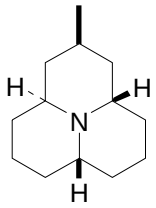
Rejzek M., Stockman R. A. and Hughes D. L.:

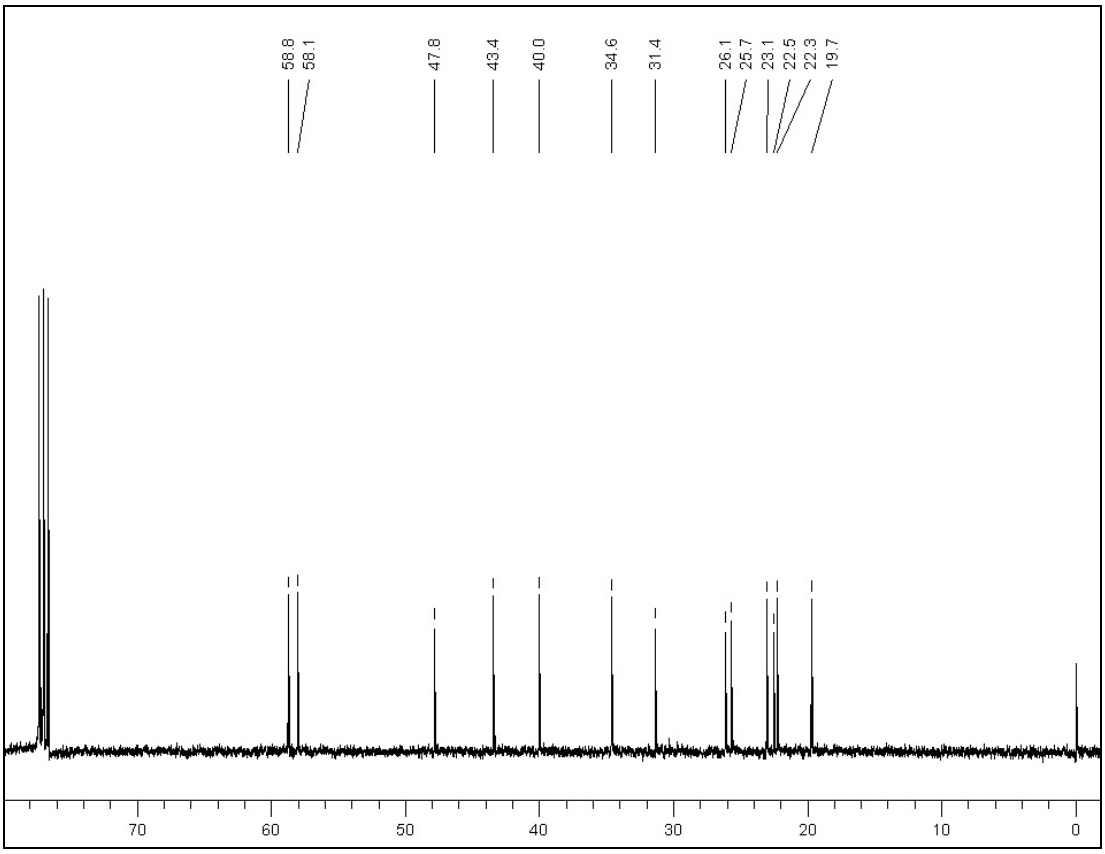
Combining Two-Directional Synthesis and Tandem Reactions, Part 5: An Efficient Strategy for the Total Syntheses of (±)-Hippodamine and (±)-*epi*-Hippodamine

