Convergent Access to Polycyclic Cyclopentanoids from α,β-Unsaturated Acid Chlorides and Alkynes through a Reductive-Coupling, Nazarov Cyclization Sequence

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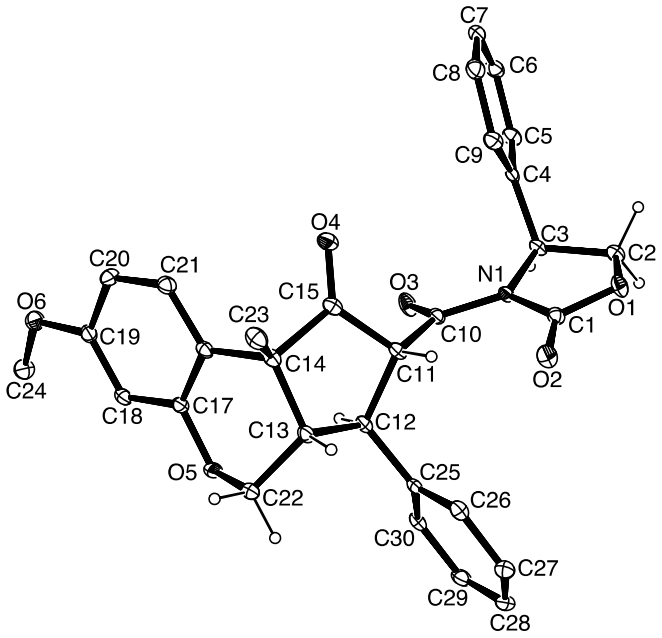
Crystallographic data for 17

Intensity data for **17** were collected at 130 K with a Bruker SMART Apex CCD diffractometer using Mo-Kα radiation (graphite crystal monochromator λ = 0.71073).[1] Data were reduced using the program SAINT. The structure was solved by direct methods and difference Fourier synthesis.[2] The thermal ellipsoid plot was generated using the program ORTEP-3[3] integrated within the WINGX[4] suite of programs.



Thermal ellipsoid plot of **17**, ellipsoids are at the 20% probability level.

Crystal data for **17**. $C_{30}H_{27}NO_6$, M = 497.52, T = 130.02) K, λ = 0.71073, Monoclinic, space group $P2_1$ a = 6.1224(11), b = 12.237(2), c =16.495(3) *Å*, β= 99.471(4)°, V = 1219.0(4)) $Å^3$, Z = 2, D_c = 1.356 mg M^{-3} μ(Mo-Kα) 0.095 mm^{-1}, F(000) = 524, crystal size 0.50 x 0.35 x 0.20 mm^3, 6410 reflections measured, 4026 independent reflections (R_{int} = 0.0481), the final R was 0.0385 [I > 2σ(I)] and wR(F^2) was 0.0976 (all data)

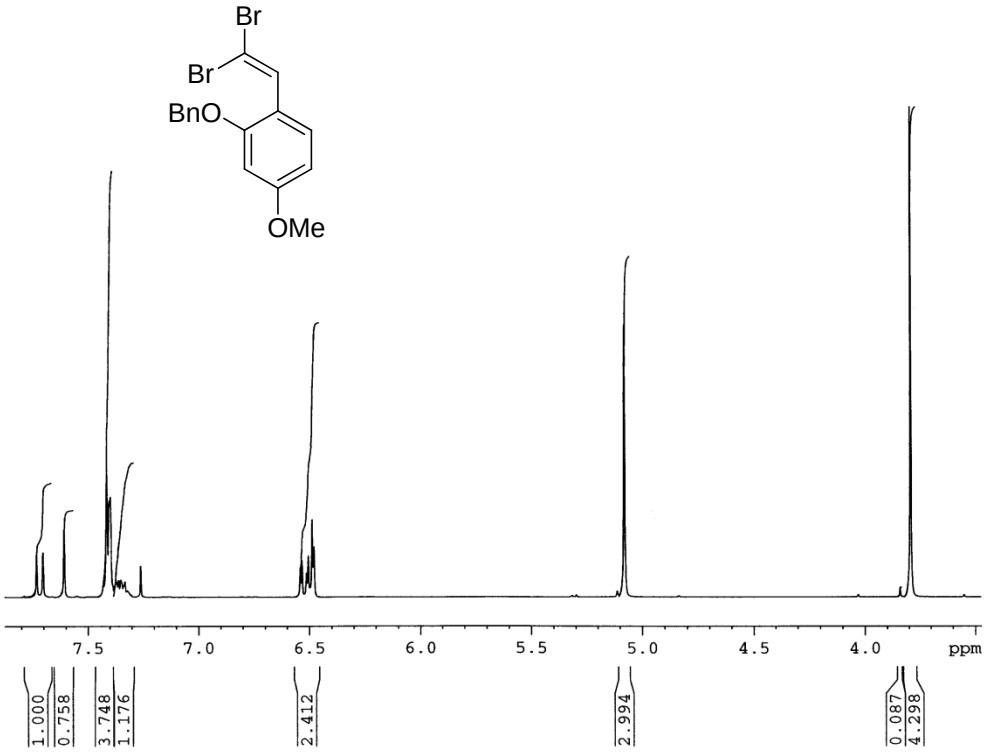
Table 1. Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters ($Å^2$ × 10^3) for **17**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	3933(5)	391(3)	533(2)	26(1)
C(2)	985(5)	-161(3)	-418(2)	28(1)
C(3)	128(5)	777(3)	61(2)	25(1)
C(4)	-520(5)	1764(3)	-468(2)	24(1)
C(5)	-2731(5)	1890(3)	-825(2)	31(1)
C(6)	-3390(6)	2729(3)	-1373(2)	35(1)
C(7)	-1857(6)	3451(3)	-1577(2)	35(1)
C(8)	375(6)	3345(3)	-1221(2)	36(1)
C(9)	1016(5)	2507(3)	-669(2)	31(1)
C(10)	1952(5)	1619(3)	1385(2)	24(1)
C(11)	4053(5)	2027(3)	1905(2)	25(1)
C(12)	4290(5)	1764(3)	2834(2)	24(1)
C(13)	6066(5)	2598(3)	3206(2)	26(1)
C(14)	5526(5)	3663(3)	2720(2)	26(1)
C(15)	4164(5)	3263(3)	1896(2)	25(1)
C(16)	4144(5)	4430(3)	3154(2)	25(1)
C(17)	3521(5)	4164(3)	3910(2)	25(1)
C(18)	2206(5)	4851(3)	4301(2)	27(1)
C(19)	1480(5)	5830(3)	3936(2)	27(1)
C(20)	2086(6)	6122(3)	3184(2)	31(1)
C(21)	3380(5)	5431(3)	2813(2)	30(1)
C(22)	6173(6)	2756(3)	4125(2)	31(1)
C(23)	7629(6)	4227(3)	2522(2)	35(1)
C(24)	-870(6)	6189(3)	4928(2)	37(1)
C(25)	4853(5)	596(3)	3086(2)	25(1)
C(26)	6772(5)	95(3)	2920(2)	31(1)
C(27)	7279(6)	-965(3)	3173(2)	36(1)
C(28)	5868(6)	-1553(3)	3592(2)	38(1)
C(29)	3962(6)	-1062(3)	3756(2)	36(1)
C(30)	3456(5)	-3(3)	3509(2)	29(1)
N(1)	2099(4)	974(2)	696(1)	22(1)
O(1)	3365(3)	-184(2)	-161(1)	32(1)
O(2)	5766(3)	375(2)	925(1)	34(1)
O(3)	140(4)	1889(2)	1527(1)	37(1)
O(4)	3363(4)	3846(2)	1338(1)	36(1)
O(5)	4147(4)	3196(2)	4310(1)	30(1)
O(6)	159(4)	6561(2)	4260(1)	35(1)

[1] Siemens 1999, SMART, SAINT, SADABS, Siemens Analytical X-ray Instruments Inc. Madison, Wisconsin, USA
[2] Sheldrick, G.M., *Acta Cryst.* **2008**, *A64*, 112.
[3] Farrugia, L. J.; *J. Appl. Cryst.* **1997**, *30*, 565.
[4] Farrugia, L. J.; *J. Appl. Cryst.* **1999**, *32*, 837.

