

(+)-2-*d*-mandelate (1.6 g) was converted to the (+)-2 base (0.93 g, 94%) of mp 219–222 °C, as described above for the enantiomer (–)-2. Recrystallization from EtOH gave fine needles of pure (+)-2: mp 225–226 °C; $[\alpha]_D^{27} +28.9^\circ$ (c 1.14, 1 N HCl). Anal. (C₁₆H₂₃NO) C, H, N.

(–)-1,4,12β-Trimethyl-10-hydroxy-2,3,4,5,6,7-hexahydro-1,6-methano-1*H*-4-benzazonine [(–)-3]. To a suspension of 3 (2.71 g, 11.05 mmol) in MeOH (40 mL) was added *d*-tartaric acid (0.83 g, 5.53 mmol) in MeOH (10 mL). The mixture was heated to solution and allowed to stand at room temperature for 5 h. The solid was filtered, washed with cold MeOH, and dried to give 1.1 g of fine cubes. Three recrystallizations from MeOH gave optically pure (–)-3-*d*-tartrate: yield 0.91 g (50.8%); mp 208–211 °C; $[\alpha]_D^{27} -17.7^\circ$ (c 2.0, H₂O). Anal. (C₃₂H₄₆N₂O₂·C₄H₆O₆) C, H, N. The neutral tartrate (0.9 g) was dissolved in H₂O (20 mL), basified with NH₄OH, filtered, washed with H₂O, and dried to give 0.67 g (97%) of (–)-3. Recrystallization from MeOH–Me₂CO (9:1) gave pure (–)-3 as prisms: mp 175–176 °C; $[\alpha]_D^{29} -34.3^\circ$ (c 1.53, 1 N HCl). Anal. (C₁₆H₂₃NO) C, H, N.

(+)-1,4,12β-Trimethyl-10-hydroxy-2,3,4,5,6,7-hexahydro-1,6-methano-1*H*-4-benzazonine [(+)-3]. The combined filtrates and washings from 0.9 g of (–)-3-*d*-tartrate were evaporated to a syrup and dissolved in H₂O (50 mL), made alkaline with NH₄OH, filtered, washed with H₂O, and dried to give 1.8 g of a solid mixture of (±) and (+) bases. The mixed bases (1.8 g, 7.35 mmol) were heated to solution with *l*-tartaric acid (0.56 g, 3.73 mmol) in MeOH (30 mL). The solution was allowed to stand at room temperature for 6 h. The resulting crystals were filtered, washed with cold MeOH, and dried to give 1.1 g of fine cubes. Two recrystallizations from MeOH gave optically pure (+)-3-*l*-tartrate: yield 0.97 g (58%); mp 207–210 °C; $[\alpha]_D^{27} +17.4^\circ$ (c 2.0, H₂O). Anal. (C₃₂H₄₆N₂O₂·C₄H₆O₆) C, H, N. The neutral tartrate (0.97 g) was converted to the (+)-3 base (0.69 g, 99%) as described above for its enantiomer (–)-3. Recrystallization from MeOH–

Me₂CO (9:1) gave pure (+)-3: prisms; mp 173–175 °C; $[\alpha]_D^{29} +36.3^\circ$ (c 1.53, 1 N HCl). Anal. (C₁₆H₂₃NO) C, H, N.

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References and Notes

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- (4) Groups of ten albino male mice, dd strain, were tested at five dose levels. ED₅₀ and LD₅₀ values were calculated by the Lichfield–Wilcoxon method.⁹
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Additions and Corrections

1978, Volume 21

James Z. Ginos,* George C. Cotzias, and David Doroski: New Dopaminergic and Potential Anti-Parkinson Compounds, *N,N*-Disubstituted β-(3,4-Dihydroxyphenyl)ethylamines..

Page 162. In Table III, the melting point for compound 25 should read as 136.5–137.5 °C, after crystallizing twice from absolute ethanol–ether.

1979, Volume 22

Arthur E. Jacobson,* Kenner C. Rice, Jurgen Reden, Lillian Lupinacci, Arnold Brossi, Richard A. Streaty, and Werner A. Klee: Paradoxical Effects of *N*-Cyanoalkyl Substituents upon the Activities of Several Classes of Opioids.

Page 329. In Table II, footnote *d* should read as follows: Binding constant from rat brain homogenates, in nmol/L.