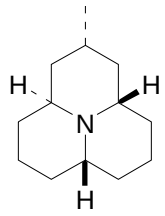
SUPPLEMENTAL DATA

^{13}C NMR Spectra

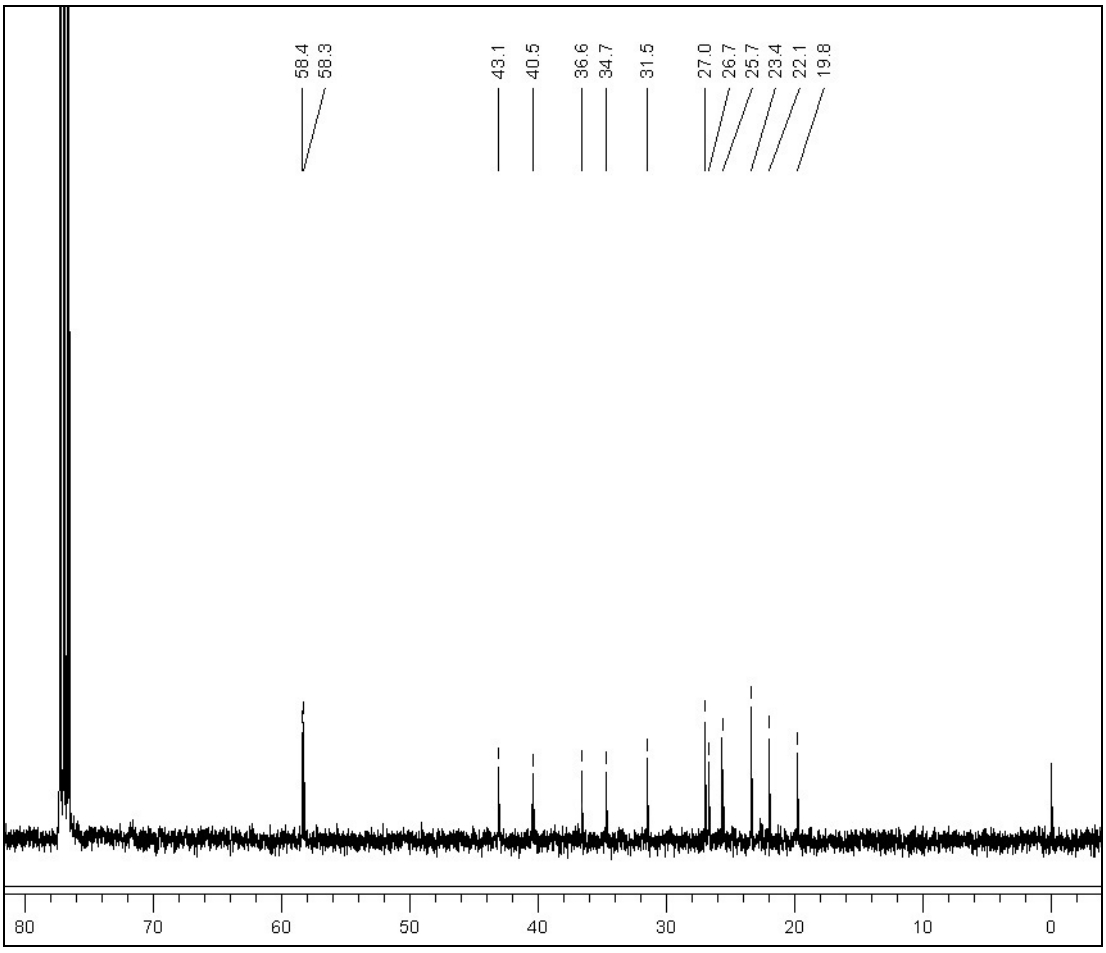


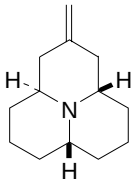
Compound **1**: (100 MHz, $CDCl_3$)