^{1}H and ^{13}C NMR SPECTRA

^{1}H NMR ($CDCl_3$) 300 MHz Compound 9

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F1 - Processing parameters
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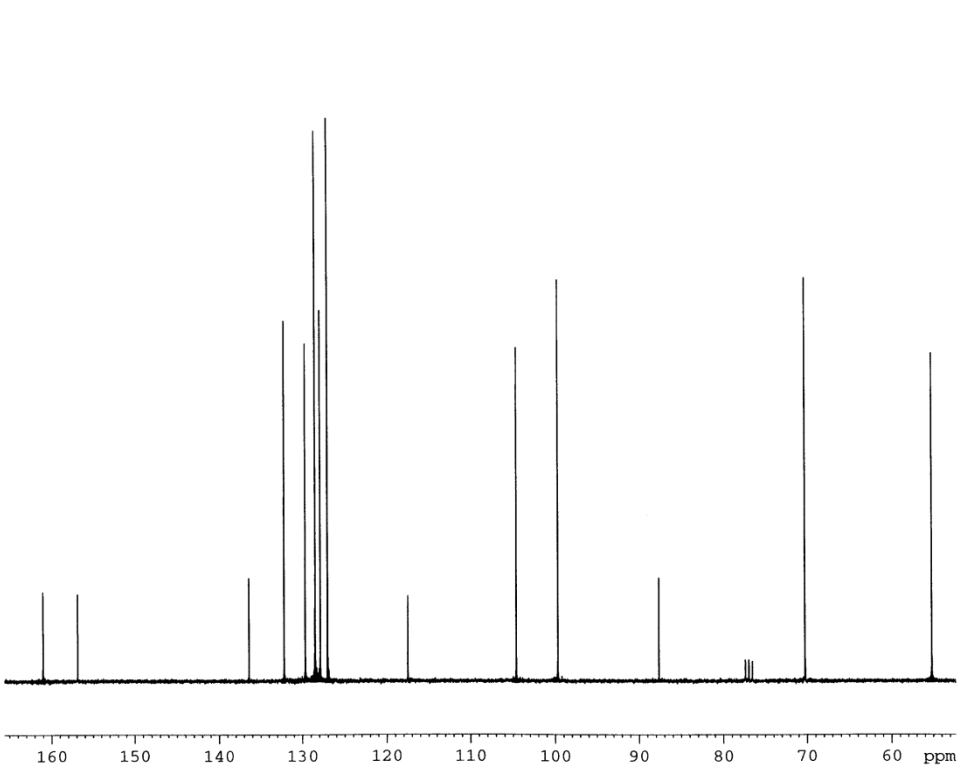
^{13}C NMR ($CDCl_3$) 75 MHz Compound 9

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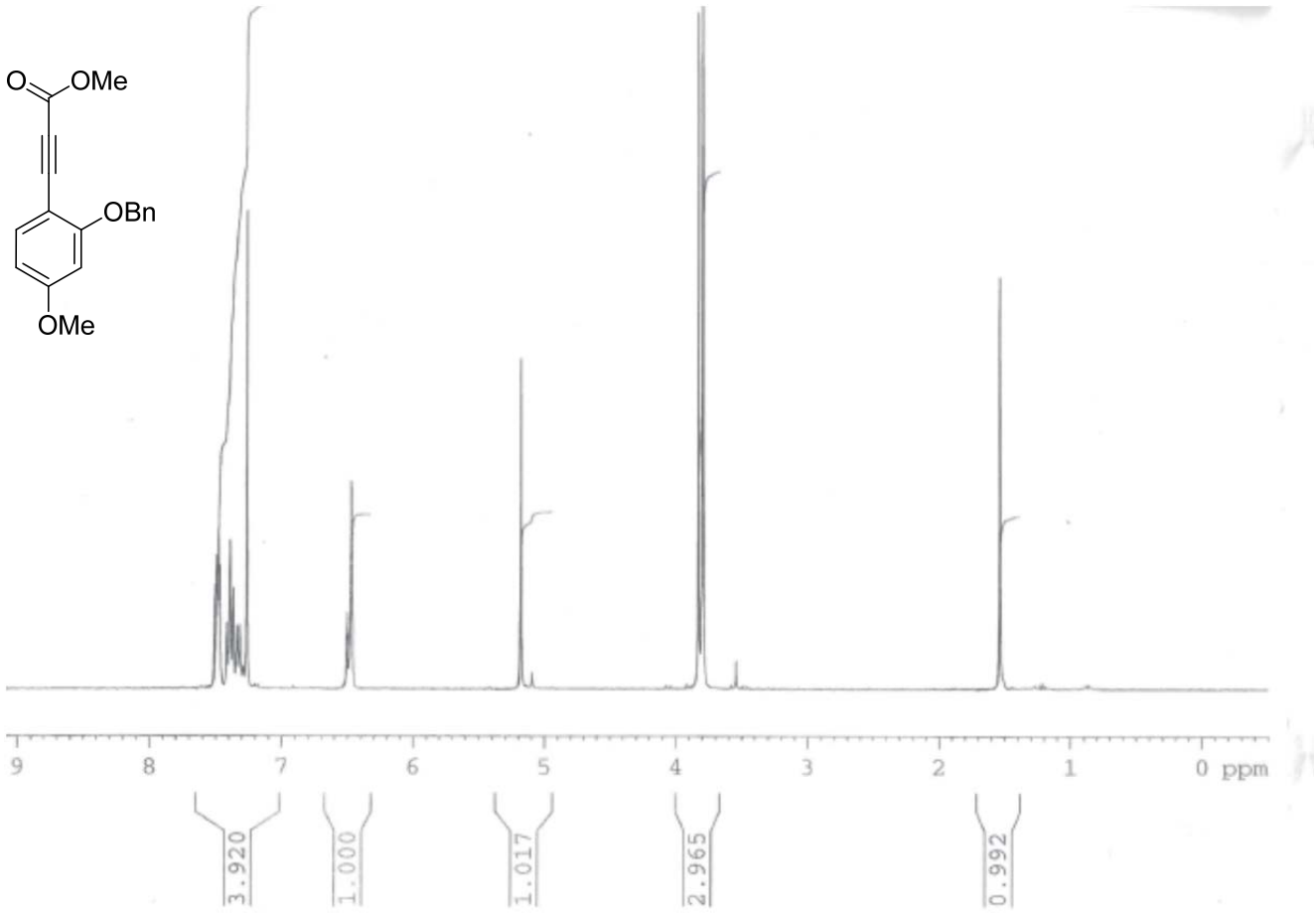

