Supporting Information

Synthetic studies towards the natural product tripartin, the first natural histone lysine demethylase inhibitor

Dattatraya H. Dethe*[a] and Raghavender Boda[ab]

[a] Department of Chemistry, Indian Institute of Technology–Kanpur, Kanpur – 208016, India
[b] Aurigene discovery technologies limited, Bengaluru, Karnataka – 560100, India
Tel: + 91-512-2596537, Fax**:** + 91-512-2597436.

Table of Contents:

Figure S1a: ^{1}H NMR spectrum of compound 8

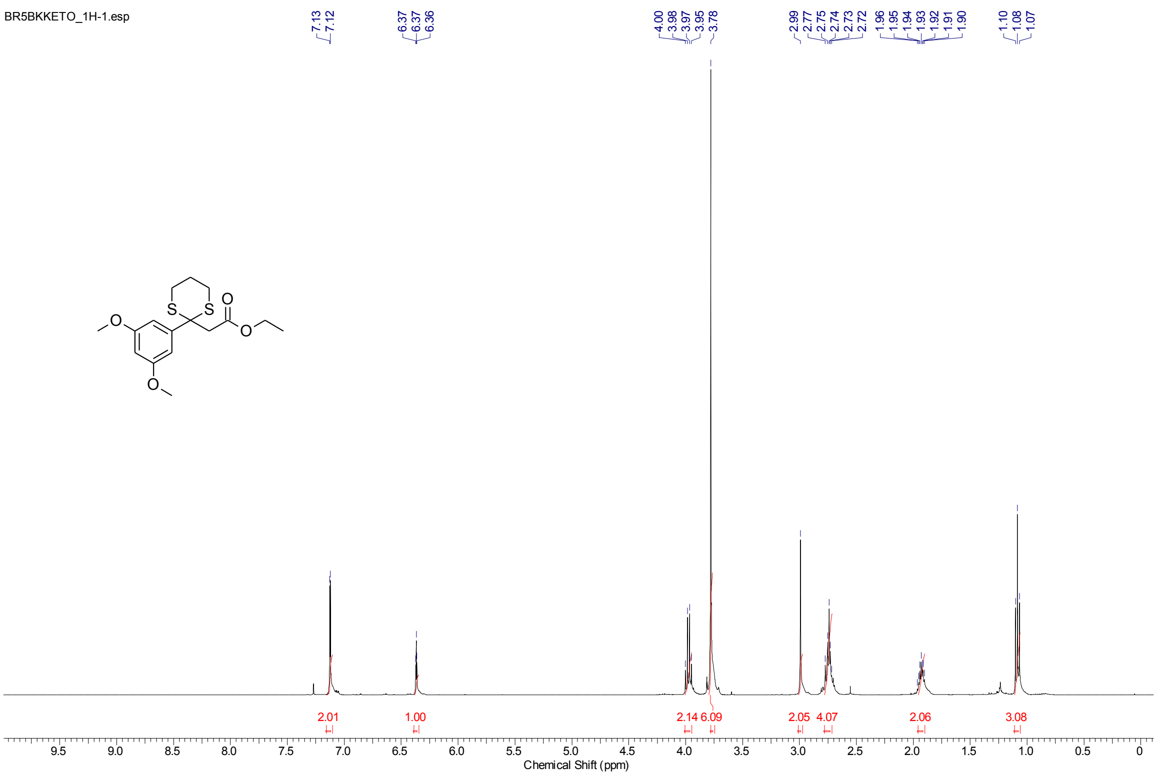


Figure S1b: ^{13}C NMR spectrum of compound 8

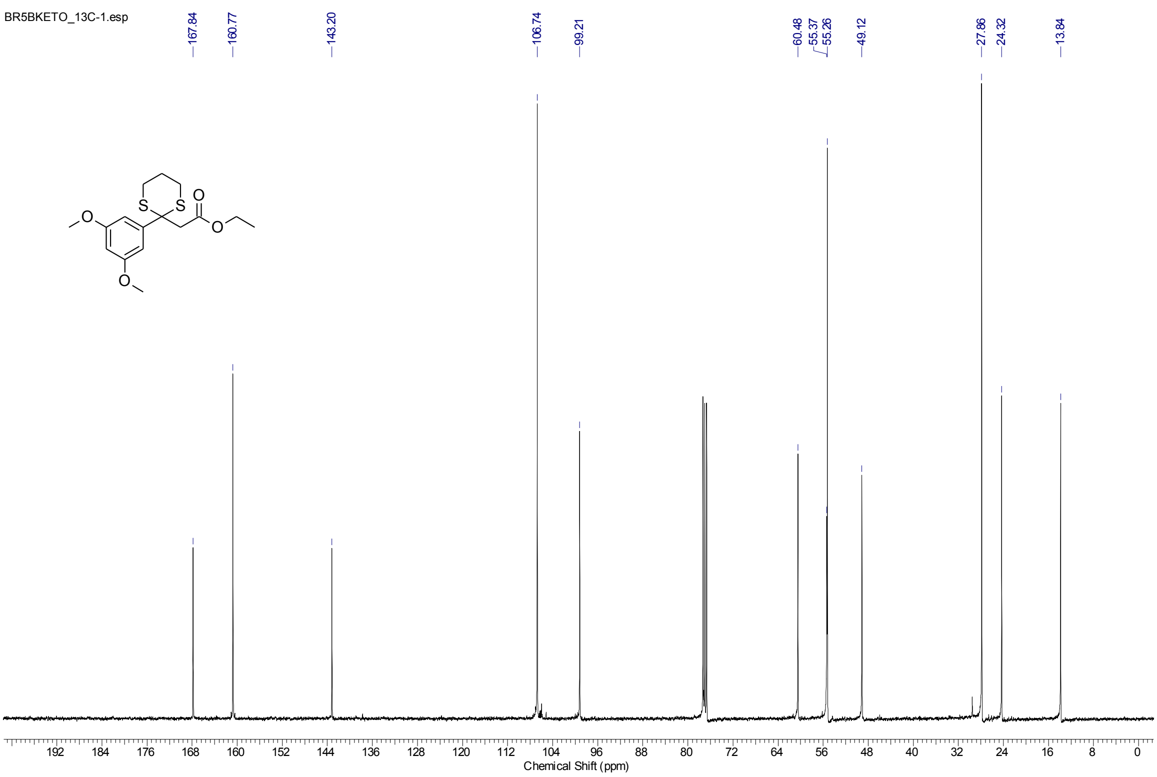


Figure S2a: ^{1}H NMR spectrum of compound 10

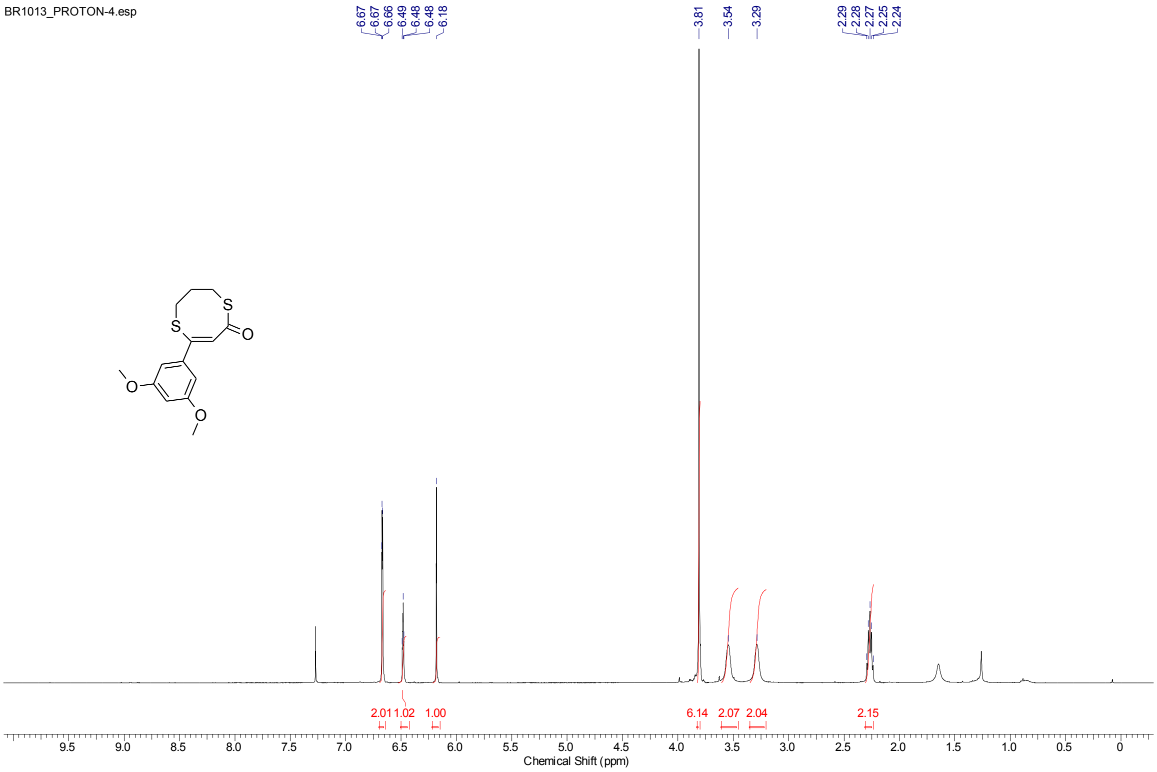


Figure S2b: ^{13}C NMR spectrum of compound 10

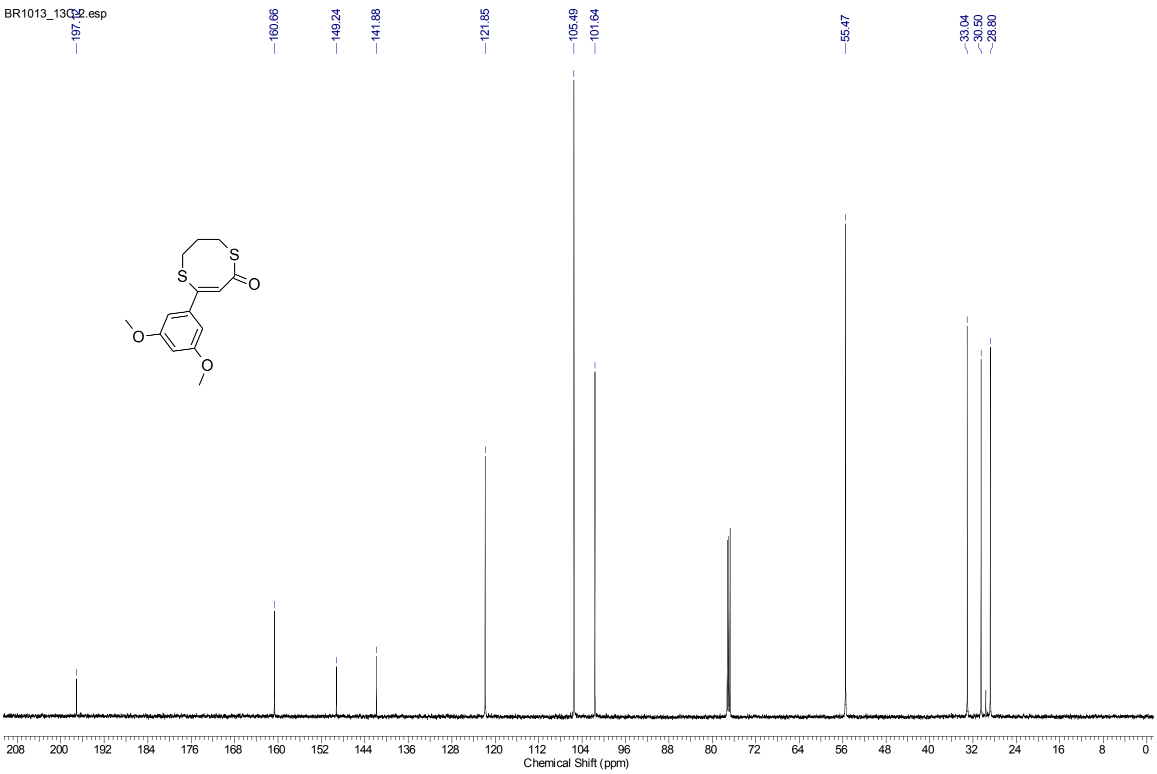


