in vitro for β -adrenergic activity and both have been found to be inactive.

6-Aminoisoproterenol Dihydrochloride (1). A solution of 5 (20 mg or 0.042 mmol) in acetic acid (4 mL) with 10% Pd/C (2 mg) was stirred under 1 atm of H₂ for 17 h. The solution was filtered through Celite under N₂ into 4 mL 0.1 N HCl. The solvent was evaporated and the product was purified on HPLC, using a Whatman Magnum ODS-3 column (0.01 N HCl) to yield 8.0 mg or 63.3% of an amorphous, air-sensitive solid that was pure by HPLC (>99%): NMR (360 MHz, Me₂SO-d₆) δ 8.89 (br s, 1 H), 8.27 (br s, 1 H), 6.94 (s, 1 H), 6.91 (s, 1 H), 5.08 (d, 1 H), 3.20 (br s, 6 H), 3.33 (m, 1 H), 3.20 (a) (pr of t, 2 H), 1.25 (t, 6 H). Peaks at δ 8.87, 8.27, and 3.82 disappeared on addition of D₂O. Although the extreme lability of 1 toward oxidation precluded elemental analysis, the more stable, crystalline derivative 2 analyzed correctly for C, H, N, and Cl.¹

6-Acetamidoisoproterenol Hydrochloride (3). This compound was prepared in the same way as 1, using the following quantities: 48 mg of 6 (0.099 mmol), HOAc (5 mL), and 10% Pd/C (5 mg). HPLC purification produced 22.4 mg or 74% yield of an amorphous, air-sensitive solid that was >99% pure by HPLC: NMR (360 MHz, Me₂SO-d₆) δ 9.34 (s, 1 H), 8.81 (br s, 1 H), 8.50 (br s, 1 H), 6.88 (s, 1 H), 6.65 (s, 1 H), 4.93 (d, 1 H, J = 9.5 Hz), 3.68 (br s, 3 H), 3.31 (m, 1 H), 3.09, 2.81 (pr of t, 2 H, J = 9.5 Hz), 2.06 (s, 3 H), 1.26 (t, 3 H). Peaks at δ 9.34, 8.81, 8.50, and 3.68 disappeared upon addition of D₂O. The ethyl ether derivative 4 analyzed correctly for C and N.¹

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(1) M. Avery, M. Verlander, and M. Goodman, J. Org. Chem., 45, 2750 (1980).

(2) The benzylic hydroxyl of catecholamines is known to undergo ether formation under similar conditions. C. Funk and L. Freedman, J. Am. Chem. Soc., 45, 1792 (1923); B. Tullar, *ibid.*, 70, 2067 (1948); K. Kirk, D. Cantacuzene, Y. Nimitkitpaisan, D. McCulloh, W. Padgett, J. Daly, and C. Creveling, J. Med. Chem., 22, 1493 (1979).

R. R. Heath, R. E. Doolittle, P. E. Sonnet,* and J.H. Tumlinson. Sex Pheromones of the White Peach Scale: Highly Stereoselective Synthesis of the Stereoisomers of Pentagonol Propionate.

Page 2912. Column 1, tenth line from bottom, the optical rotation data for the $R_{,Z}$ isomer should read: $[\alpha]^{25}_{D}$ -6.03° (c 10.5, CHCl₃). Other rotational data reported are correctly stated. We thank Dr. R. Carney of Zoecon Corp., Palo Alto, for bringing the error to our attention.

F. W. Vierhapper,* E. L. Eliel,* and G. Zuñiga. Carbon-13 Spectra of Saturated Heterocycles. 10. Effect of Lone Electron Pairs on Nitrogen on the Chemical Shift of Antiperiplanar Vicinal Methyl Carbons.

Page 4846. Table IV, entry for C-6 for compound 20 should read 27.1_4 .

Herman G. Richey, Jr.* and Rouvain M. Bension. Stereochemistry of Addition of Allylic Grignard Reagents to 3-(Hydroxymethyl)cyclopropanes.

Page 5040. The upper right-hand H in structure I and the lower right-hand H in structure II should be boldface.

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Francoise R. Quigley and Heinz G. Floss.^{*} Mechanism of Amino Acid α -Hydroxylation and Formation of the Lysergyl Moiety in Ergotamine Biosynthesis.

Page 464. Column 2: The configurations of compounds II and I at C-2 of the alanine or α -hydroxyalanine moiety, respectively, are incorrect as shown. Compound I should have R and II should have S configuration at that center. The error in the drawing does not affect the conclusions and interpretations given in the text.

Anjani J. Varma and Conrad Schuerch.* Synthesis of Substituted 2,6-Dioxabicyclo[3.1.1]heptanes. 1,3-Anhydro-2,4,6-tri-O-(p-bromobenzyl)- β -D-mannopyranose.

Page 800. Column 1, paragraph 2, the first sentence should read: "In previous syntheses of 1,2-anhydro- and 1,3-anhydroglycopyranoses, a key intermediate has been a 1,2- or 1,3-diol, which has then been converted to the corresponding glycosyl chloride."

C. Wentrup,* B. Gerecht, D. Laqua, H. Briehl, H.-W. Winter, H. P. Reisenauer, and M. Winnewisser. Organic Fulminates, R-O-NC.

Page 1047. Figure 1, the formula given in the IR spectrum of Figure 1b should read as follows: C_6H_5 -O-NC.

Jang B. Rampal, K. Darrell Berlin,* James P. Edasery, and N. Satyamurthy. Carbon-Phosphorus Heterocycles. Synthesis and Conformational Analysis of Alkyl-Substituted 1,2,6-Triphenyl-4-phosphorinanones and Derivatives.

Page 1171. Table III, lines five and six of column three, the data currently read as follows: 41.41 (3.63), 52.95 (4.38) and 42.28 (0.0), 51.21 (1.97). The data should read as follows: 52.95 (4.38), 41.41 (3.63) and 51.21 (1.97), 42.28 (0.0).

Page 1171. Table III, line six of column five, "13.88 (2.23)" should read "13.88 (2.69)".