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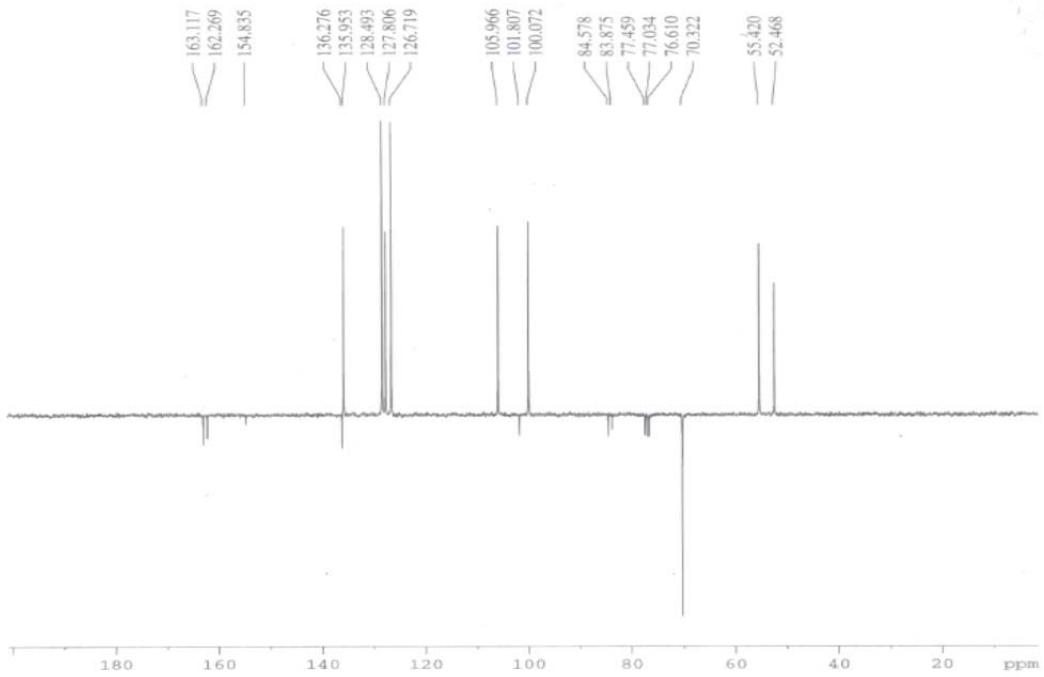

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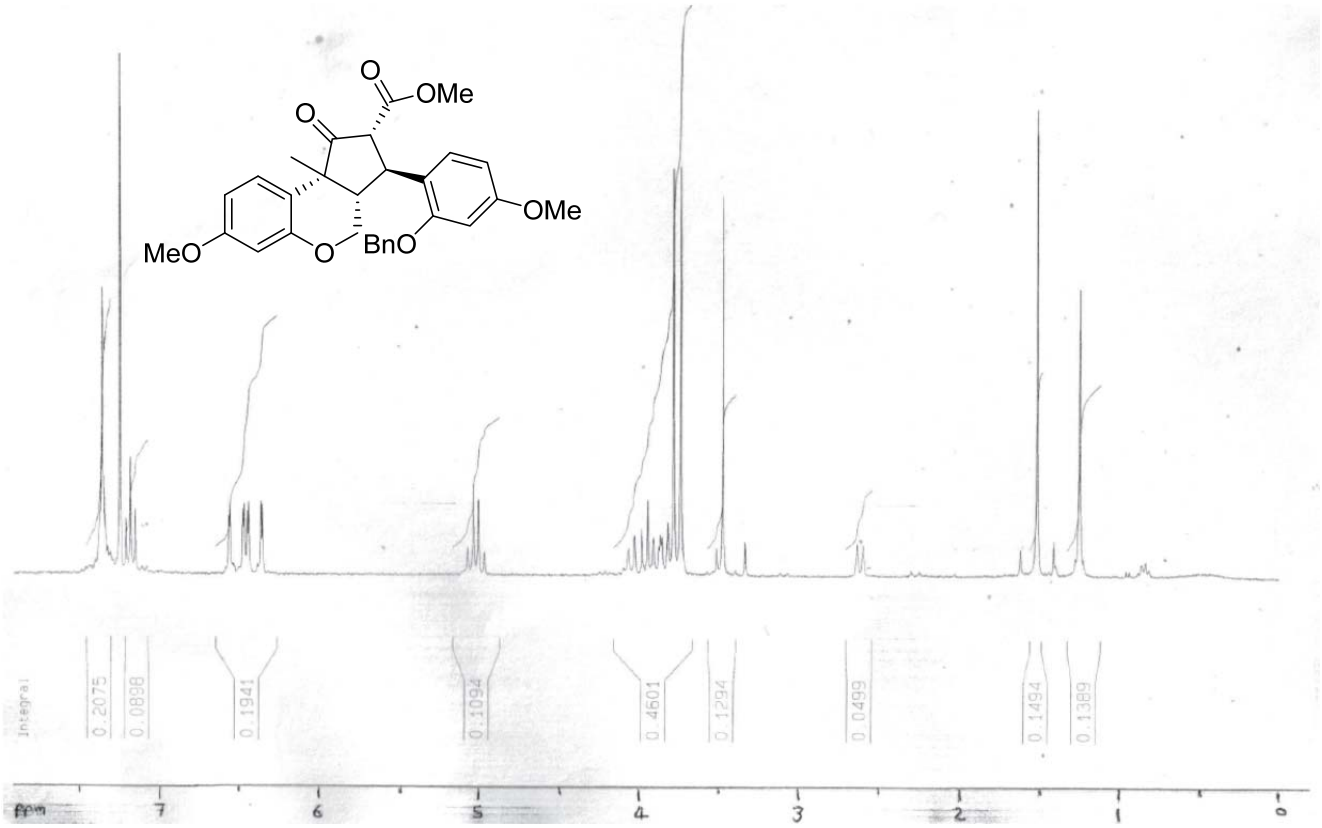

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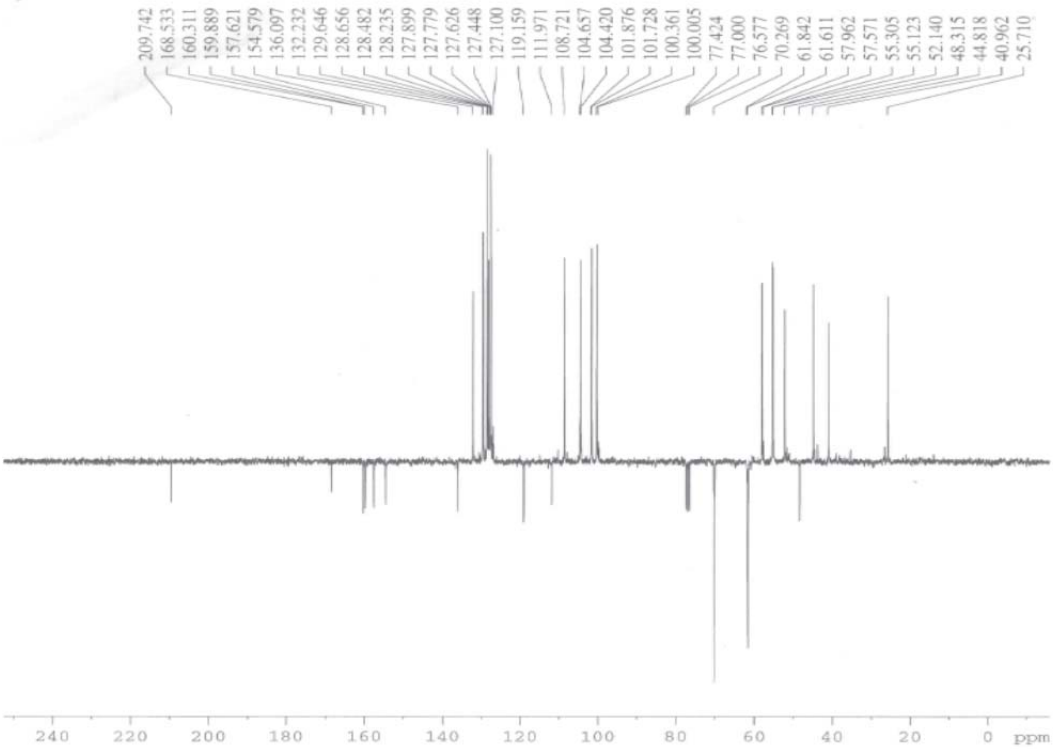

