Rejzek M., Stockman R. A. and Hughes D. L.:  

Combining Two-Directional Synthesis and Tandem Reactions,  
Part 5: An Efficient Strategy for the Total Syntheses of (±)-Hippodamine and (±)-epi-Hippodamine  

SUPPLEMENTAL DATA  

Page 2  \(^{13}\)C NMR Spectra for Compounds 1, 2, 3, 7, 10, 13, 14, 18, 22, 23, and 25.  

$^{13}$C NMR Spectra

Compound 1: (100 MHz, CDCl₃)
Compound 2: (100 MHz, CDCl₃)
Compound 3: (100 MHz, CDCl₃)
Compound 7: (100 MHz, CDCl₃)
Compound 10: (100 MHz, CDCl$_3$)
Compound 13: (100 MHz, CDCl₃)
Compound 14: (100 MHz, CDCl₃)
Compound 18: (100 MHz, CDCl₃)
Compound 22: (100 MHz, CDCl₃)
Compound 23: (100 MHz, CDCl₃)
Compound 25: (100 MHz, CDCl$_3$)
Crystal structure analyses

Compound 22. Crystal data: C_{13}H_{23}NO, M = 209.3. Monoclinic, space group P2_1/n (equiv. to no. 14), a = 7.660(2), b = 14.610(3), c = 10.367(2) Å, β = 91.13(3)°, V = 1160.0(4) Å³. Z = 4, Dc = 1.199 g cm⁻³, F(000) = 464, T = 140(1) K, μ(Mo-Kα) = 0.7 cm⁻¹, λ(Mo-Kα) = 0.71069 Å. A clear, colourless tapered, rectangular prismatic crystal, ca 0.50 x 0.25 x 0.20 mm, was mounted on a Rigaku R-Axis IIc image-plate diffractometer. Total no. of reflections recorded to θ_{max} = 25.4°, was 6174 of which 2043 were unique (Rint = 0.052); 1851 were >observed= with I > 2σ(I). Refinement gave wR₂ = 0.103 and R₁ = 0.042 for all 2043 reflections weighted w = [σ²(F_o²) + (0.0384P)² + 0.38P]⁻¹ with P = (F_o² + 2F_c²)/3; for the 'observed' data only, R₁ = 0.039.

Compound 25. Crystal data: C_{13}H_{23}NO, M = 209.3. Monoclinic, space group P2_1/c (no. 14), a = 9.766(3), b = 9.337(2), c = 13.767(2) Å, β = 102.38(2)°, V = 1226.1(5) Å³. Z = 4, Dc = 1.134 g cm⁻³, F(000) = 464, T = 293(2) K, μ(Mo-Kα) = 0.7 cm⁻¹, λ(Mo-Kα) = 0.71069 Å. A clear, colourless plate crystal, ca 0.62 x 0.21 x 0.05 mm, was mounted on a Nonius CAD4 diffractometer; of the 2511 reflections measured to θ_{max} = 25°, 2146 were unique (Rint = 0.012) and 1263 'observed' with I > 2σ(I). Refinement gave wR₂ = 0.136 and R₁ = 0.081 for all 2146 reflections weighted w = [σ²(F_o²) + (0.0645P)²]⁻¹; for the 'observed' data only, R₁ = 0.047.

Both structures were determined by direct methods procedures in SHELXS and refined by full-matrix least-squares methods, on F²'s, in SHELXL. In both structures, the hydroxyl hydrogen atom was located in a difference Fourier map and refined freely. In the final difference maps, the highest peaks were ca 0.19 eÅ⁻³ in 22 and ca 0.15 eÅ⁻³ in 25.

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(1) G. M. Sheldrick, SHELX-97 - Programs for crystal structure determination (SHELXS) and refinement (SHELXL), University of Göttingen, Germany (1997).