Figure S2c: DEPT spectrum of compound 10

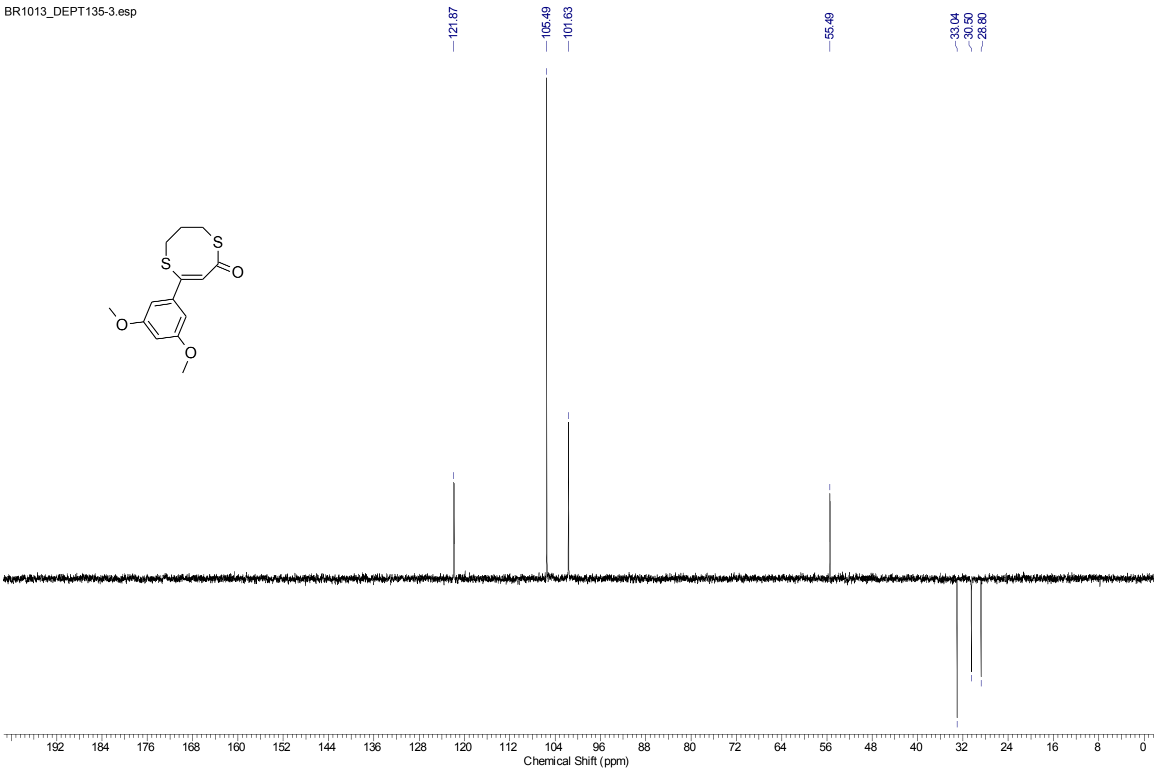


Figure S3a: ^{1}H NMR spectrum of compound 14

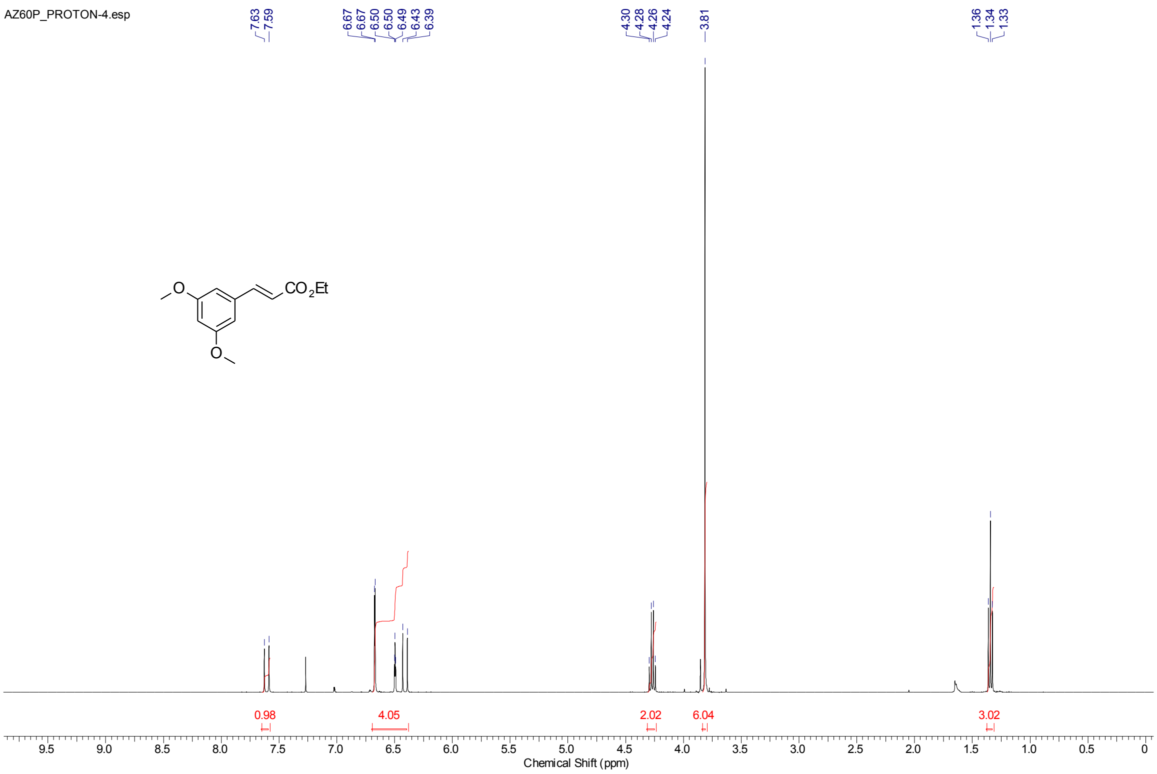


Figure S3b: ^{13}C NMR spectrum of compound 14

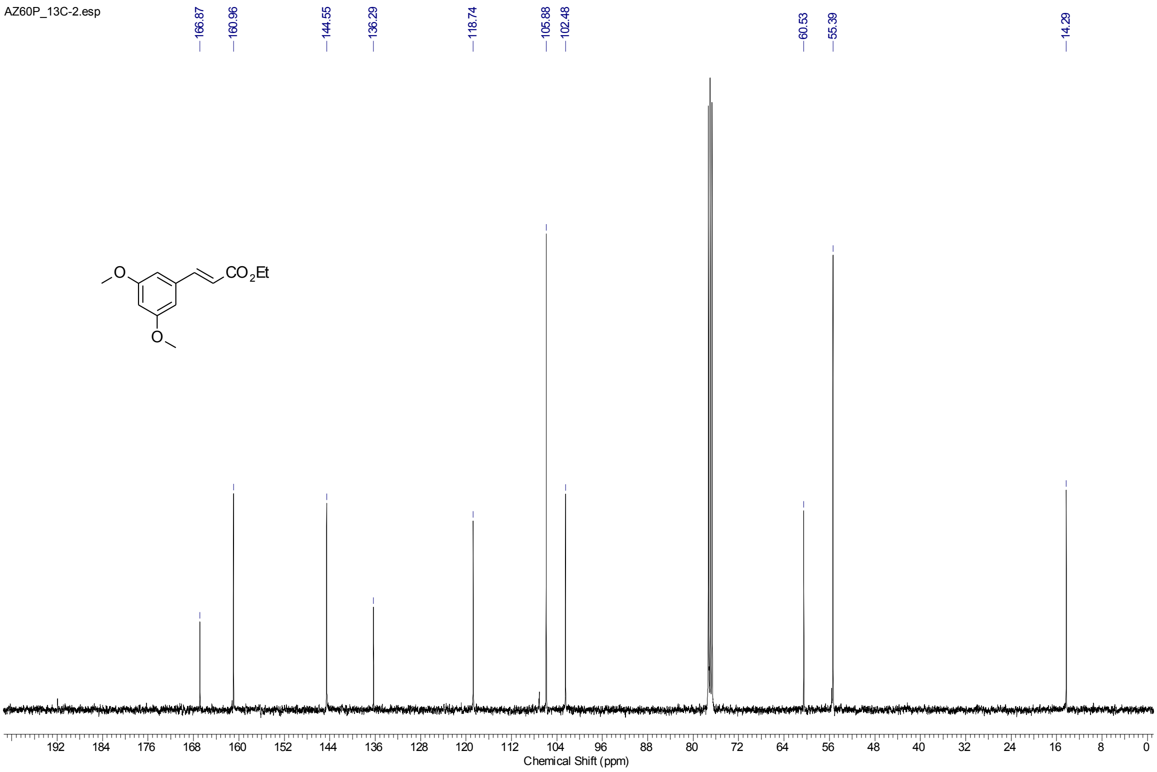


Figure S4a: ^{1}H NMR spectrum of compound 15

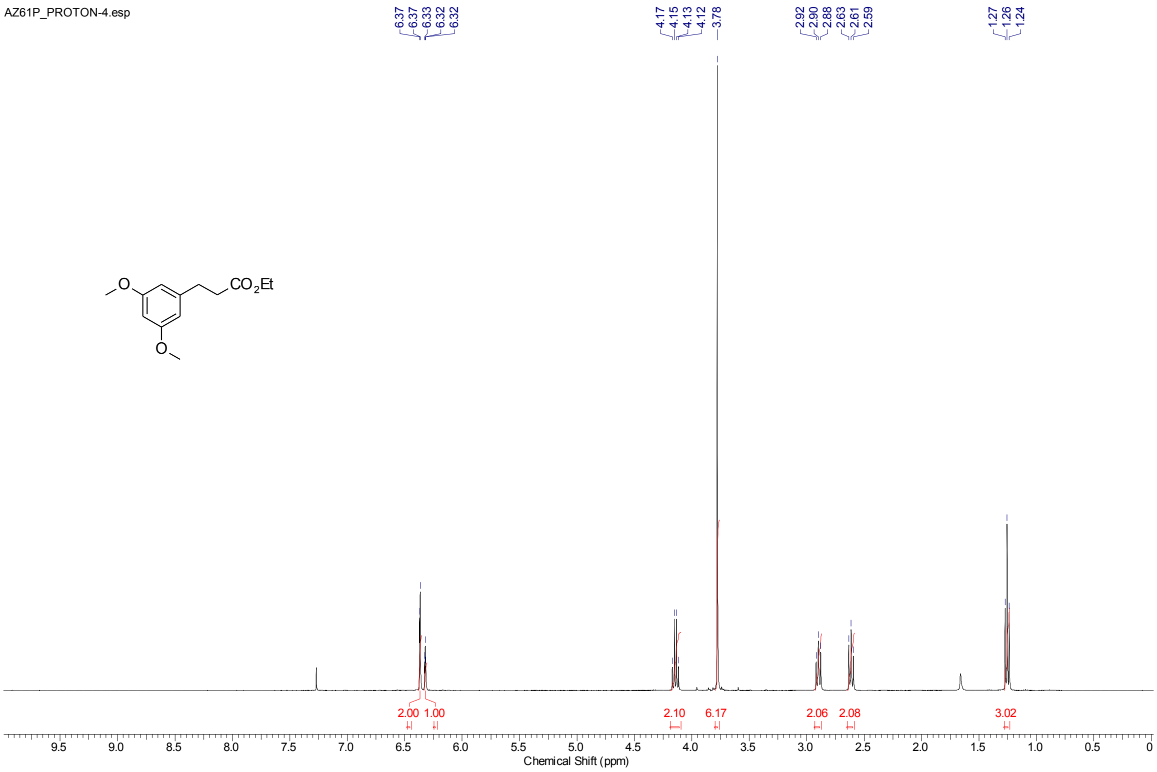


Figure S4b: ^{13}C NMR spectrum of compound 14

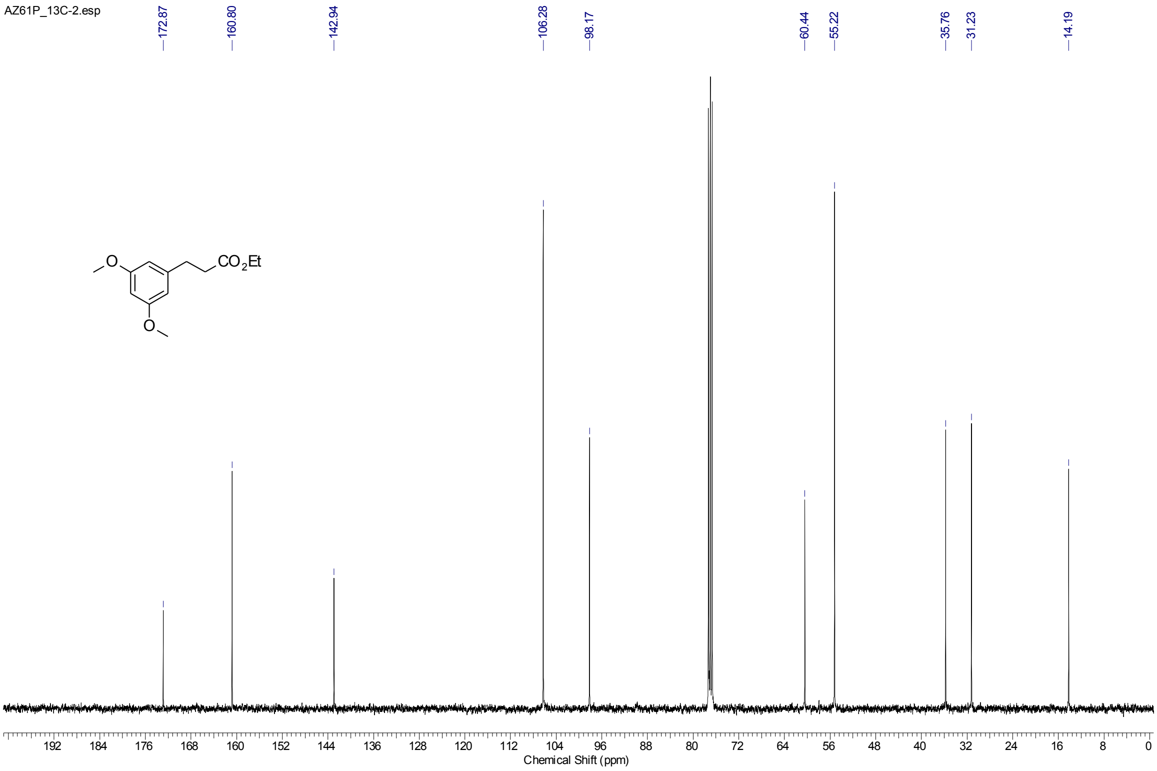


Figure S5a: ^{1}H NMR spectrum of compound 12

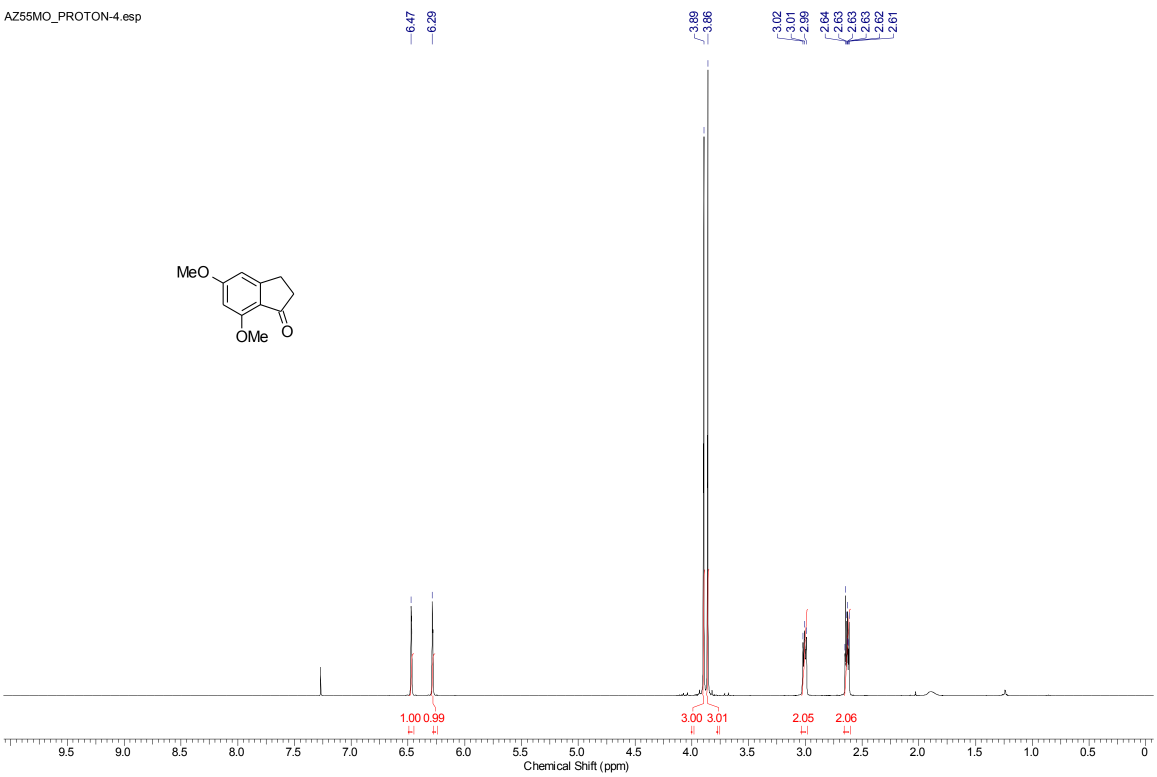


Figure S5b: ^{13}C NMR spectrum of compound 12

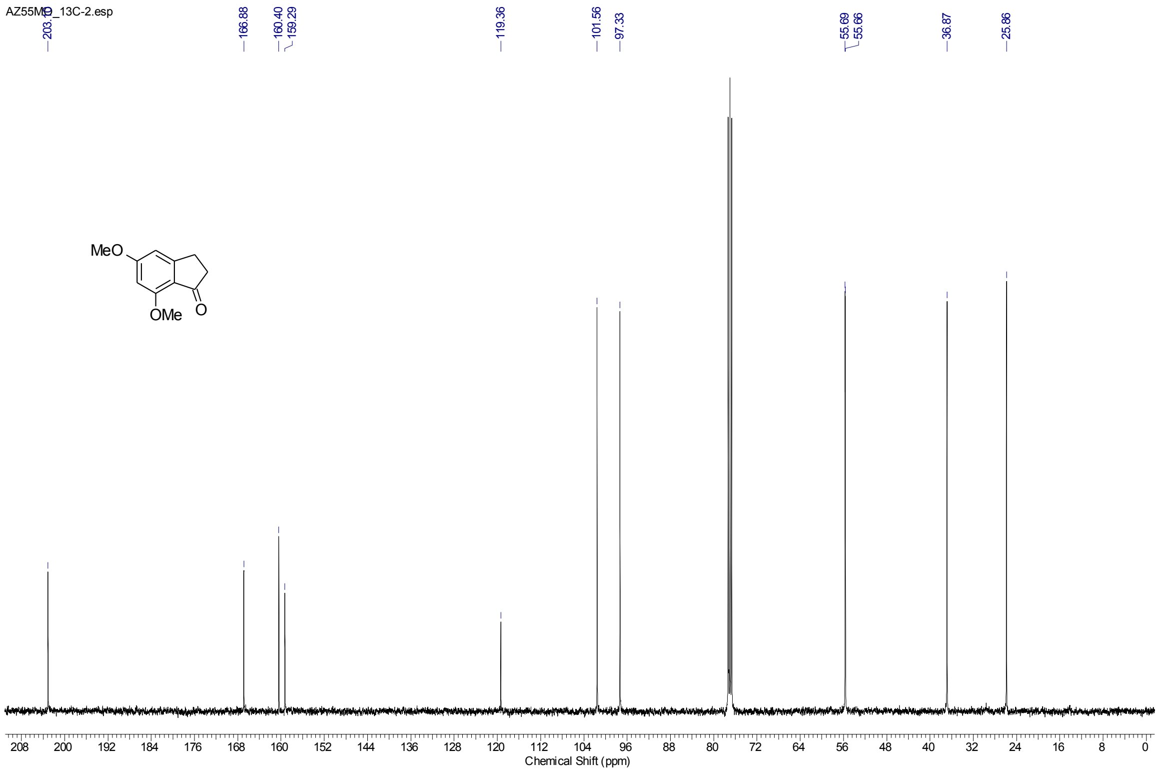


Figure S6a: ^{1}H NMR spectrum of compound 11

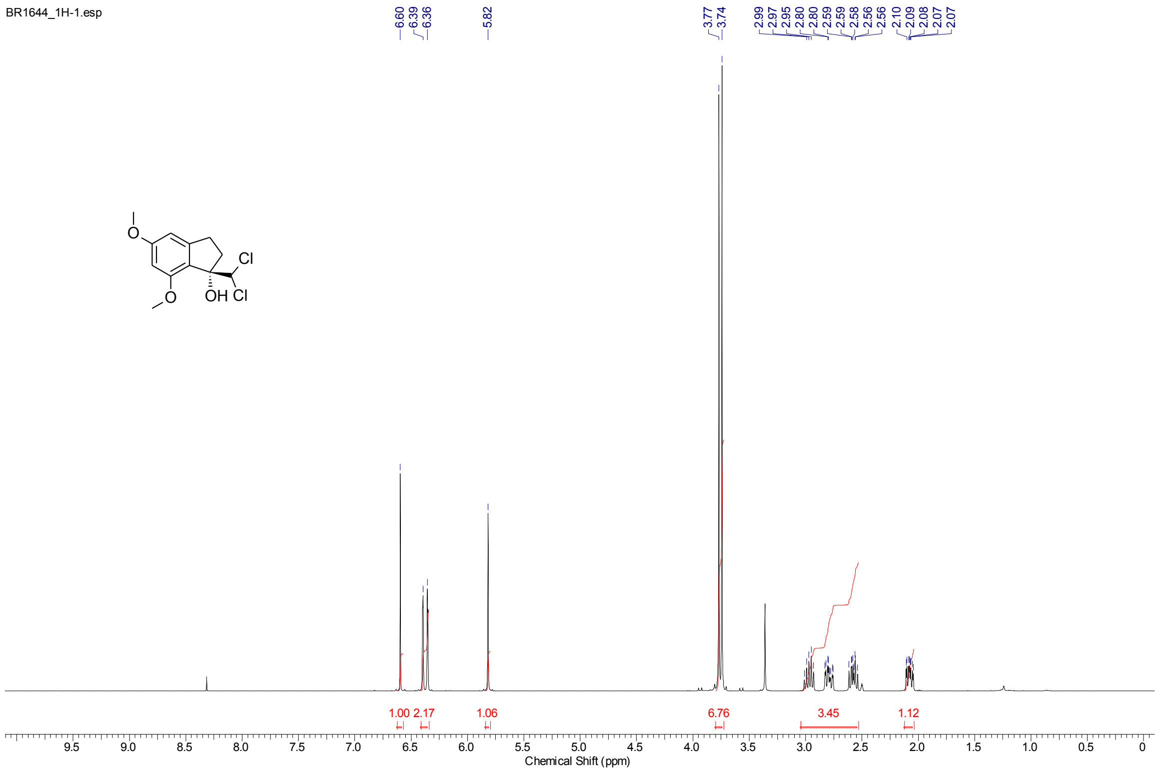


Figure S6b: ^{13}C NMR spectrum of compound 11

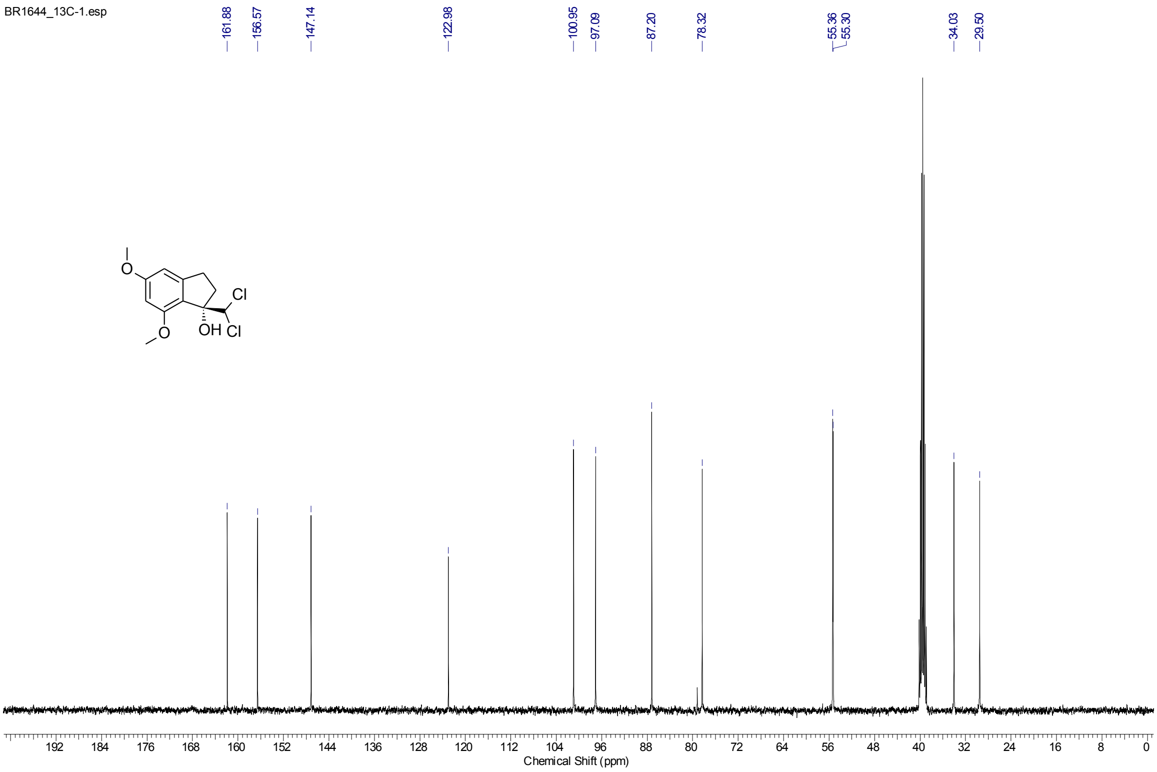