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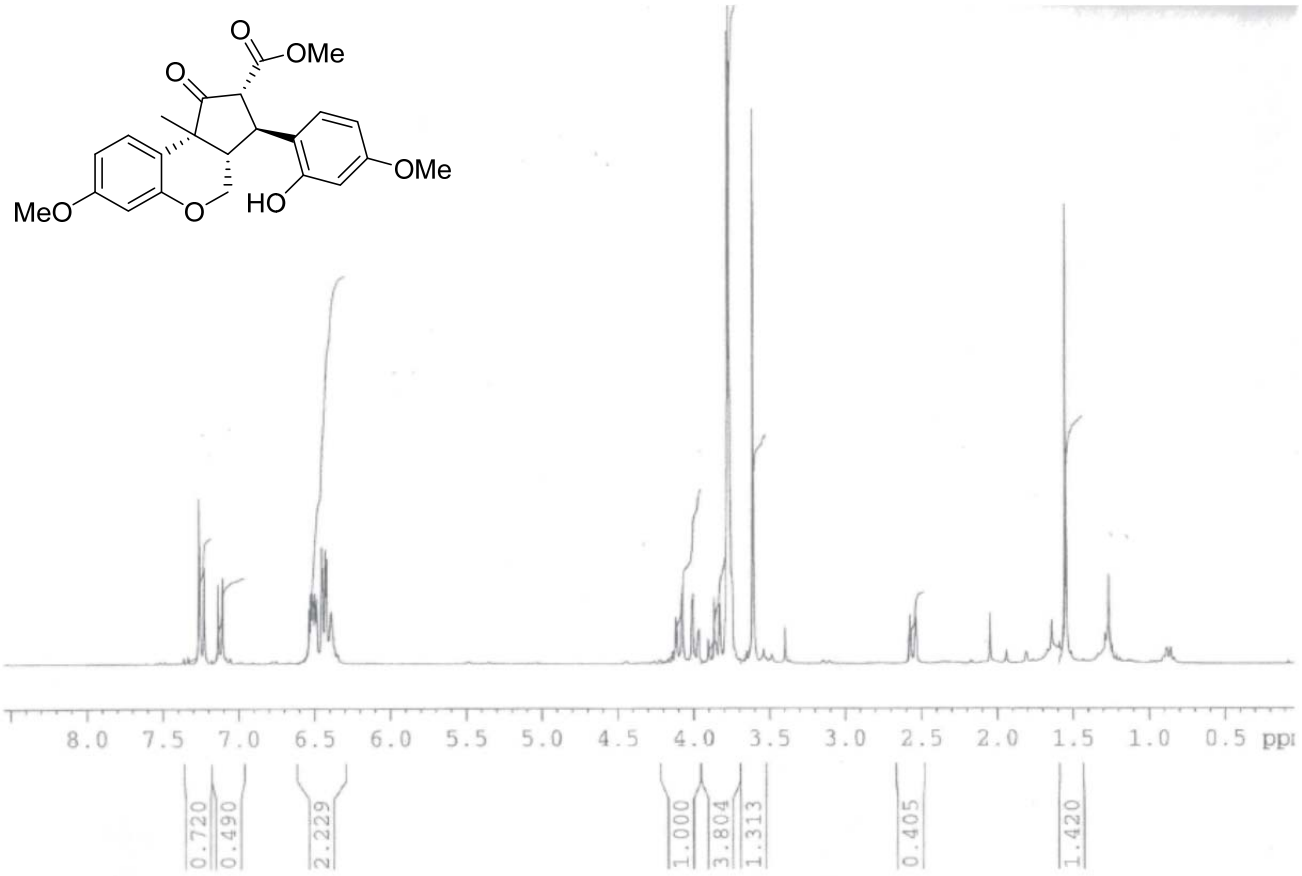

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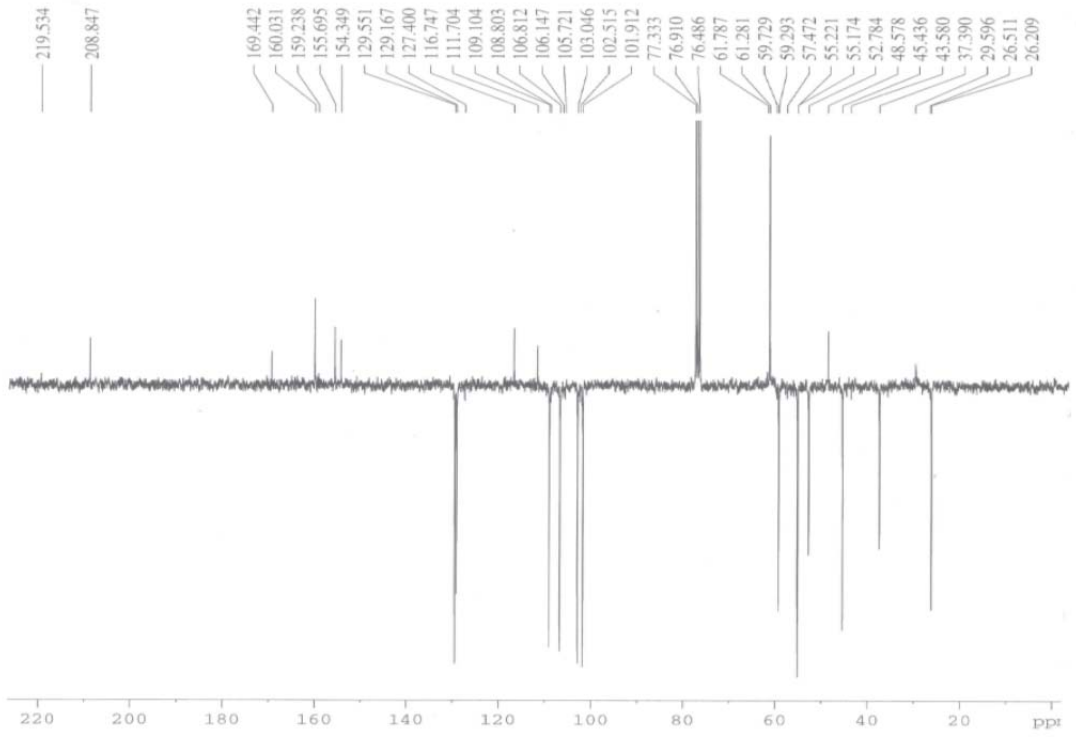

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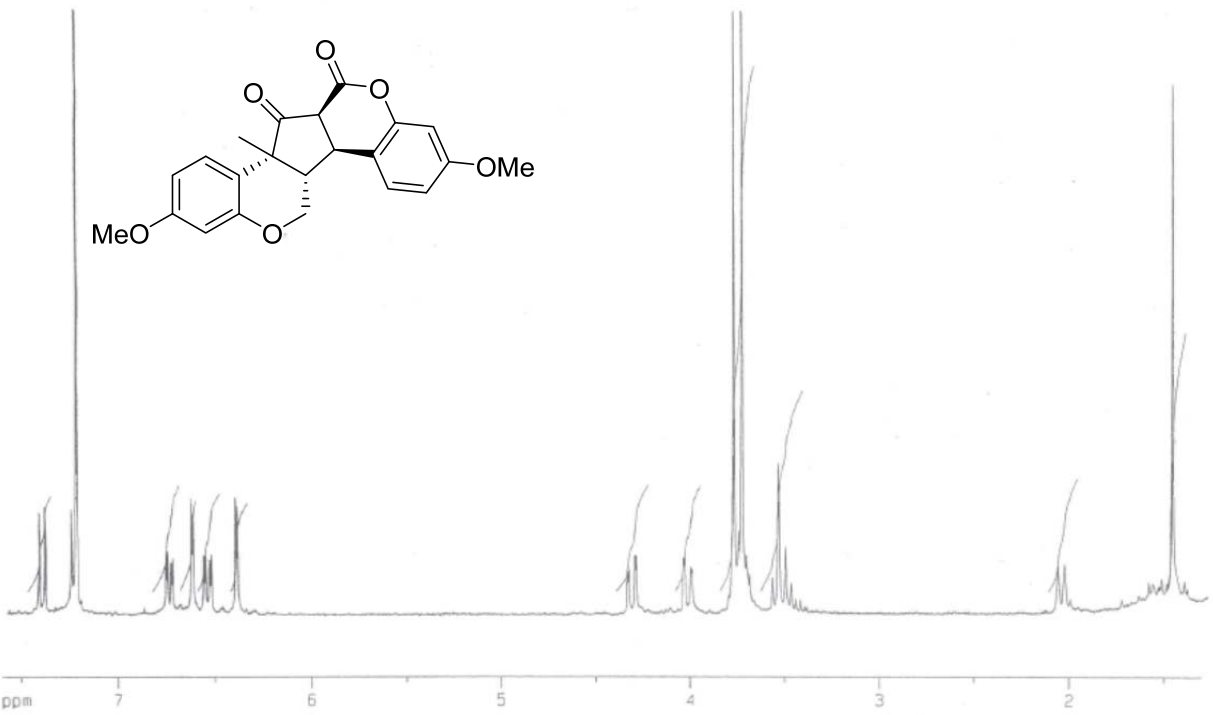