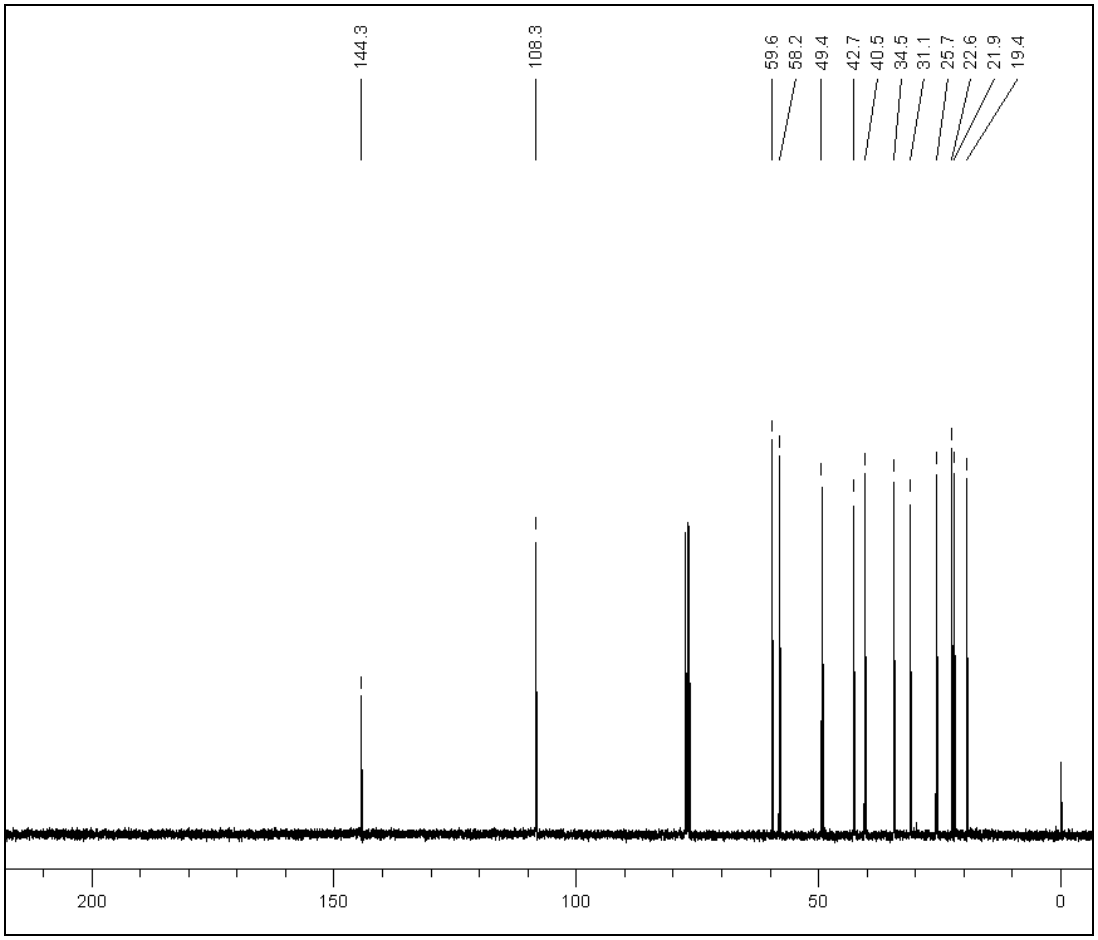


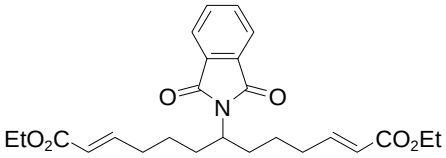
Compound **2**: (100 MHz, $CDCl_3$)



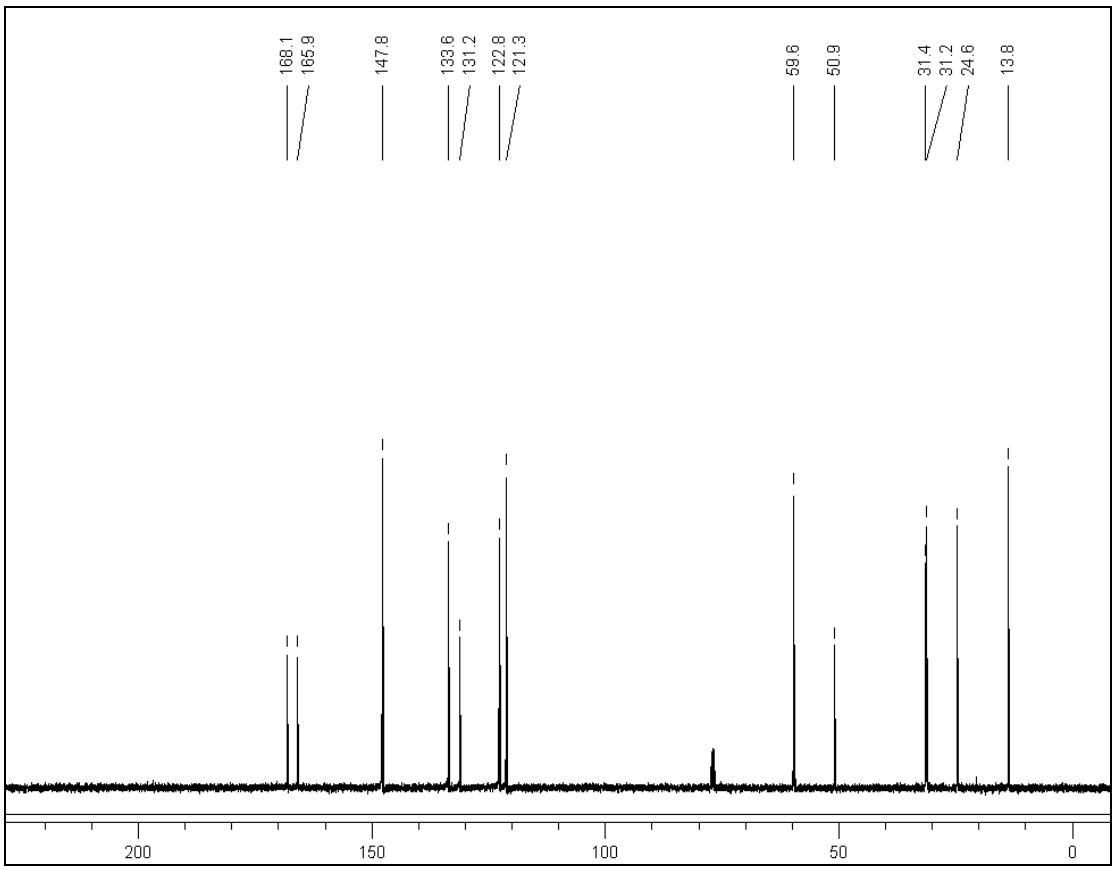


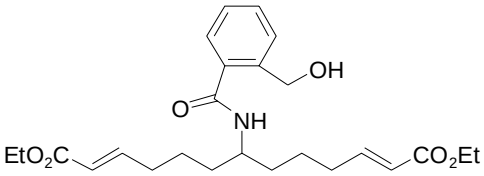
Compound **3**: (100 MHz, $CDCl_3$)