Figure S6c: DEPT spectrum of compound 11

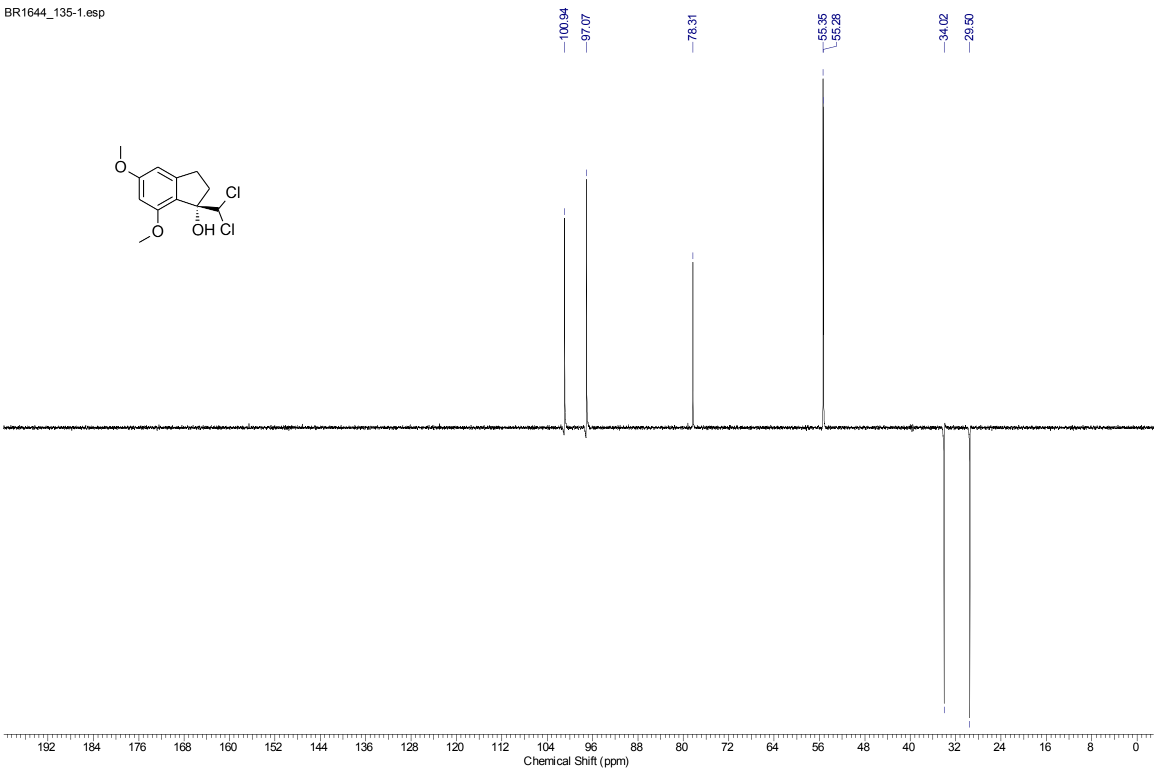


Figure S7a: ^{1}H NMR spectrum of compound 18

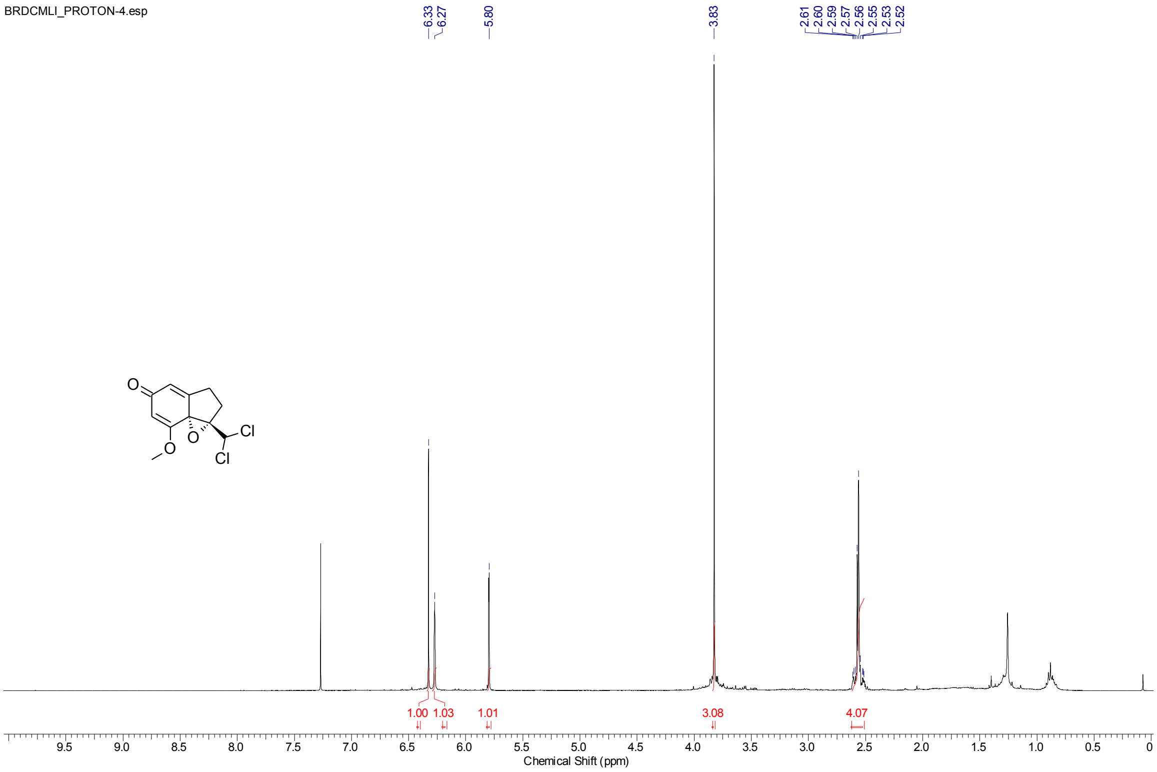


Figure S7b: ^{13}C NMR spectrum of compound 18

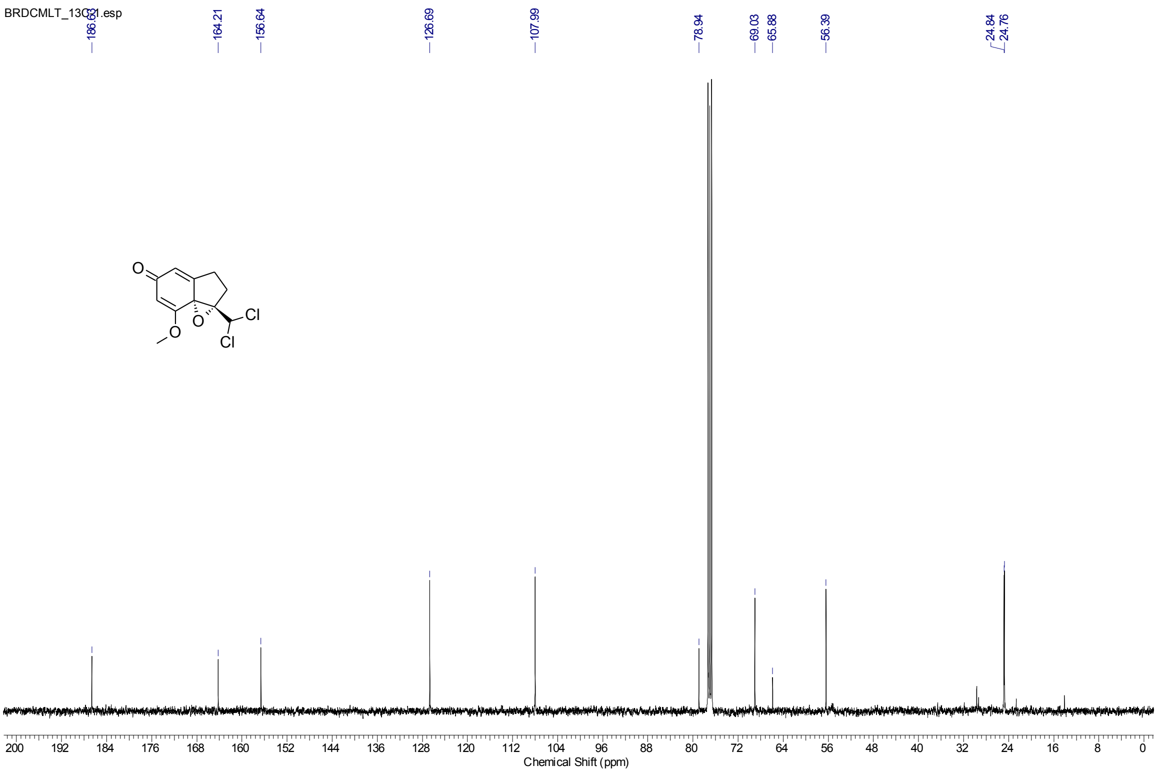


Figure S7c: DEPT spectrum of compound 18

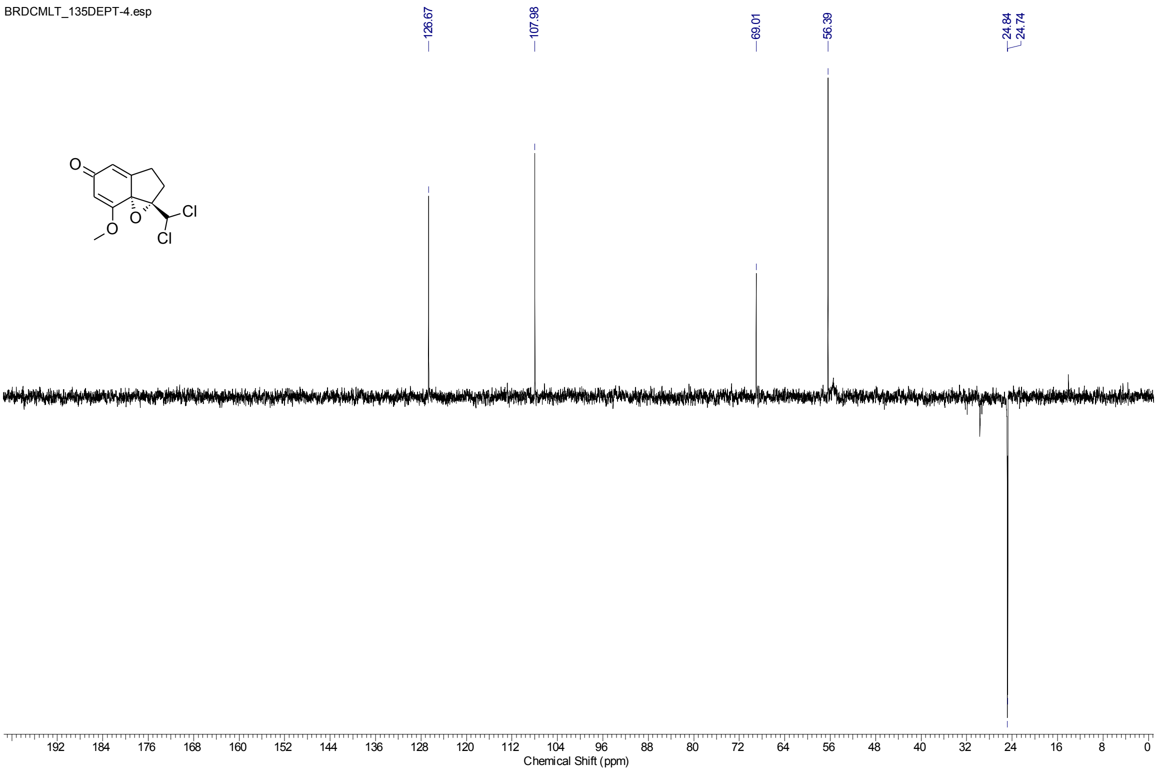


Figure S8a: [1]H NMR spectrum of compound 19

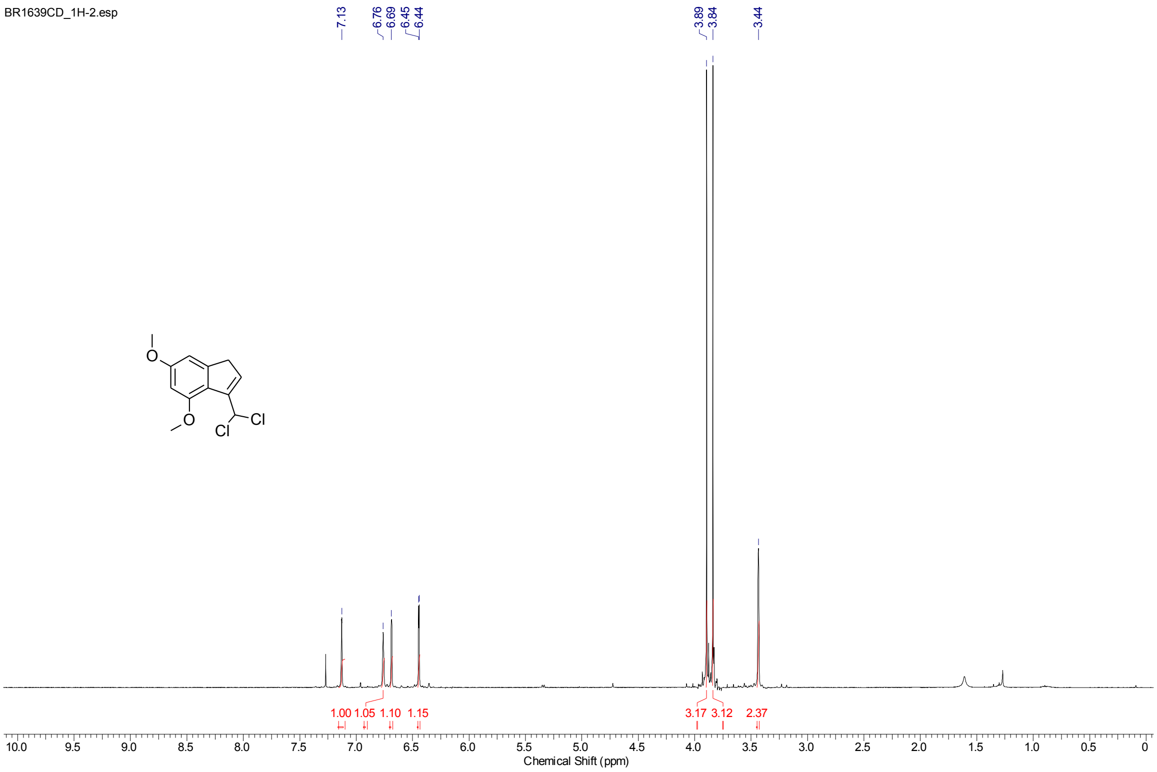


Figure S8b: ^{13}C NMR spectrum of compound 19

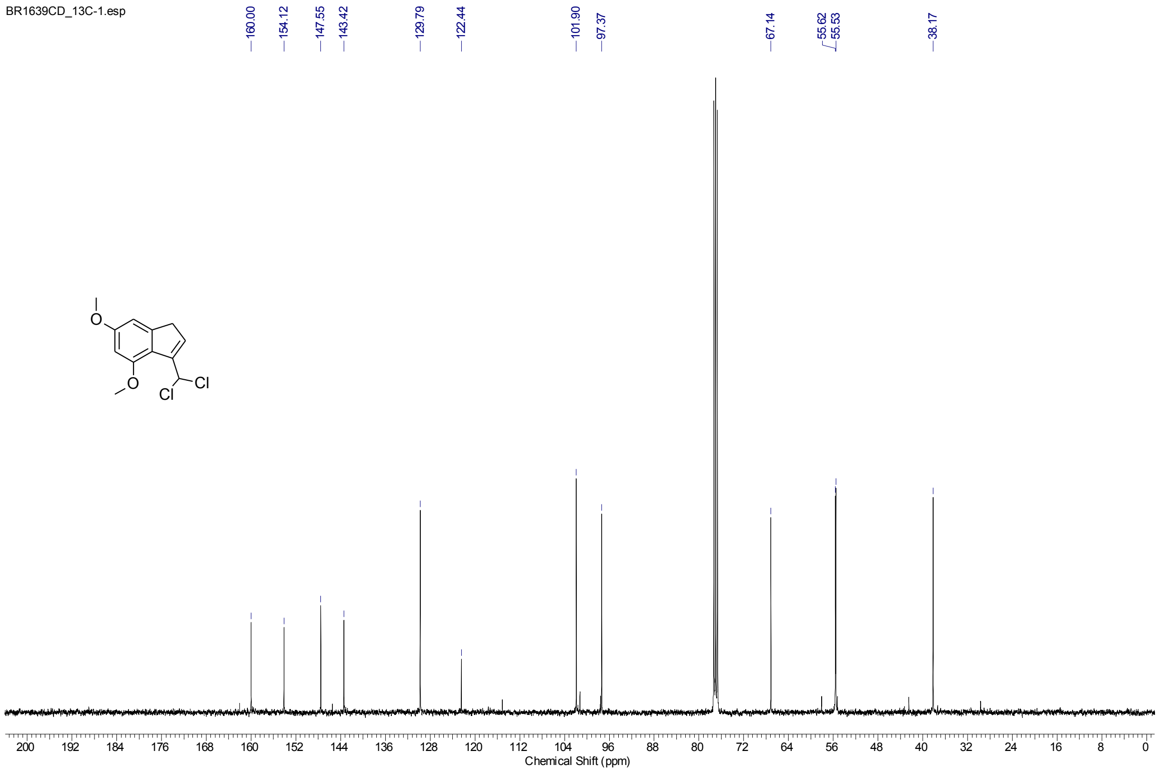


Figure S9a: ^{1}H NMR spectrum of compound 20

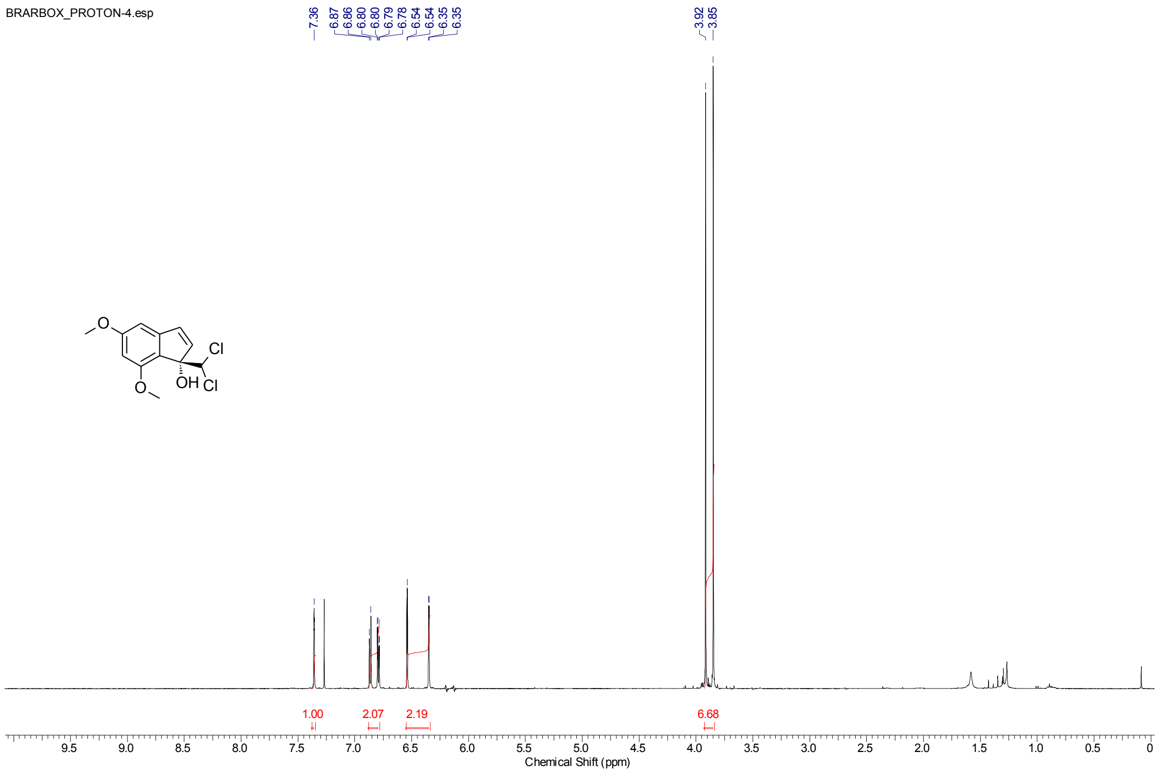


Figure S9b: ^{13}C NMR spectrum of compound 20

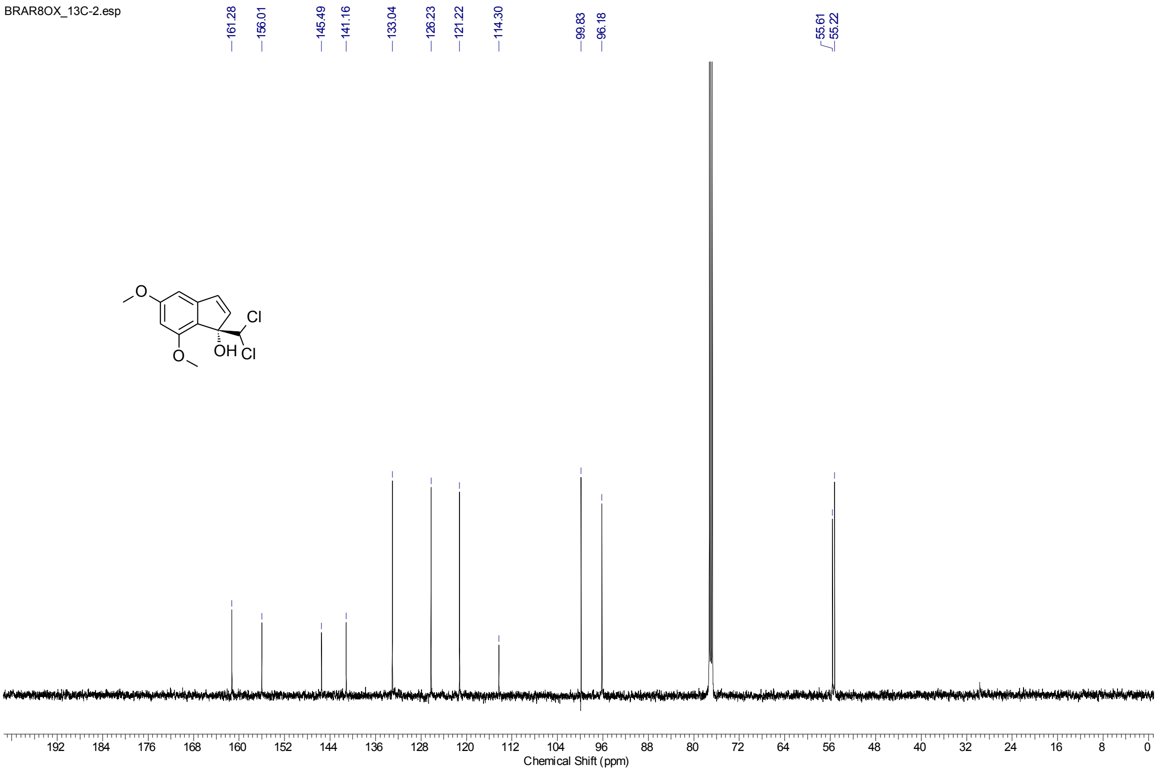


Figure S9c: DEPT spectrum of compound 20

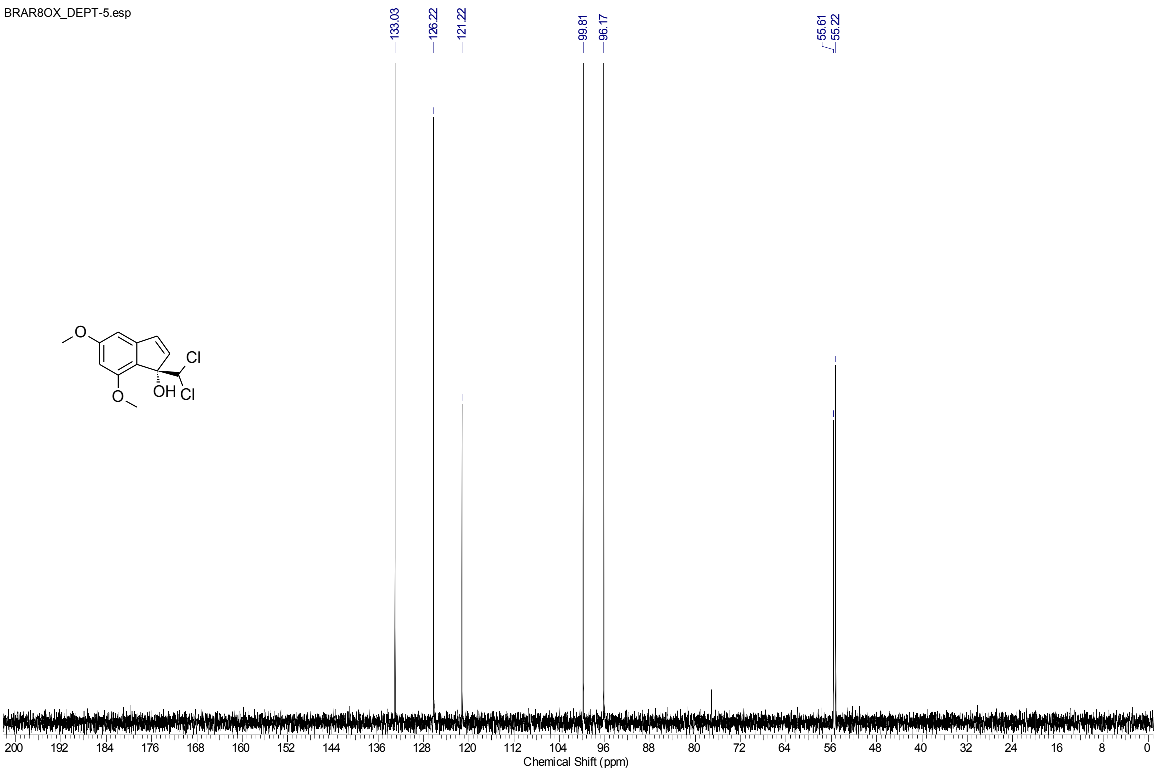