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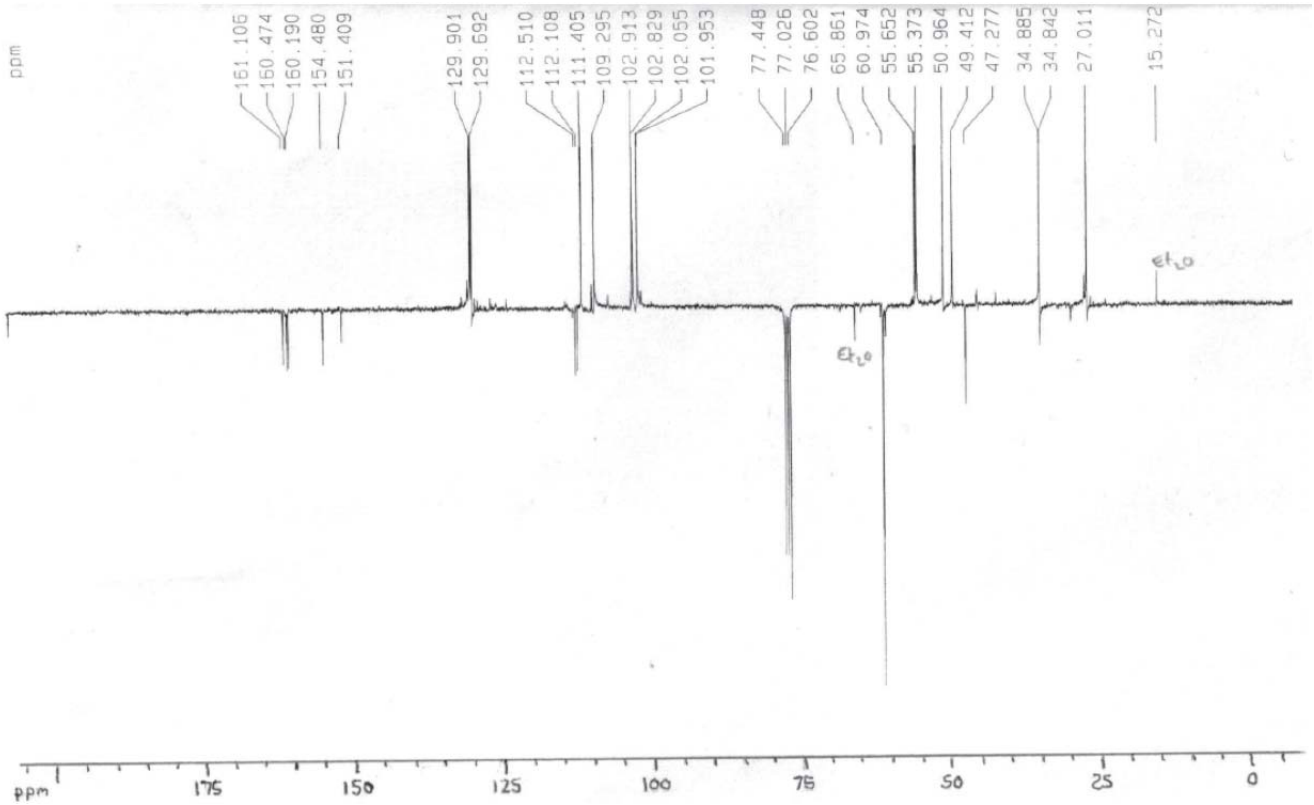