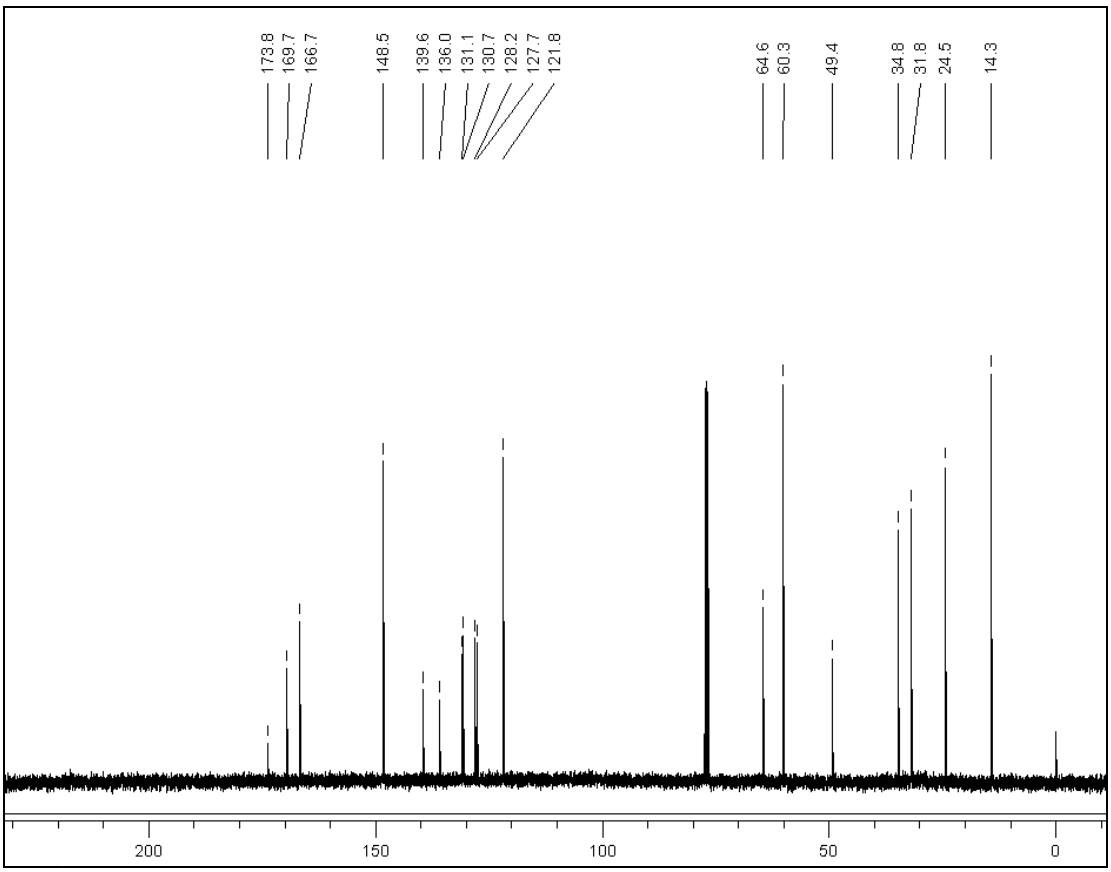


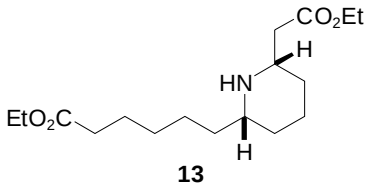
Compound **7**: (100 MHz, $CDCl_3$)





Compound **10**: (100 MHz, $CDCl_3$)