Figure S10a: ^{1}H NMR spectrum of compound 21

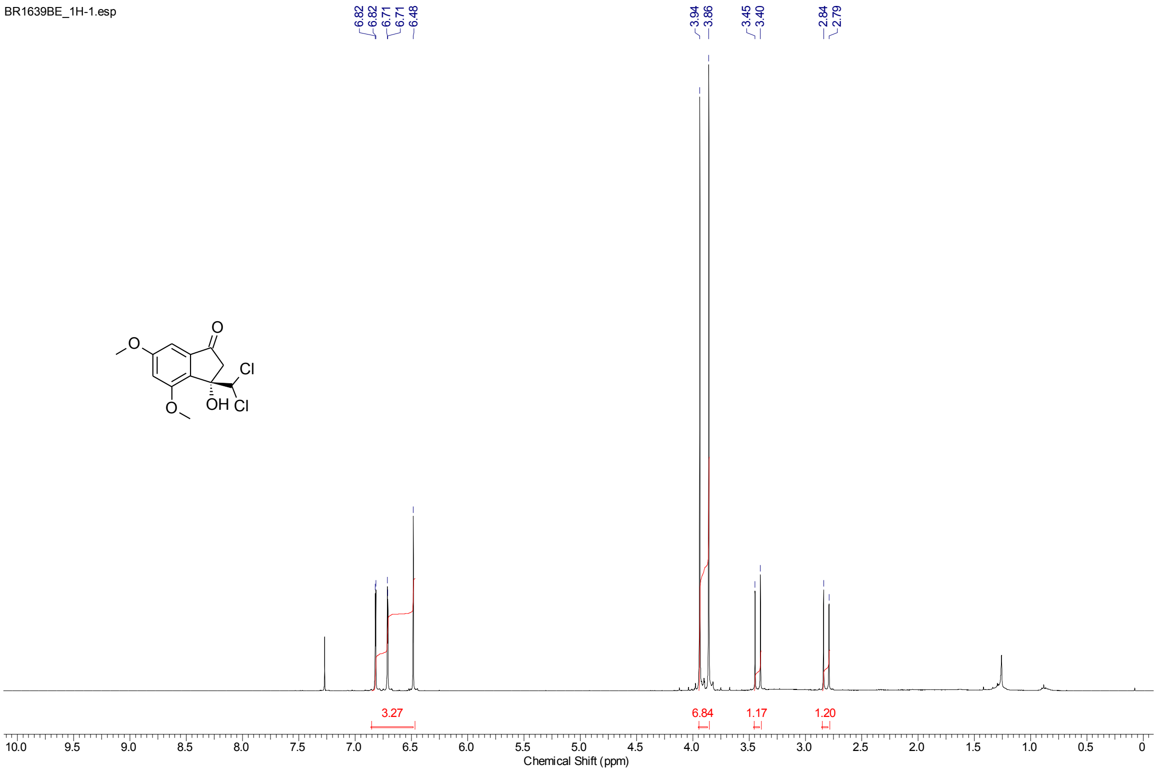


Figure S10b: ^{13}C NMR spectrum of compound 21

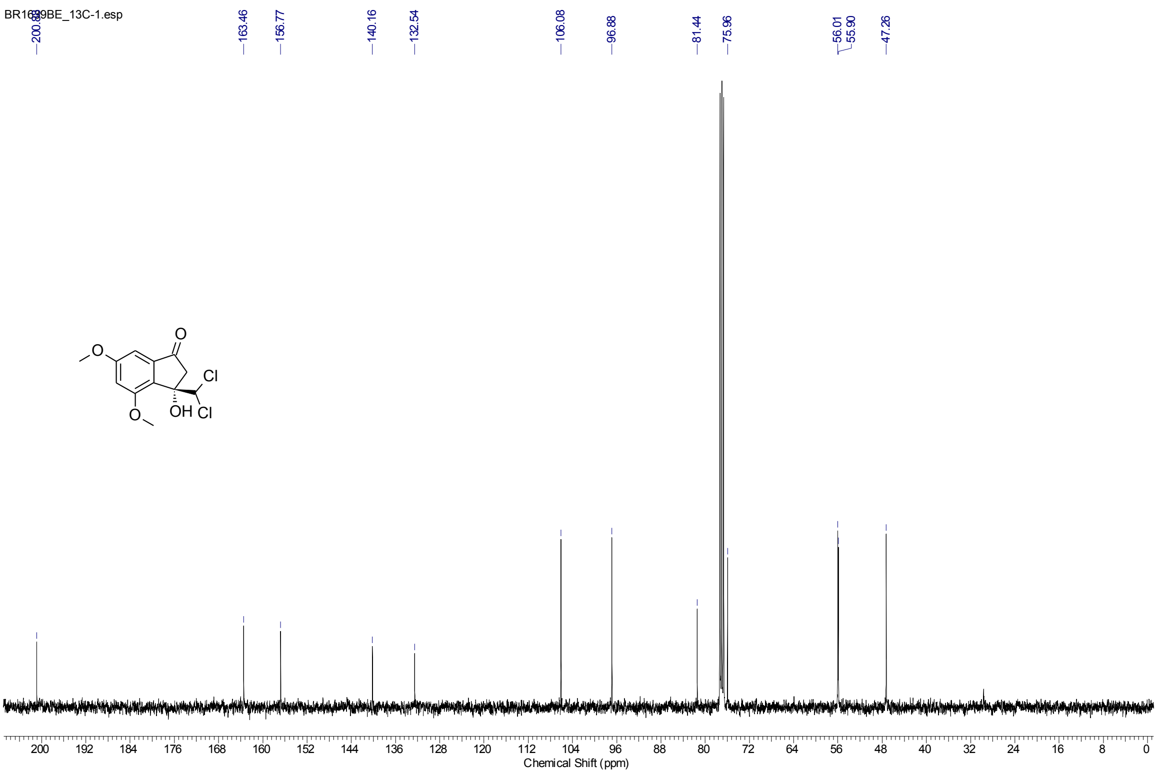


Figure S10c: DEPT spectrum of compound 21

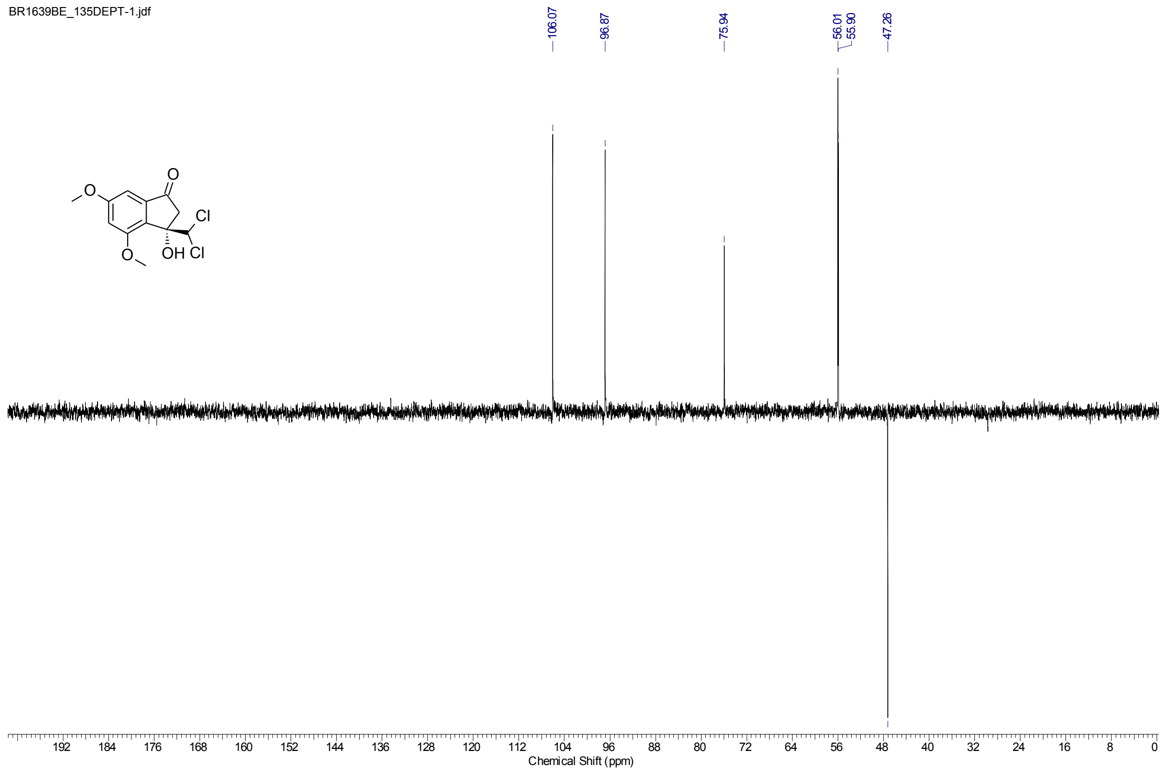


Figure S11a: ^{1}H NMR spectrum of compound 22

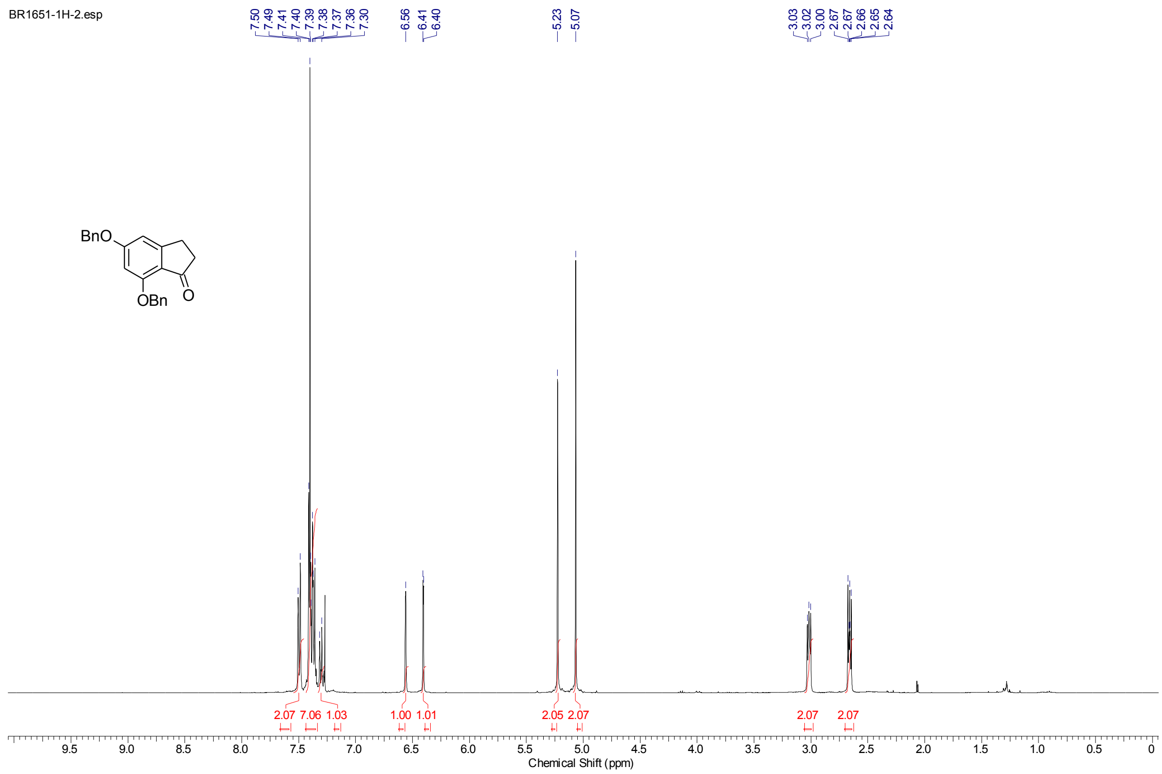


Figure S11b: ^{1}H NMR spectrum of compound 22

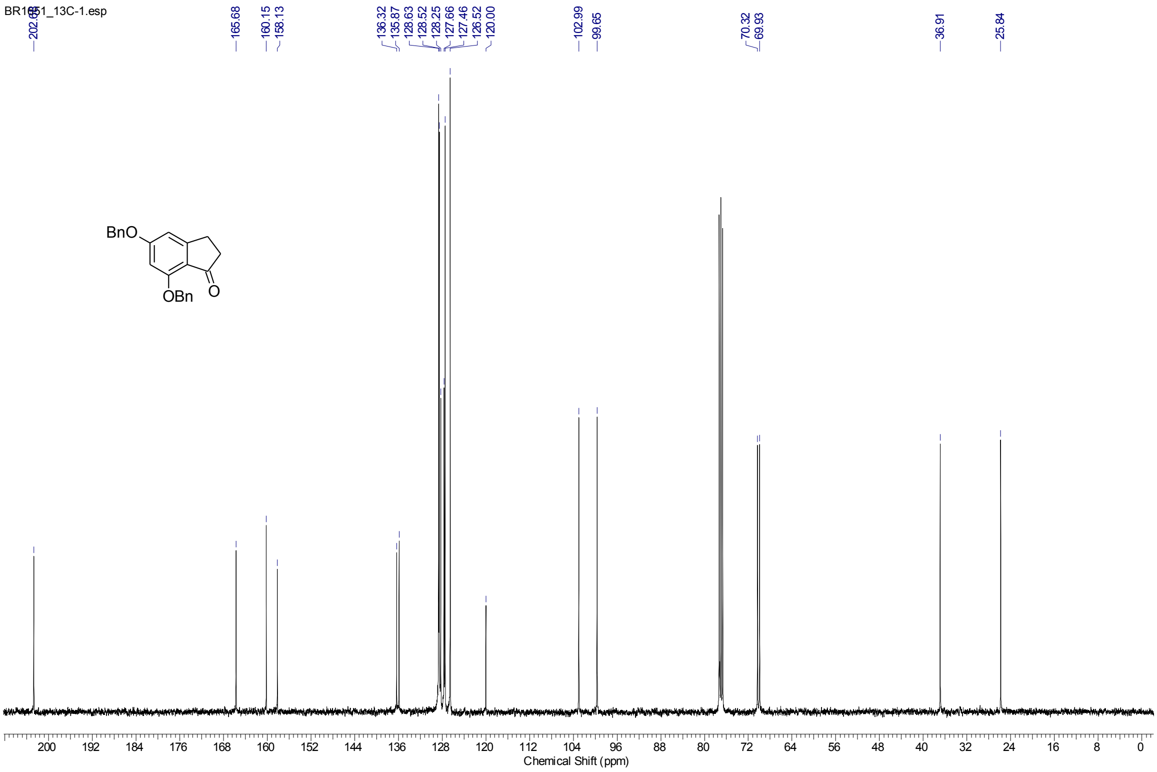


Figure S12a: ^{1}H NMR spectrum of compound 23

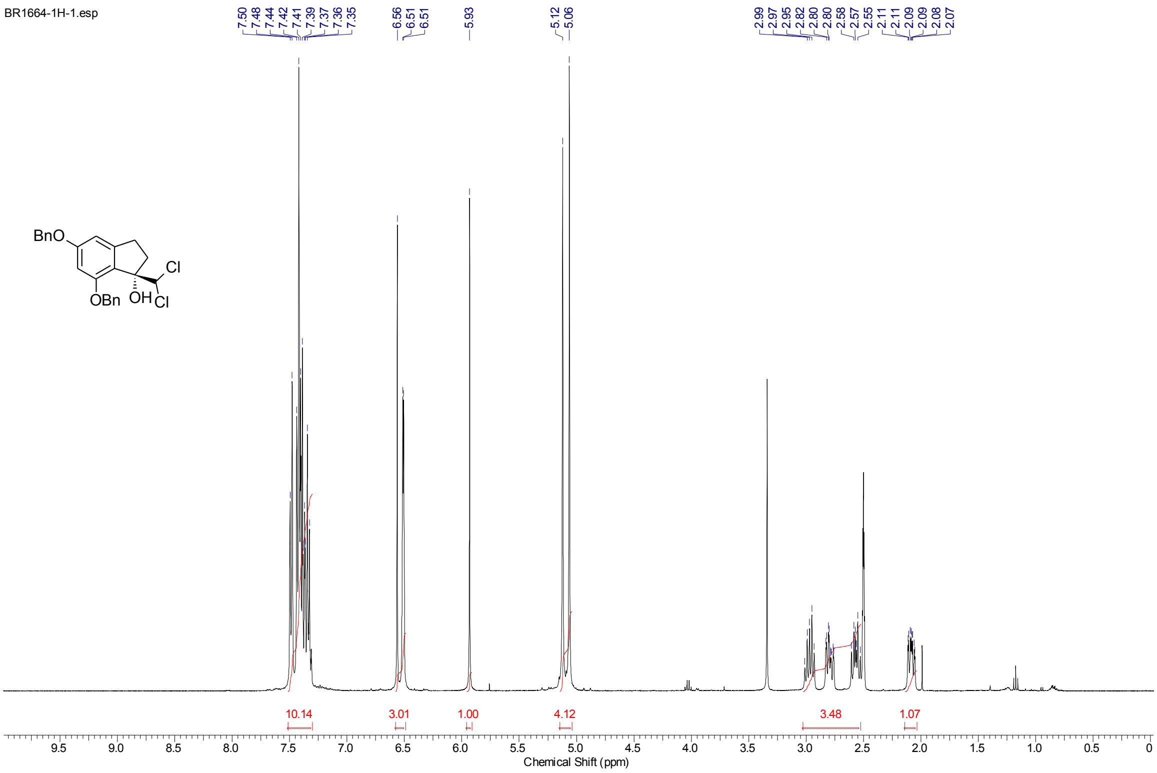


Figure S12b: ^{13}C NMR spectrum of compound 23

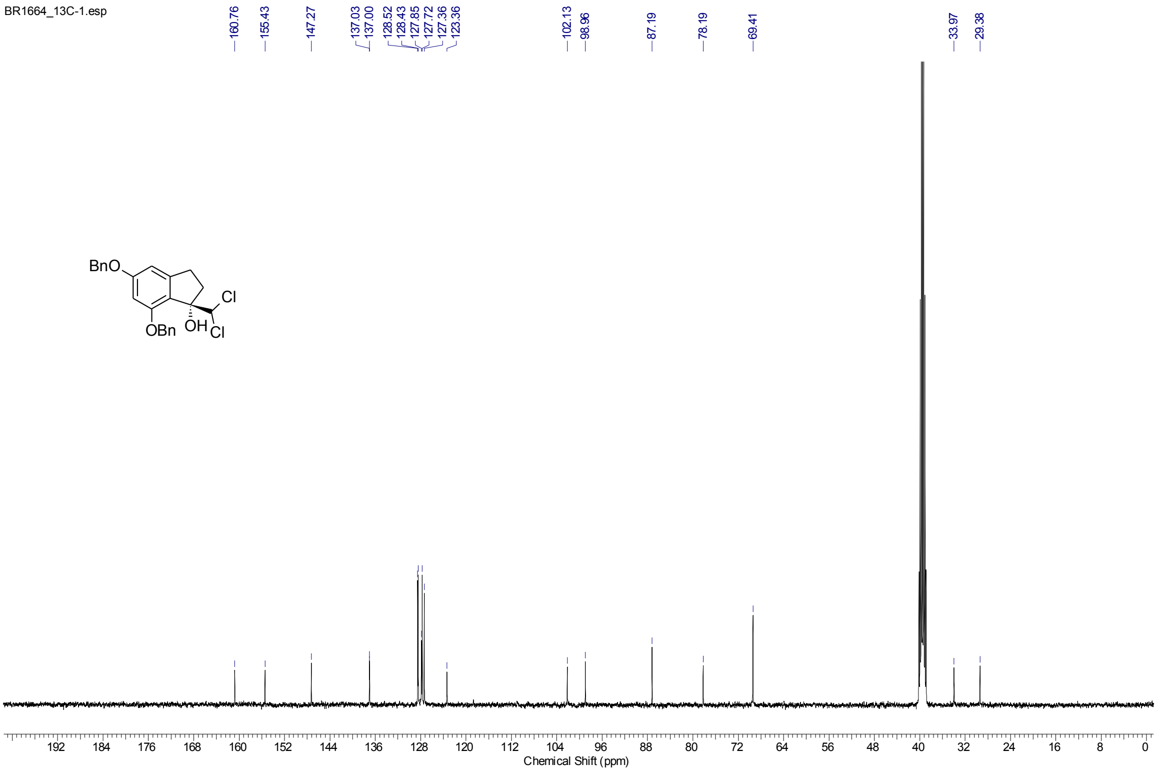


Figure S12c: DEPT spectrum of compound 23

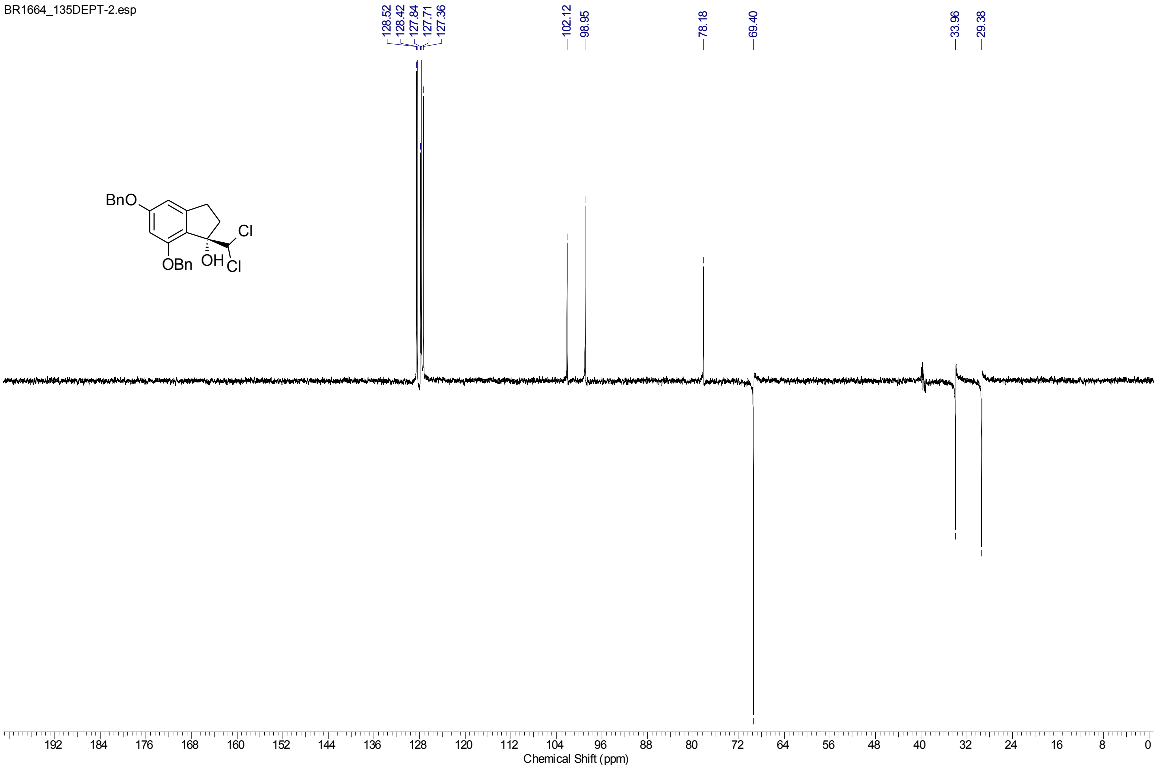


Figure S13a: ^{1}H NMR spectrum of compound 24

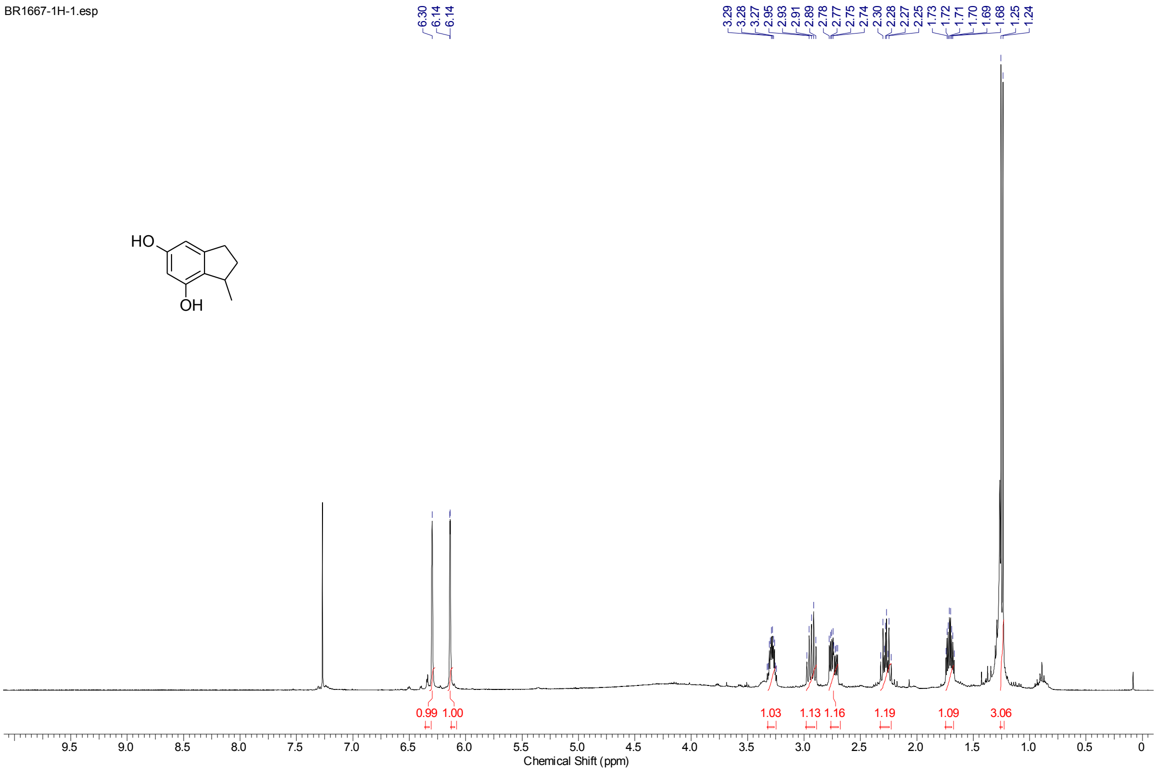