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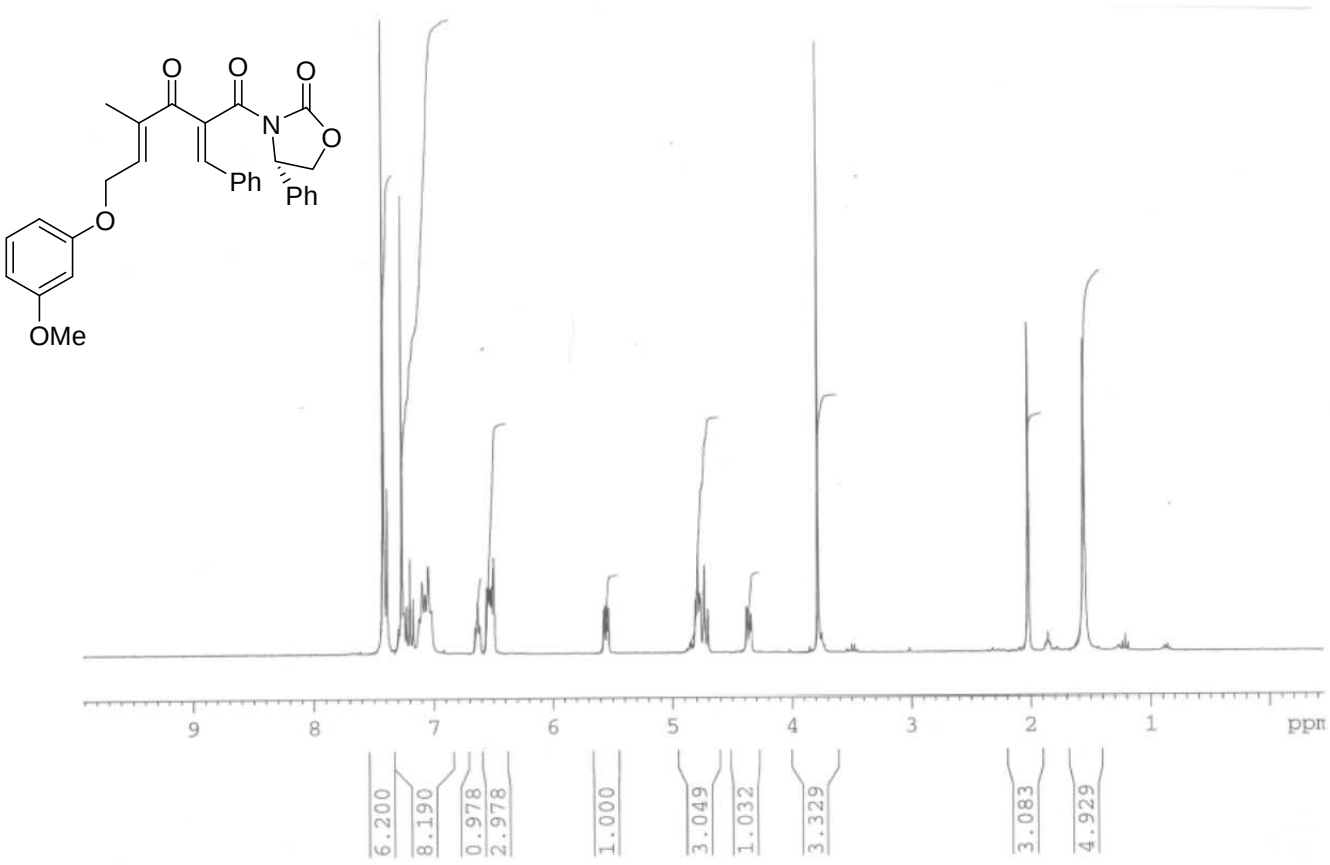
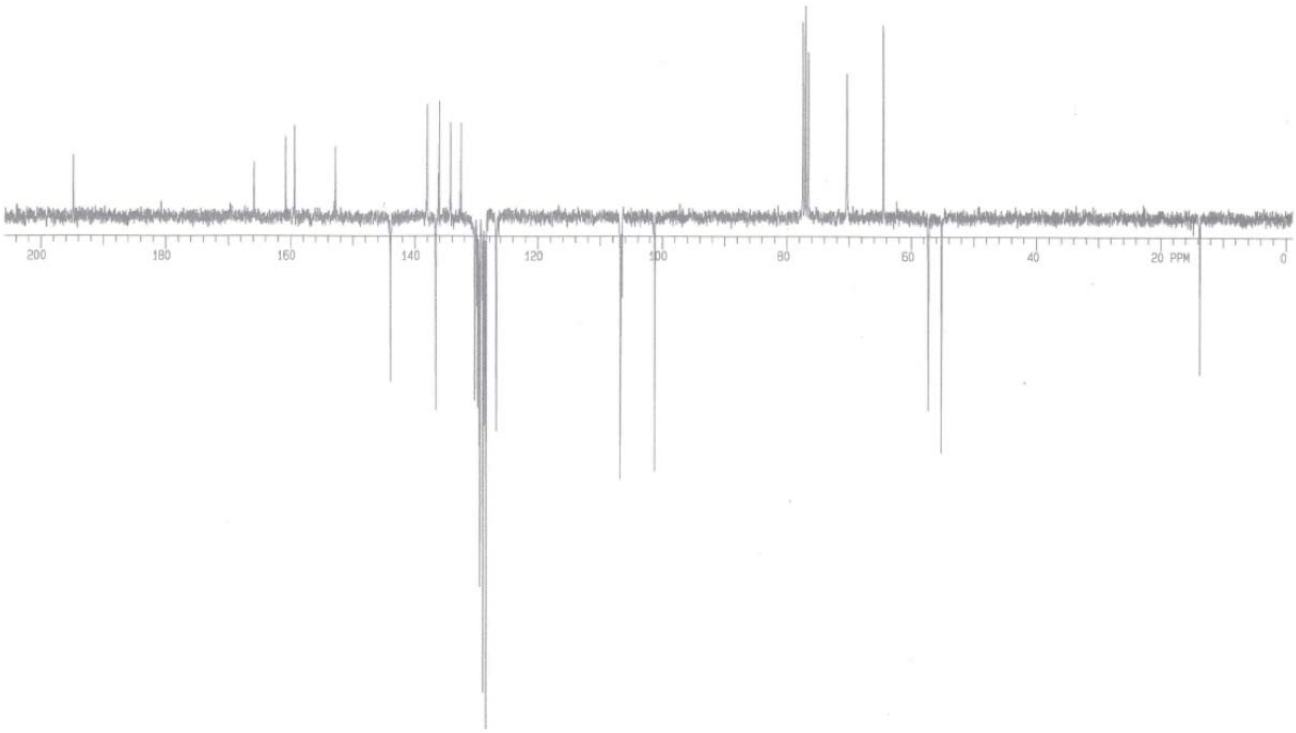