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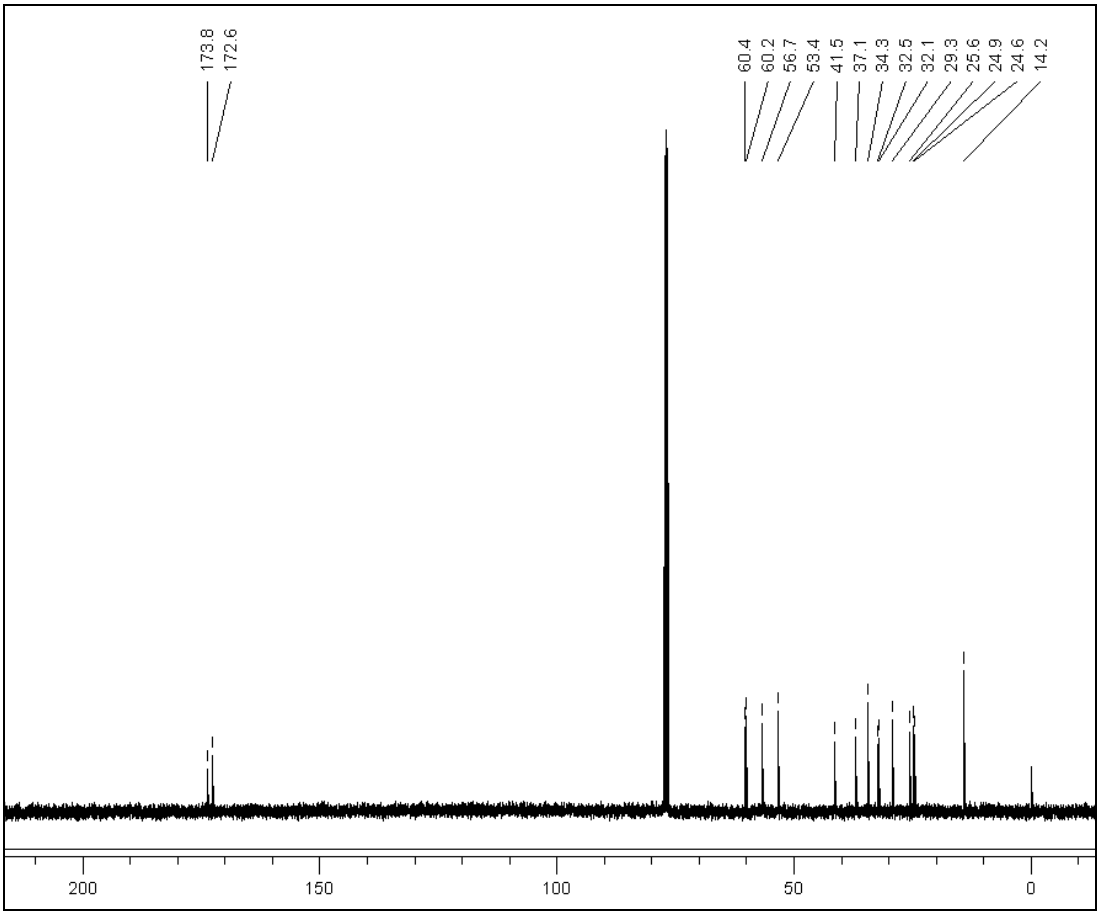
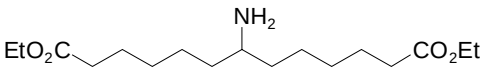
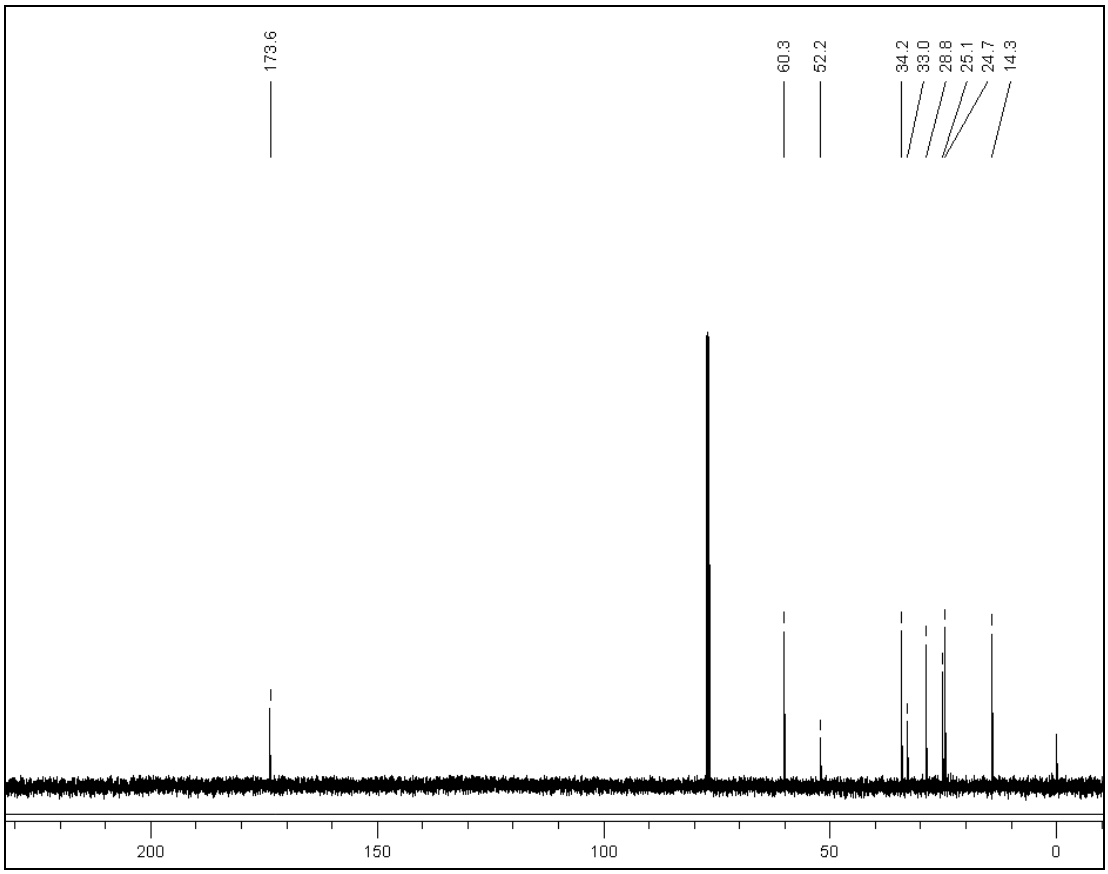
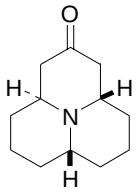
Compound **13**: (100 MHz, $CDCl_3$)