Figure S13b: ^{13}C NMR spectrum of compound 24

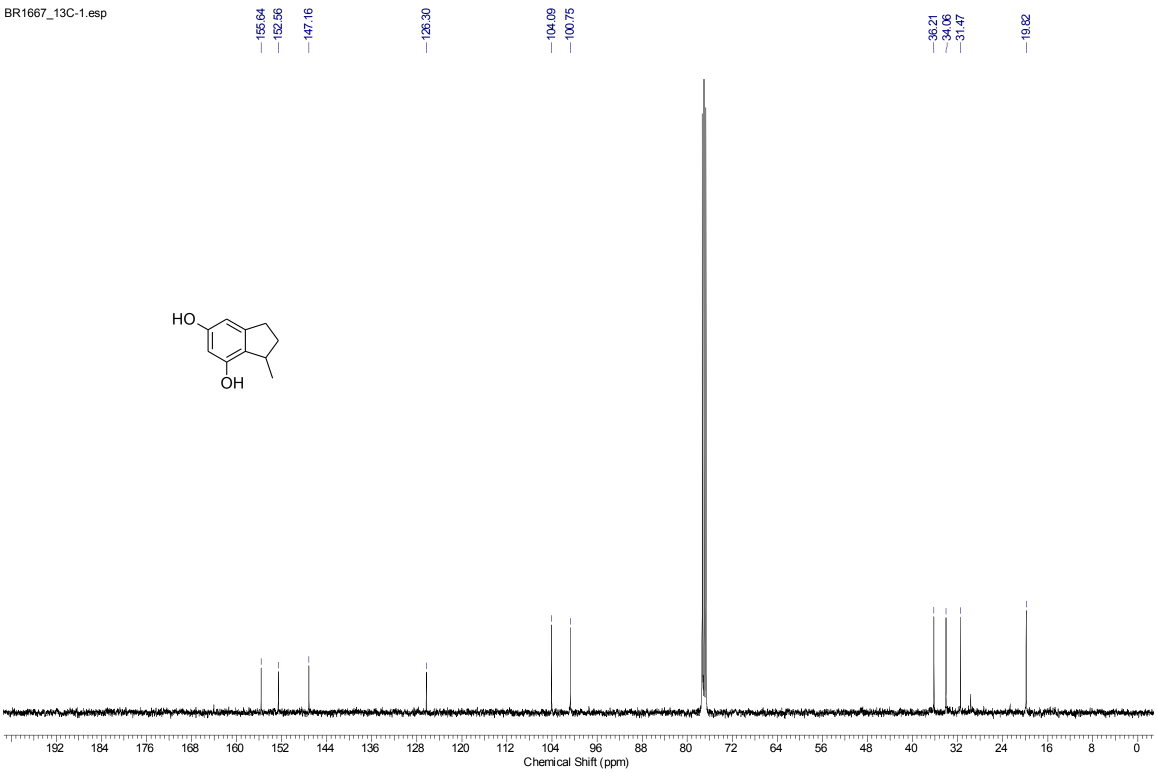


Figure S13c: DEPT spectrum of compound 24

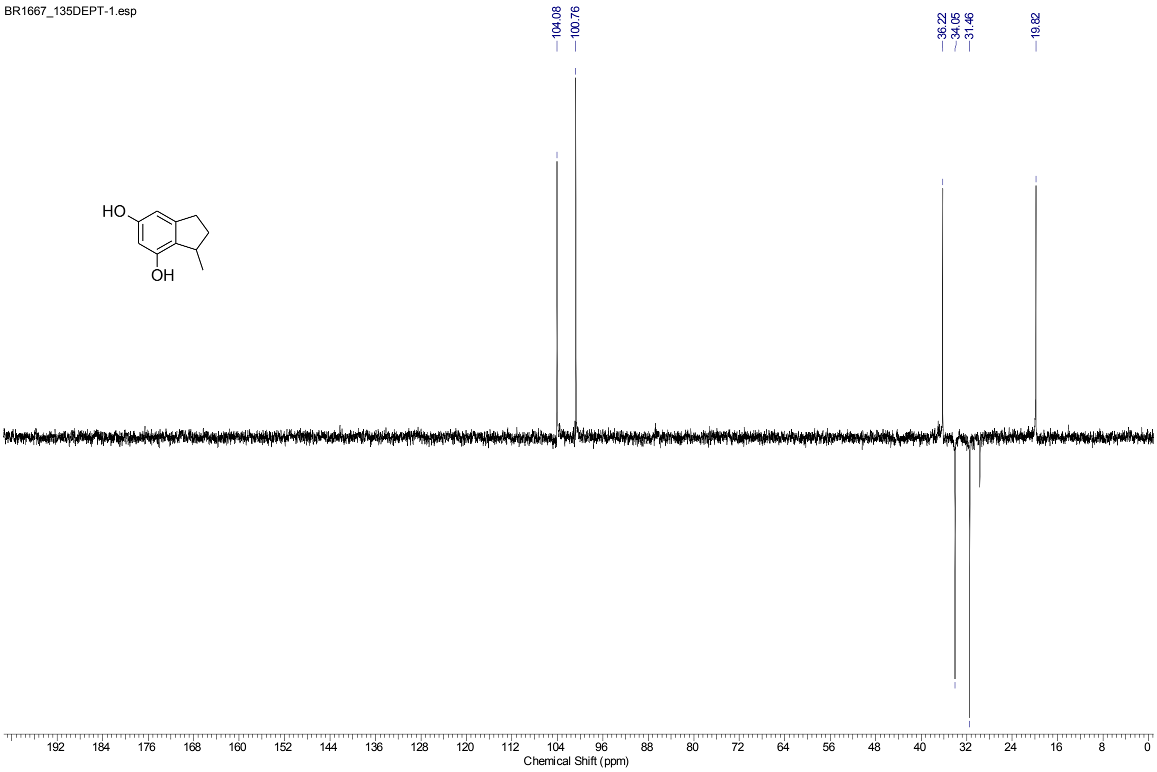


Figure S14a: ^{1}H NMR spectrum of compound 25

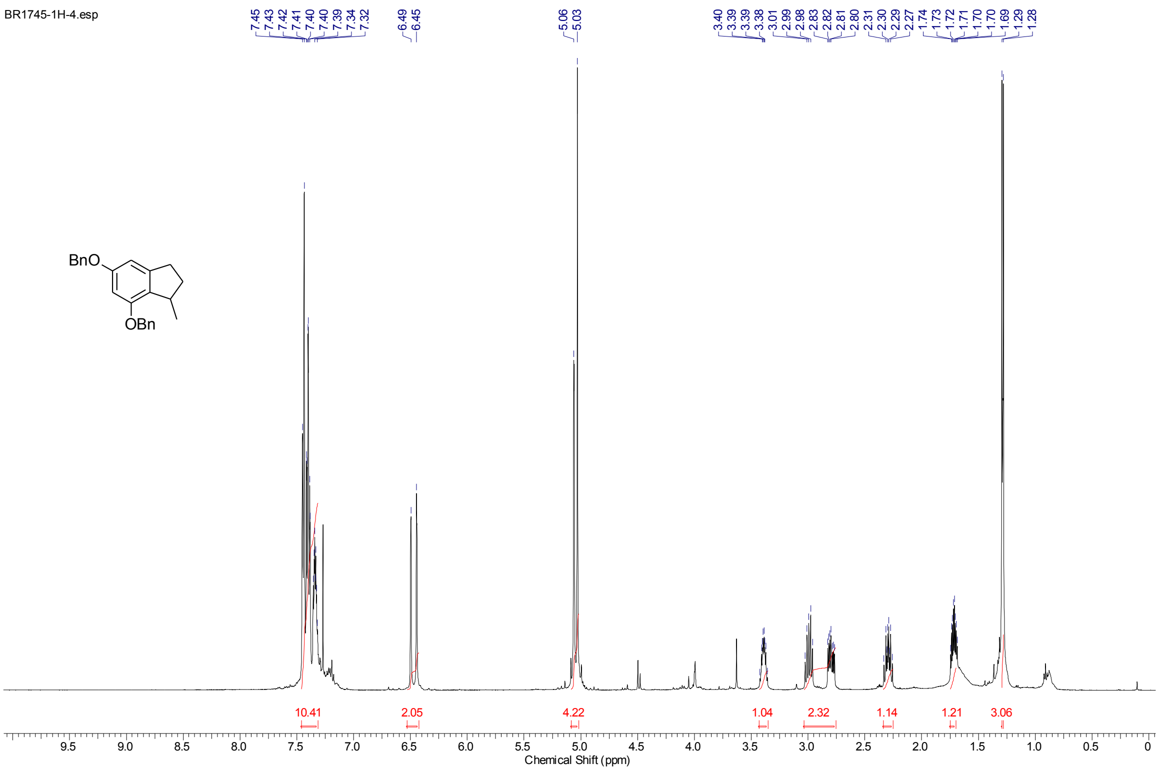


Figure S14b: ^{13}C NMR spectrum of compound 25

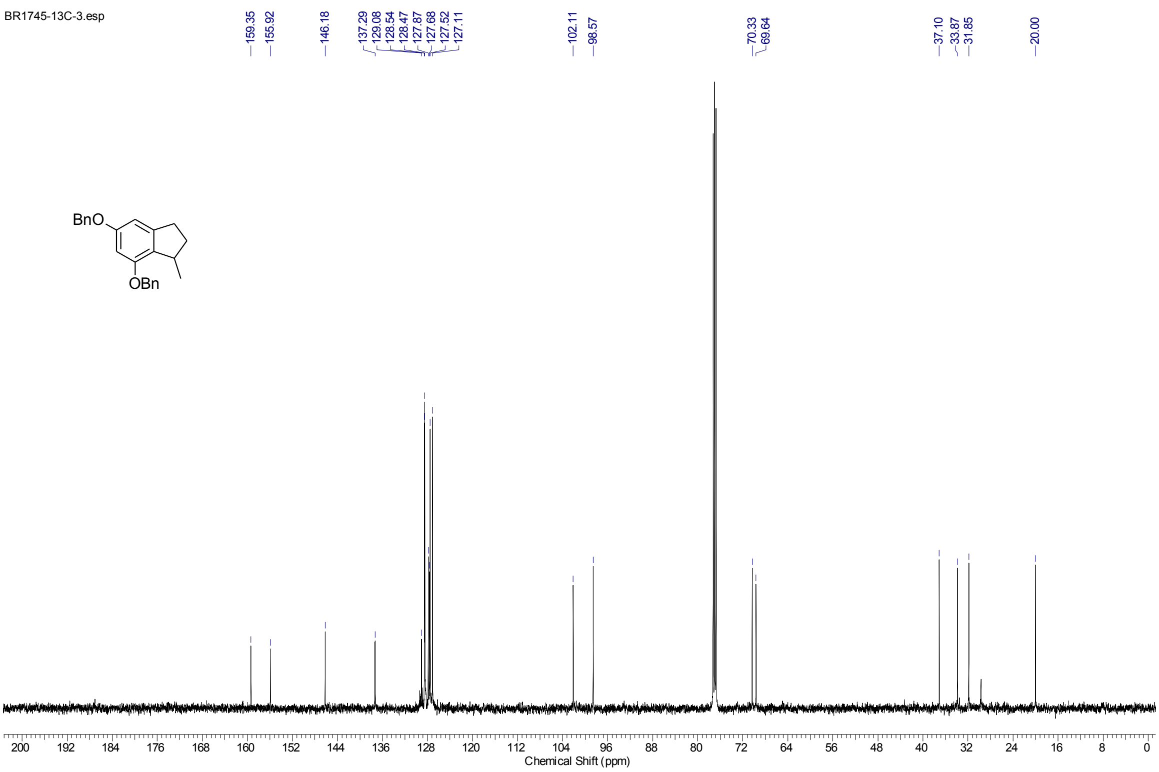


Figure S14c: DEPT spectrum of compound 25

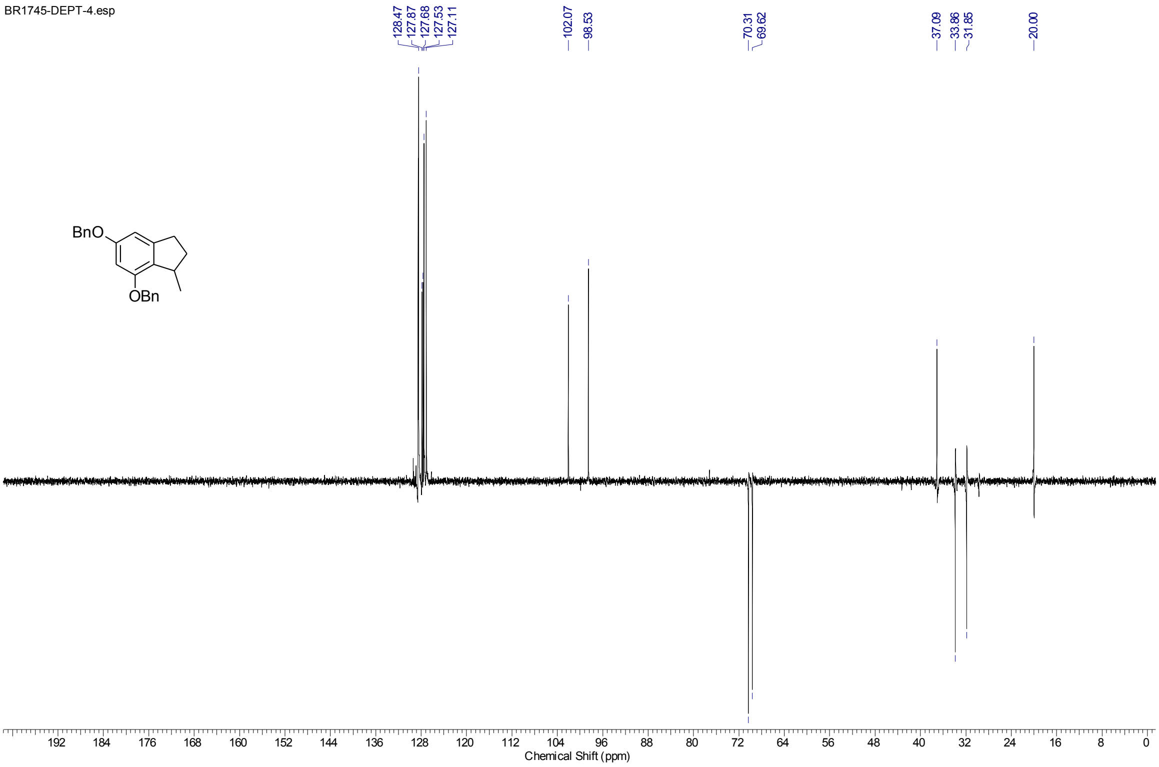


Figure S15a: ^{1}H NMR spectrum of compound 26

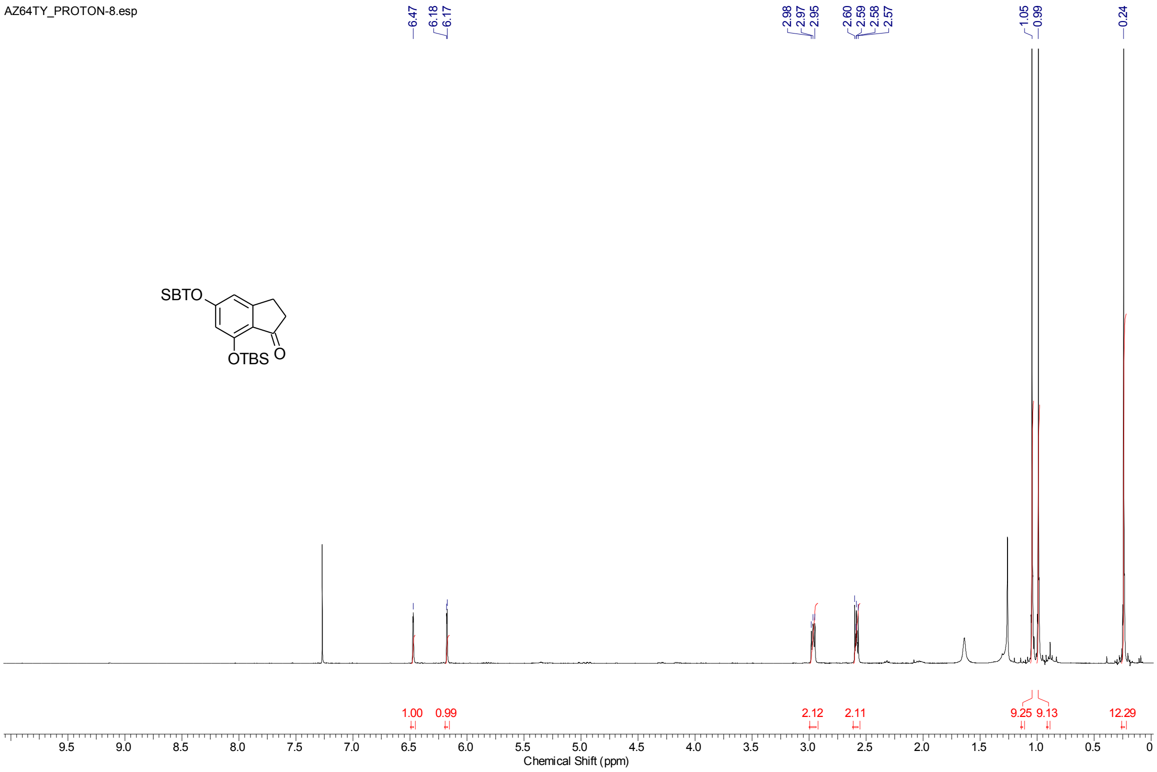


Figure S15b: ^{1}H NMR spectrum of compound 26

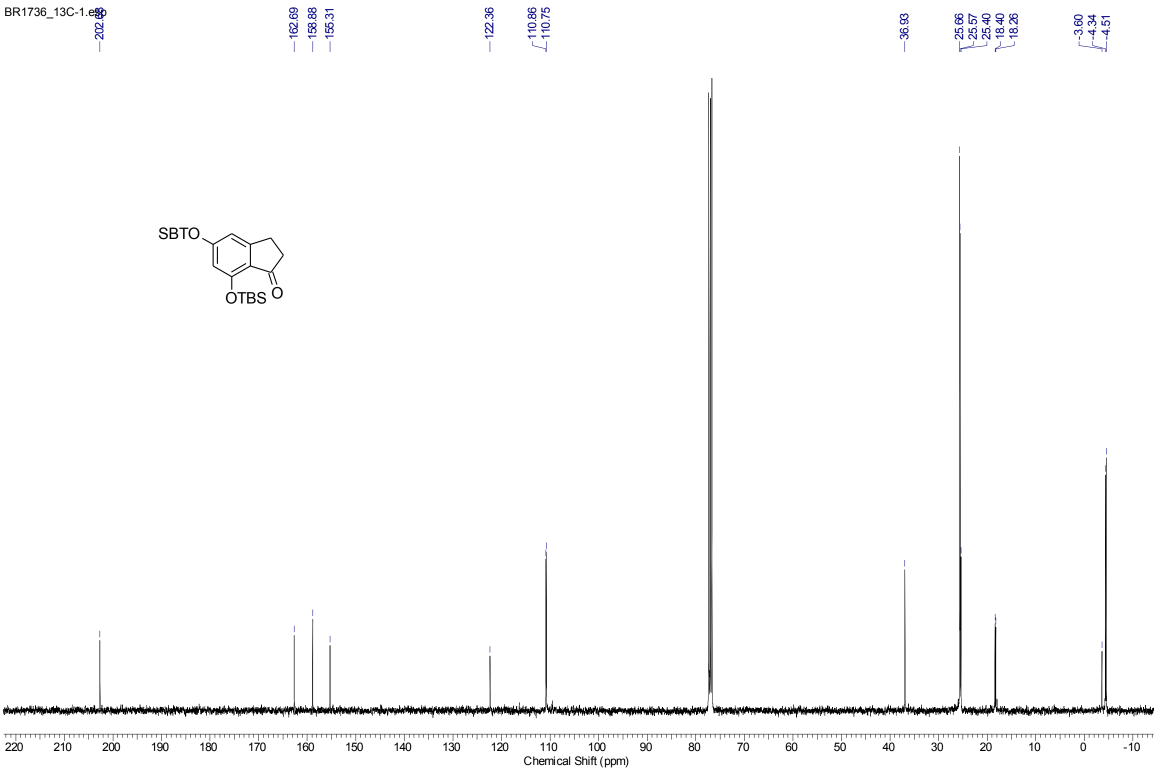


Figure S16a: ^{1}H NMR spectrum of compound 27

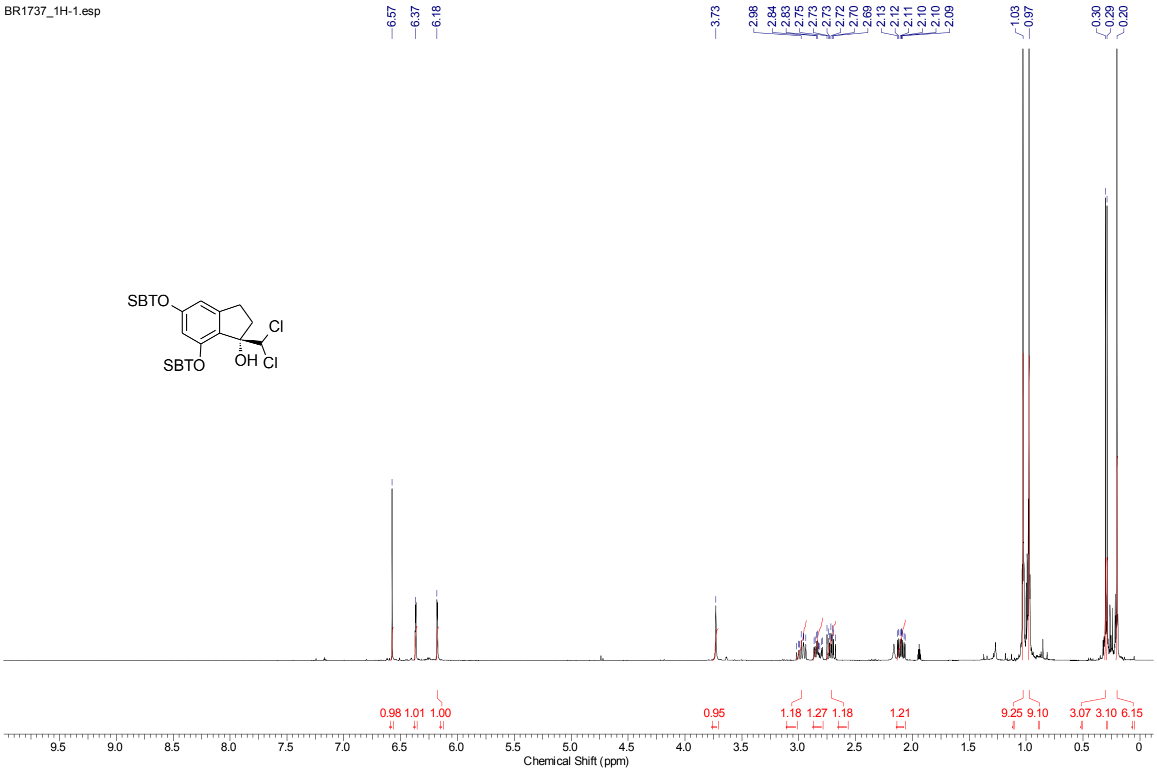


Figure S16b: ^{13}C NMR spectrum of compound 27

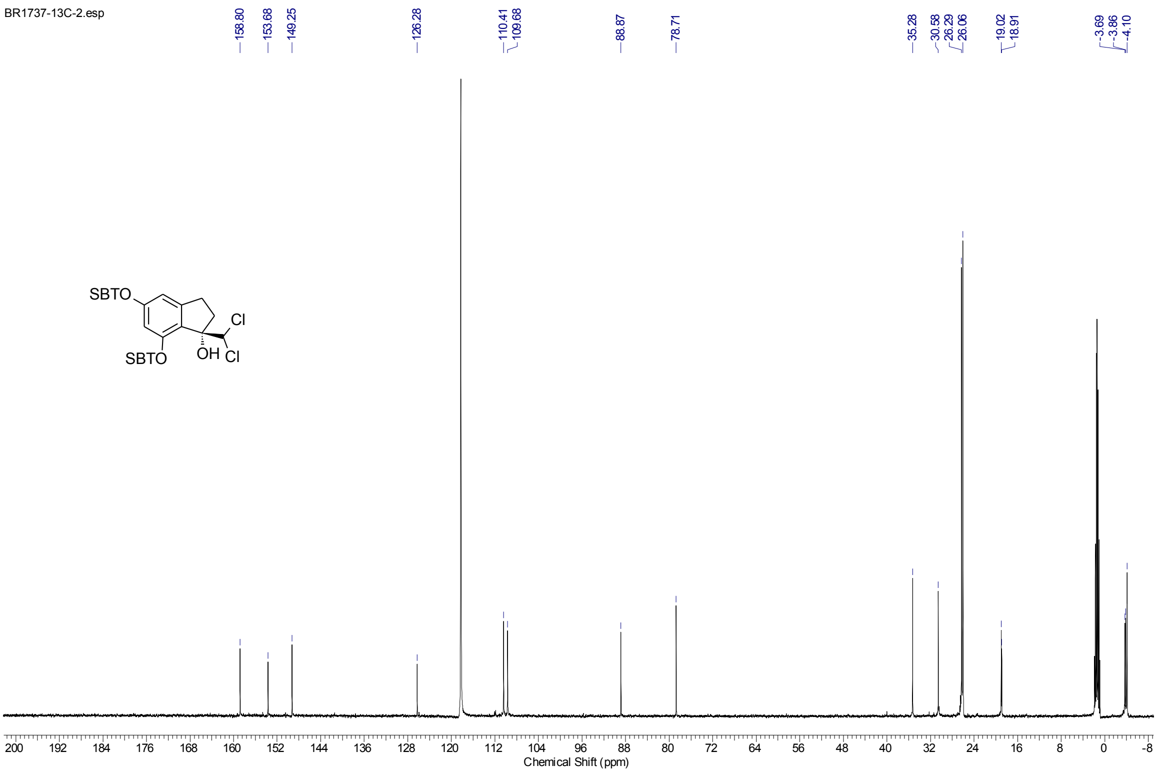