^{13}C NMR ($CDCl_3$) 75 MHz Compound 16



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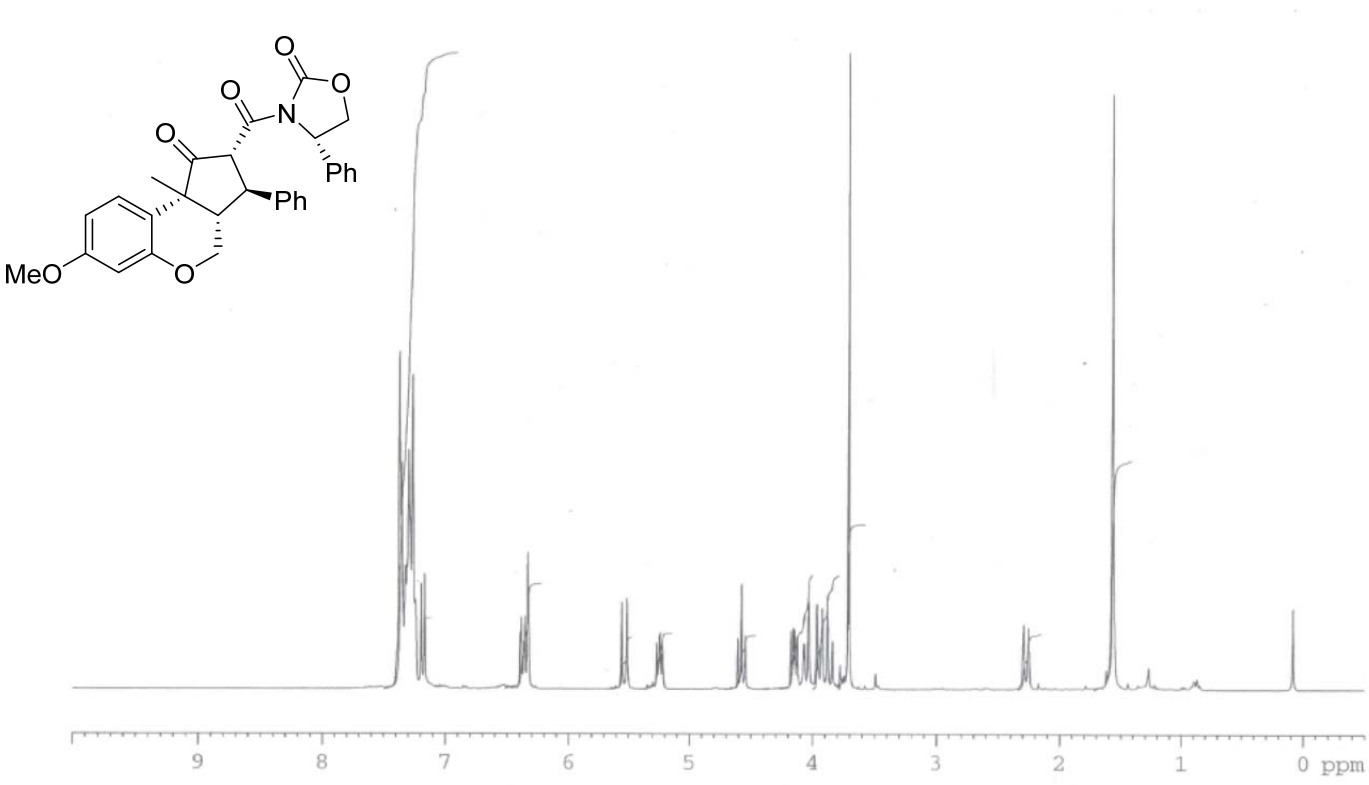
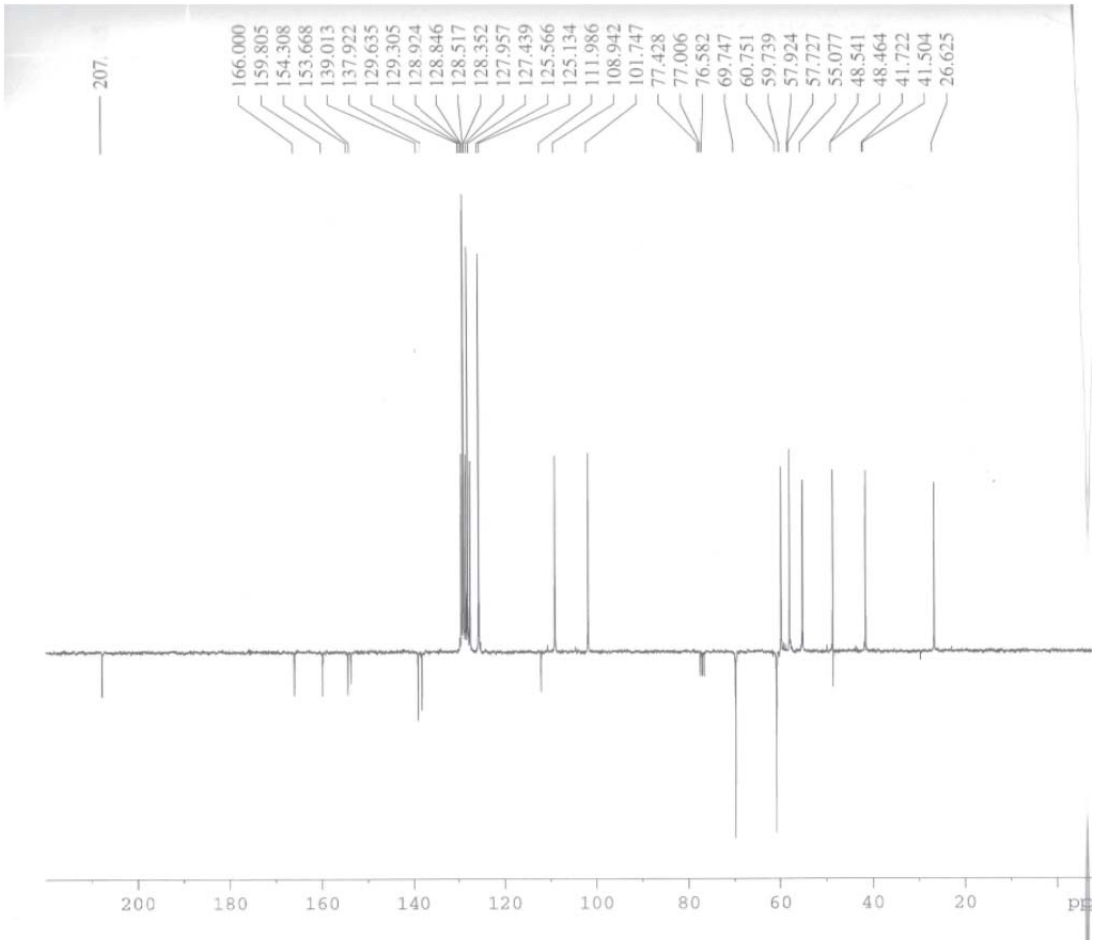
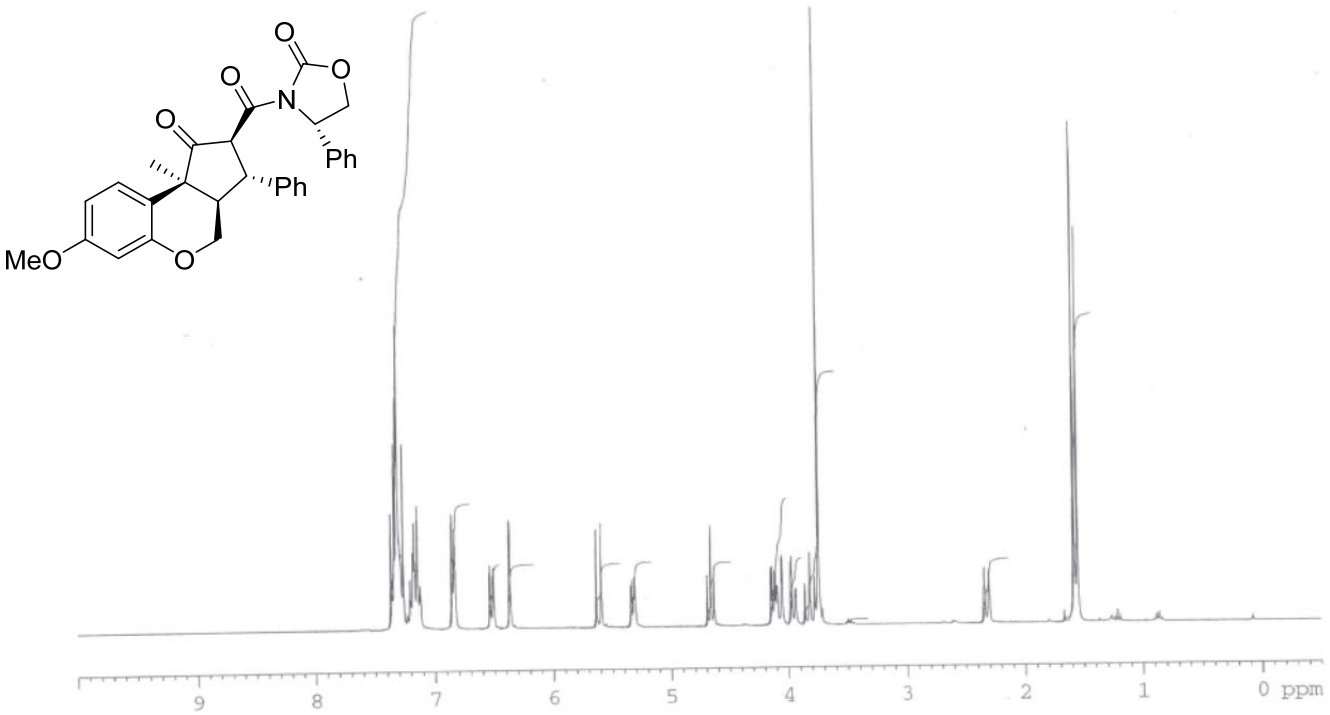


^{13}C NMR ($CDCl_3$) 75 MHz Compound 17



^{1}H NMR ($CDCl_3$) 300 MHz Compound 17'



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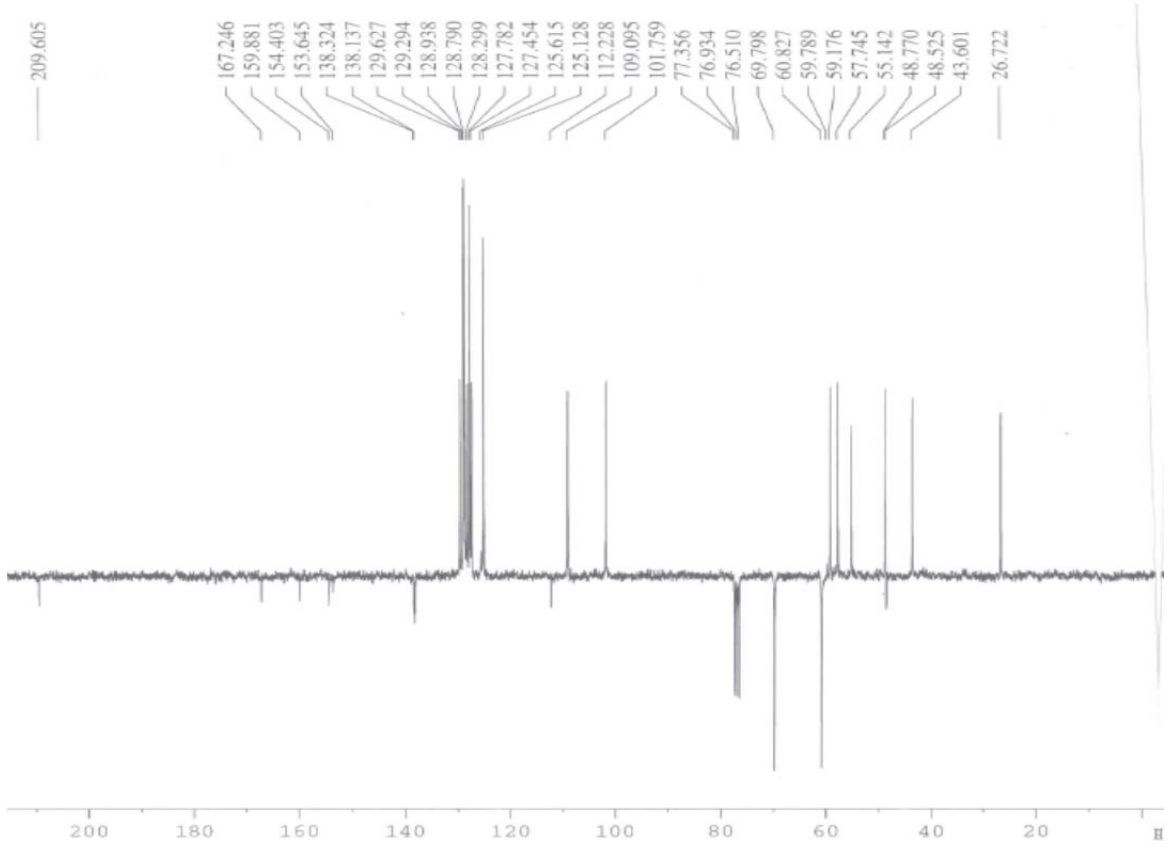