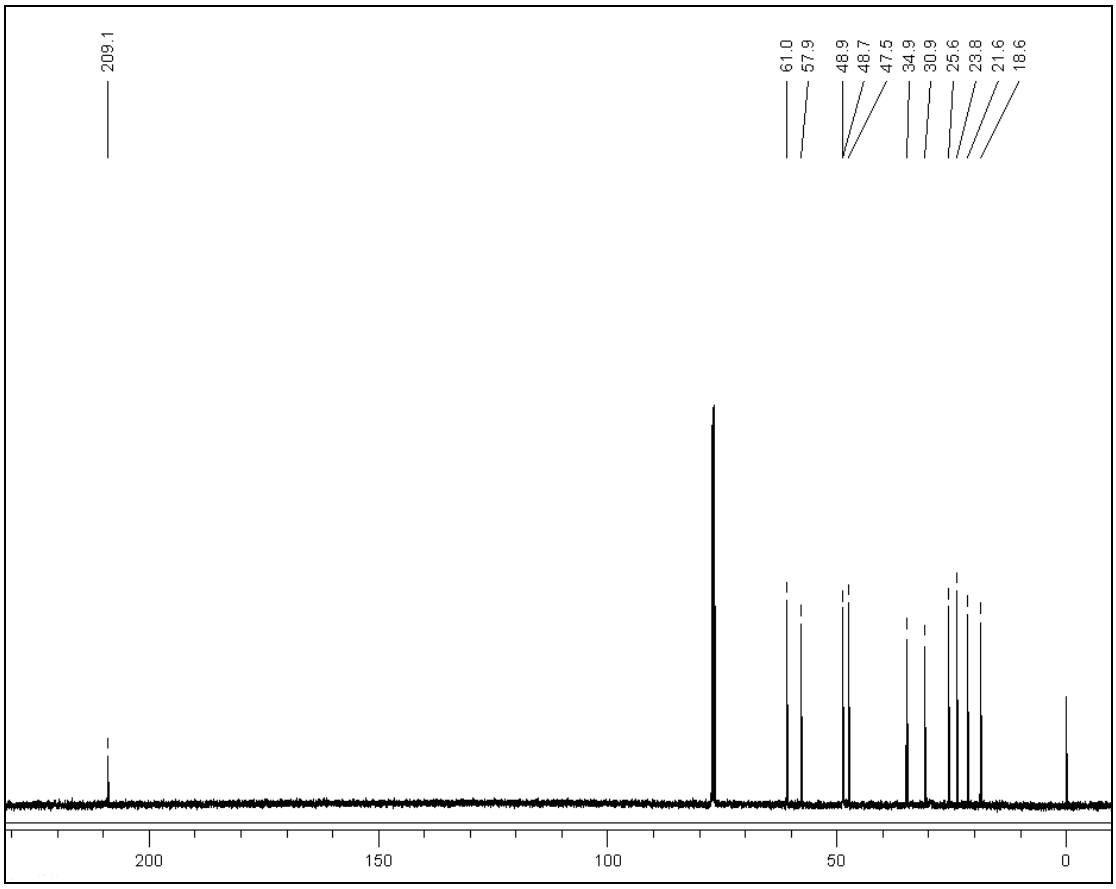


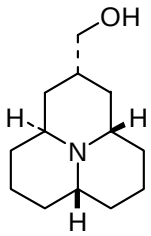
Compound **14**: (100 MHz, $CDCl_3$)