Figure S17a: ^{1}H NMR spectrum of compound 28

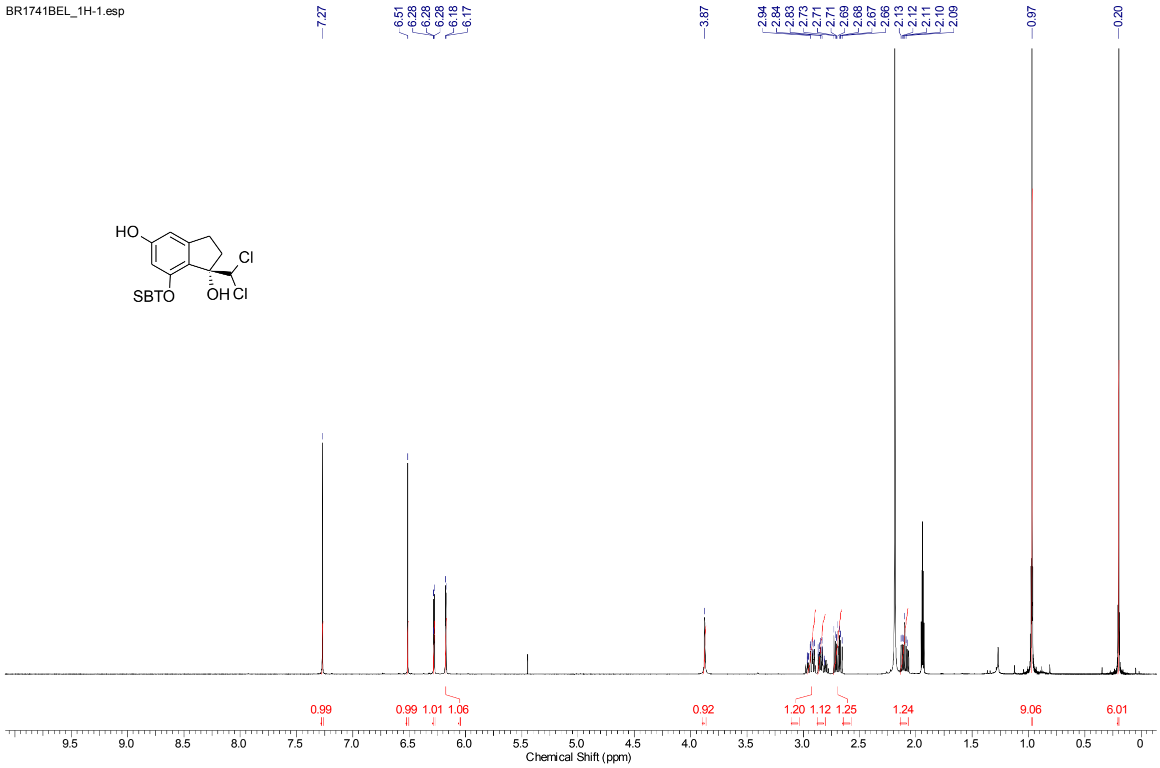


Figure S17b: ^{13}C NMR spectrum of compound 28

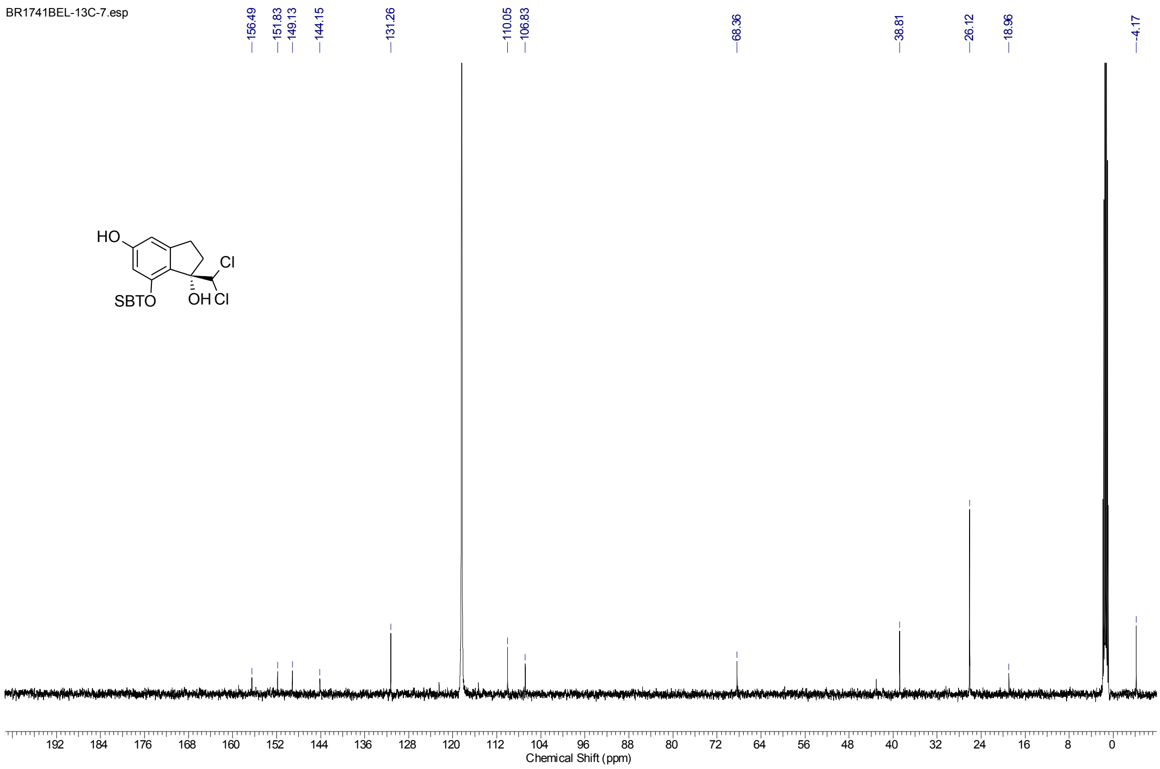


Figure S18a: ^{1}H NMR spectrum of compound 29

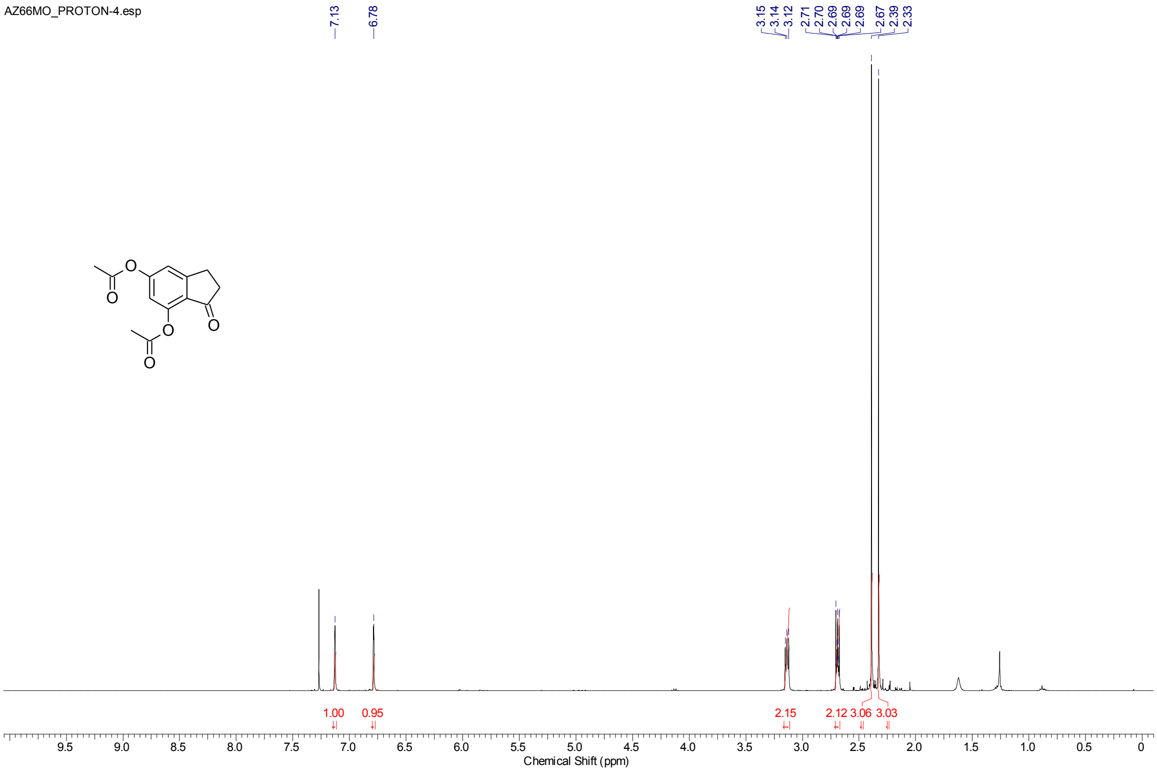


Figure S18b: ^{13}C NMR spectrum of compound 29

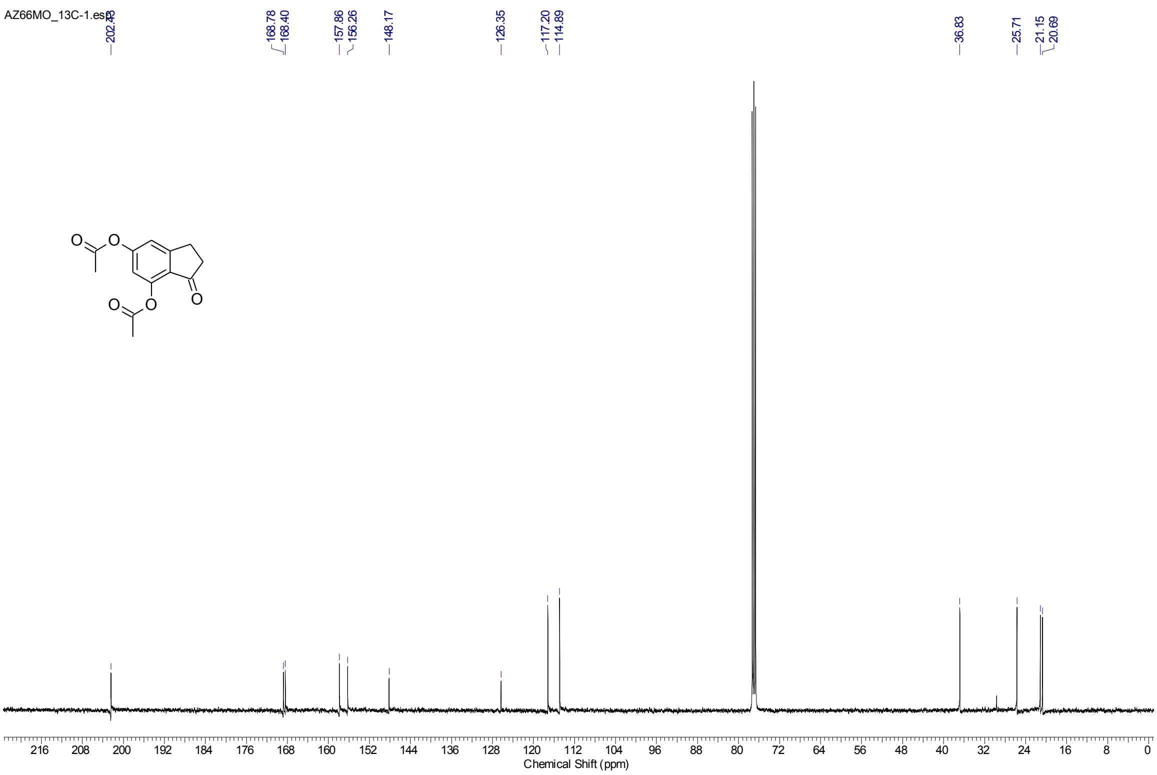


Figure S19a: ^1H NMR spectrum of compound 31

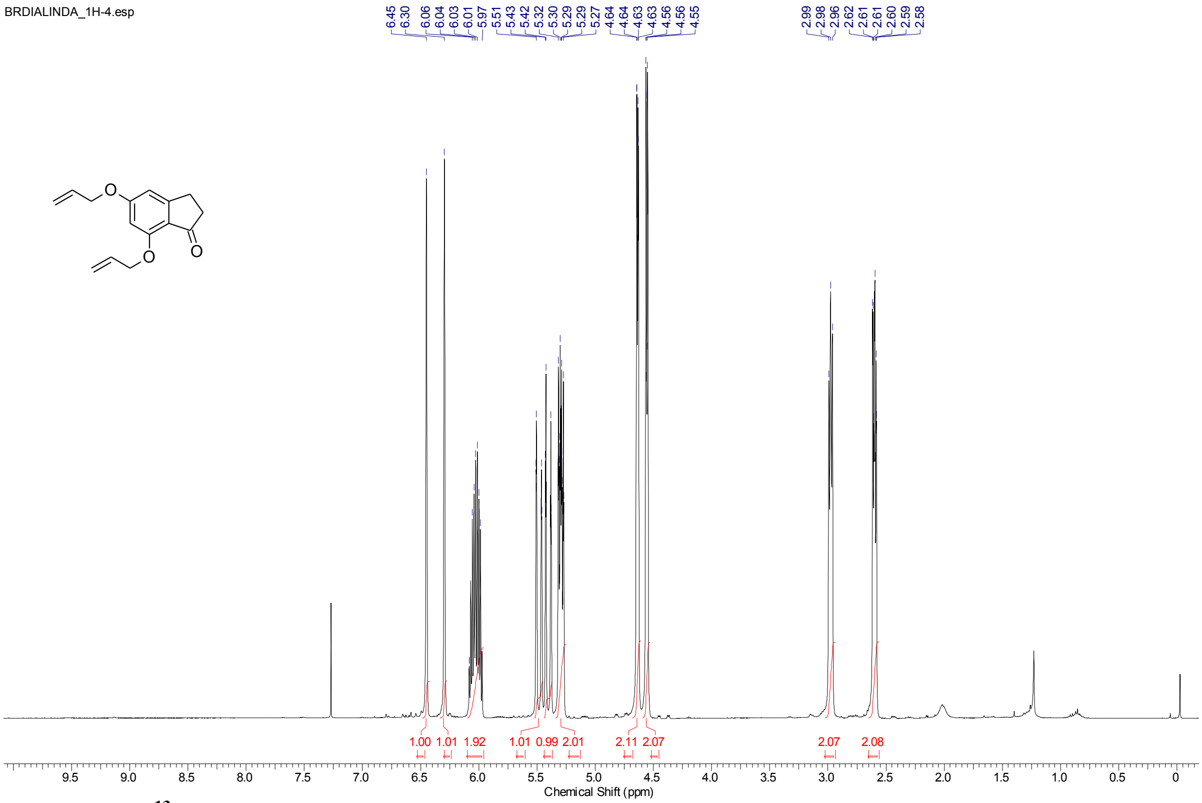


Figure S19b: ^{13}C NMR spectrum of compound 31

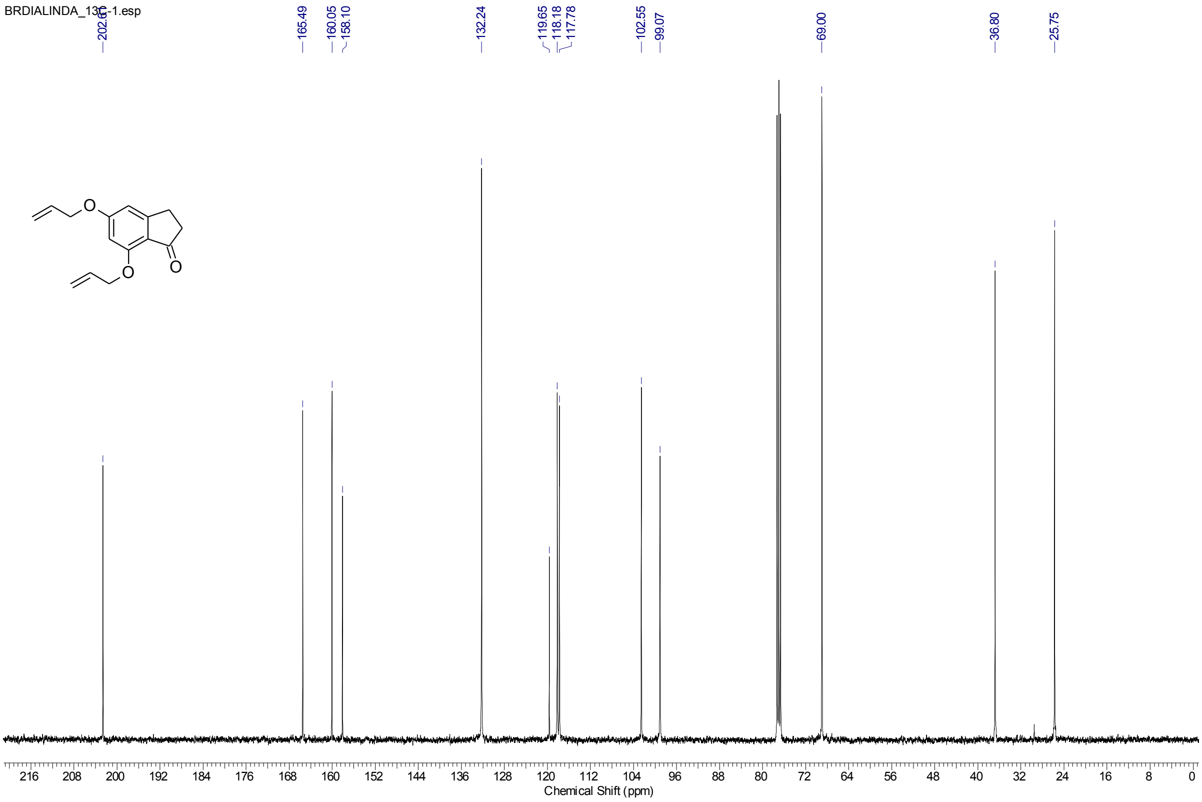


Figure S20a: ^{1}H NMR spectrum of compound 32

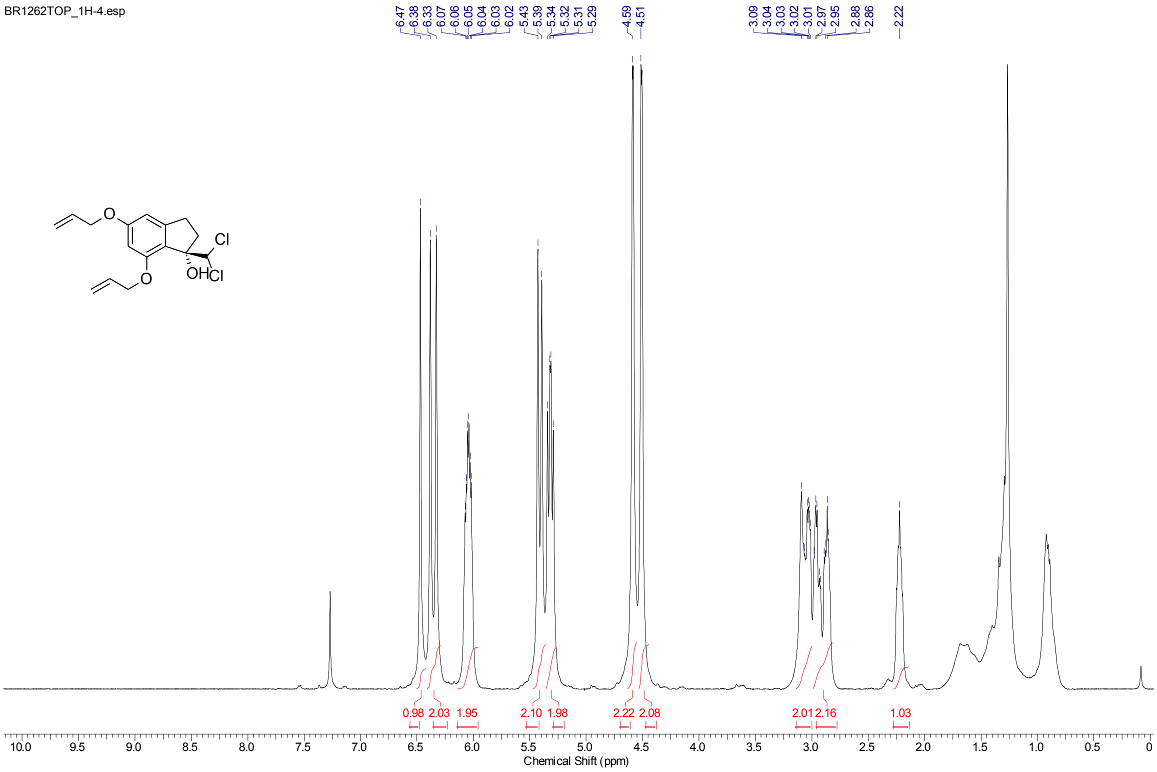
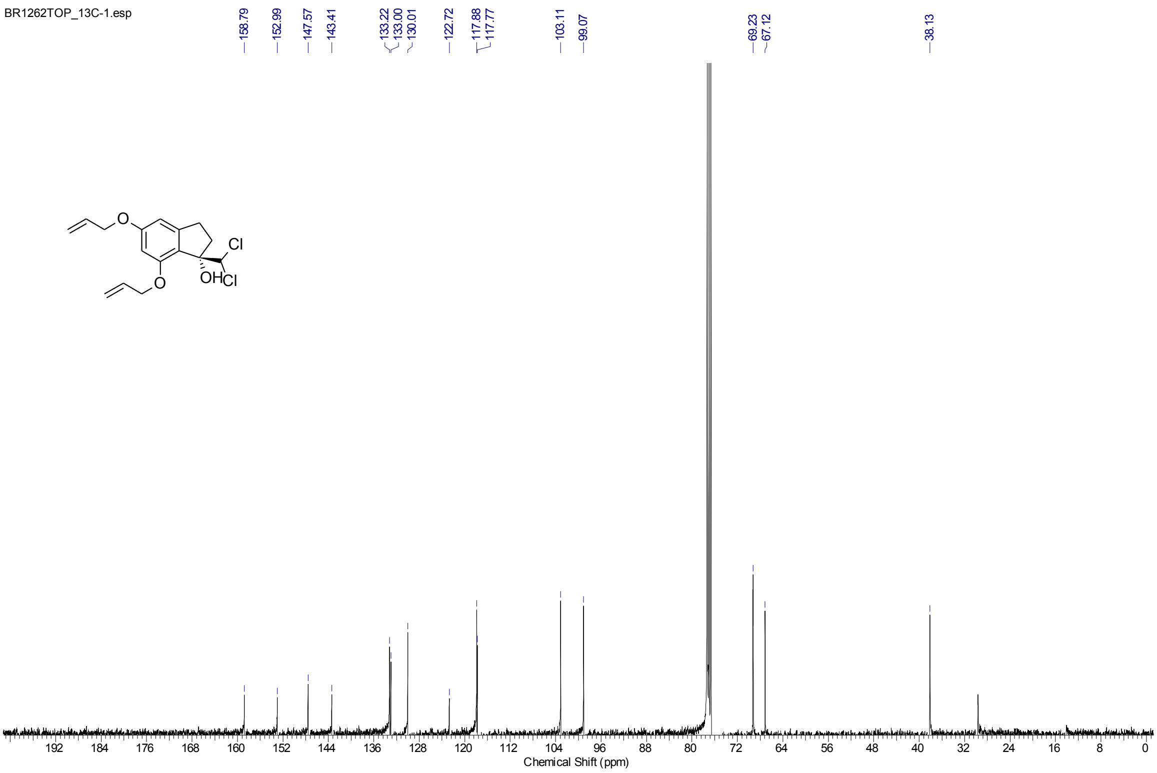
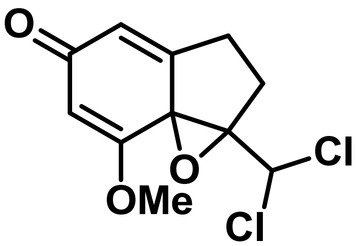


Figure S20a: ^{13}C NMR spectrum of compound 32



Single Crystal X-Ray Structure Determination. The single crystal X-ray diffraction intensity data collection for the crystals of **compound 18** was carried out on a Bruker APEX-II CCD detector system with Mo-sealed Siemens ceramic diffraction tube (λ = 0.7107 Å) and a highly oriented graphite monochromator operating at 50 kV and 30 mA. The data were collected in a hemisphere mode and processed with Bruker SAINT. Empirical absorption correction was made using Bruker SADABS. The structure was solved by using SHELXL package and refined by full matrix least-squares method based on F^2 using SHELX-2017 program (Sheldrick 2017). Hydrogens were fixed geometrically, treated as riding on their nonhydrogens, and refined isotropically, while all nonhydrogens were subjected to anisotropic refinement.

Table 1: X-ray crystallographic data and structure refinement of compound 18



Chemical formula	$C_{11}H_{10}Cl_2O_3$
M_r	261.10
Crystal system, space group	Monoclinic, C 2/c
Temperature (K)	100
a, *b*, *c* (Å)	17.488 (5), 7.707 (2), 16.253 (5)
β (°)	94.034 (3)
V (Å^3)	2185.2 (11)
Z	8
Radiation type	Mo *K*α
μ (mm^{-1})	0.58
Crystal size (mm)	0.12 × 0.11 × 0.10
Data collection	
Diffractometer	Bruker *SMART APEX* diffractometer
Absorption correction	Empirical (using intensity measurements) *SADABS*
T_{min}, T_{max}	0.931, 0.943
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10704, 2711, 2111
R_{int}	0.037
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	

$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.034, 0.087, 1.02
No. of reflections	2711
No. of parameters	145
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.27