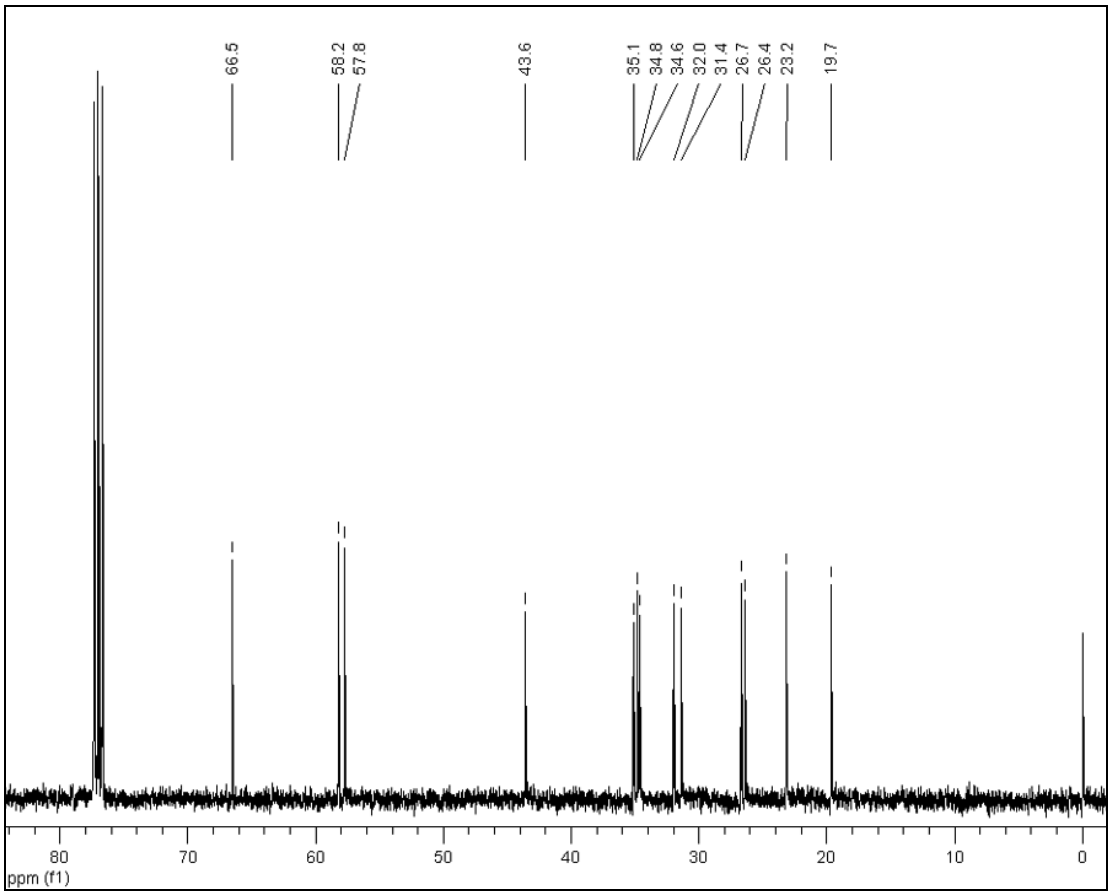


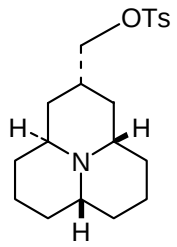
Compound **18**: (100 MHz, $CDCl_3$)



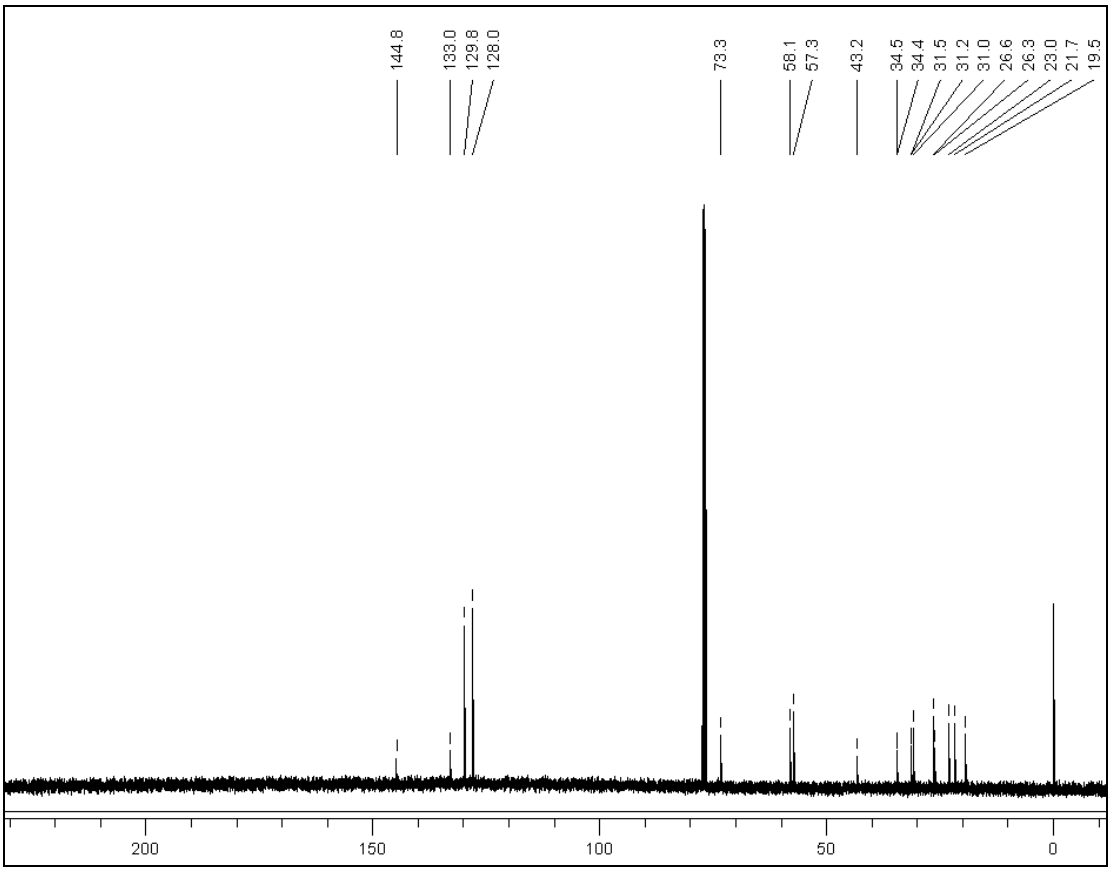


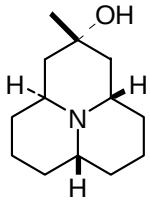
Compound **22**: (100 MHz, $CDCl_3$)



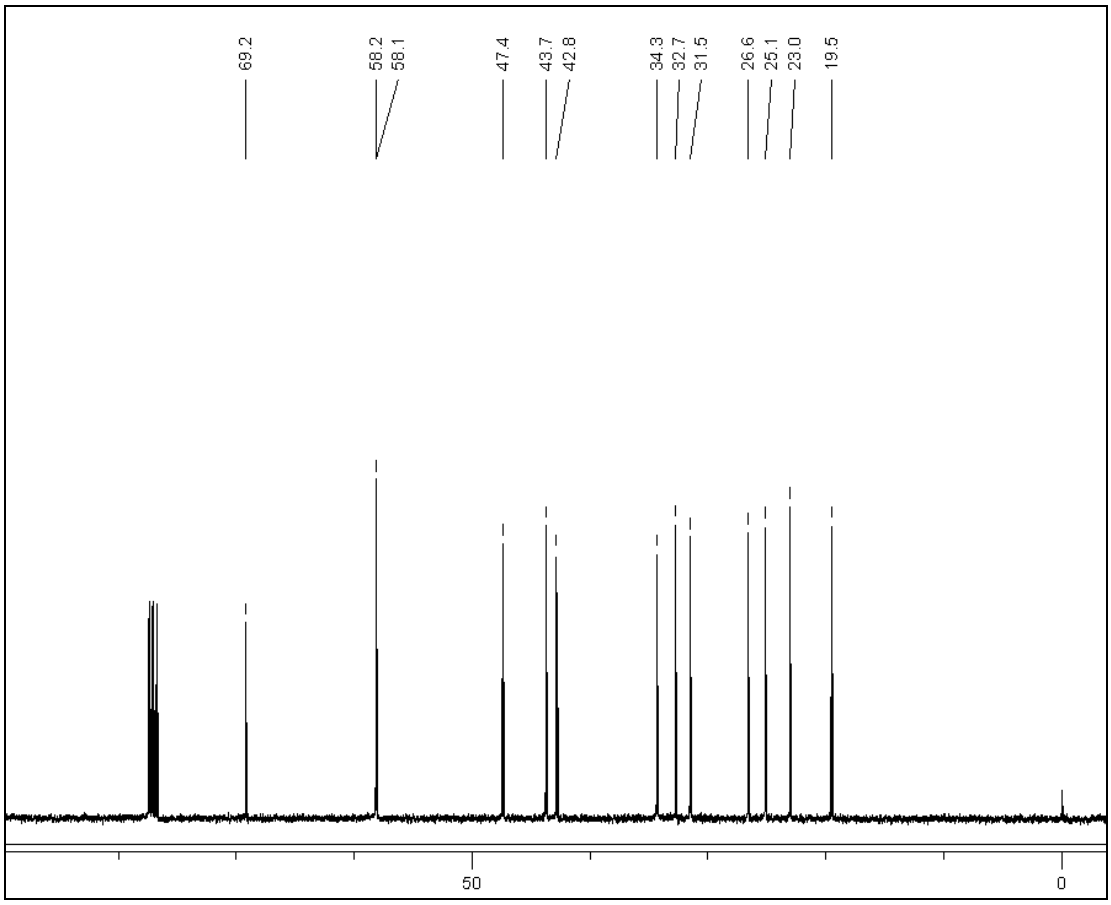


Compound **23**: (100 MHz, $CDCl_3$)





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) E, V = 1160.0(4) $Å^3$. 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} = 25.4E, was 6174 of which 2043 were unique (Rint = 0.052); 1851 were >observed= with I > $2\sigma_I$. Refinement gave wR_2 = 0.103 and R_1 = 0.042 [1] for all 2043 reflections weighted w = $[\sigma^2(F_o^2) + (0.0384P)^2 + 0.38P]^{-1}$ with P = $(F_o^2 + 2F_c^2)/3$; for the 'observed' data only, R_1 = 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) E, V = 1226.1(5) $Å^3$. 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} = 25E, 2146 were unique (Rint = 0.012) and 1263 'observed' with I > $2\sigma_I$. Refinement gave wR_2 = 0.136 and R_1 = 0.081 [1] for all 2146 reflections weighted w = $[\sigma^2(F_o^2) + (0.0645P)^2]^{-1}$; for the 'observed' data only, R_1 = 0.047.

Both structures were determined by direct methods procedures in SHELXS [1] and refined by full-matrix least-squares methods, on F^2's, in SHELXL [1]. 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**.

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