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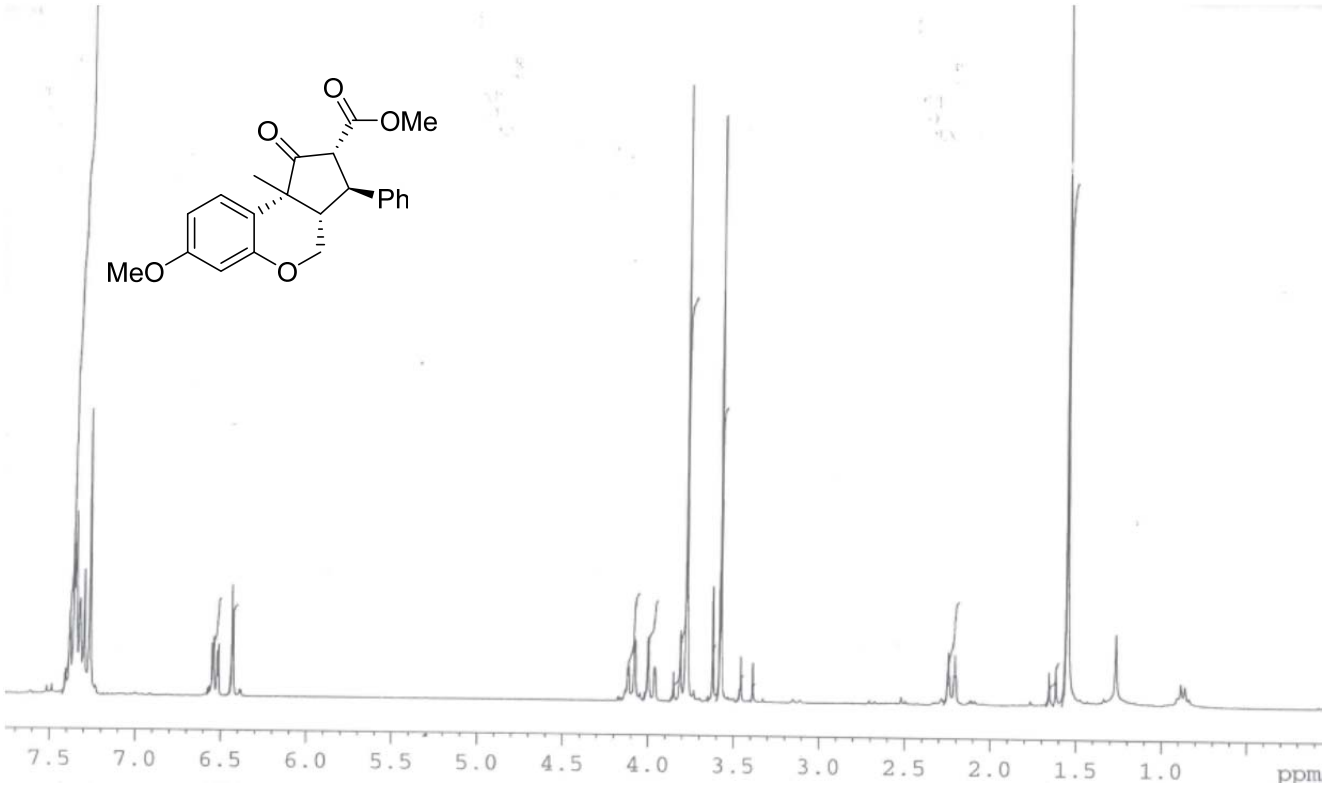

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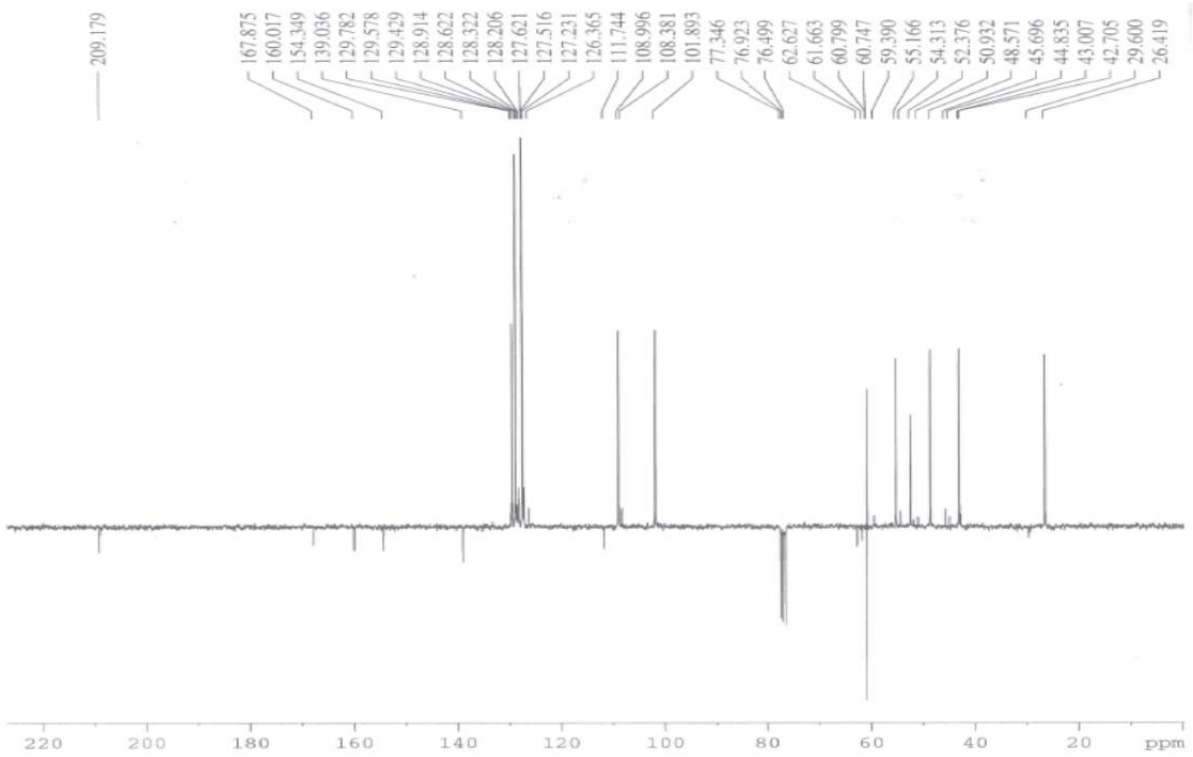

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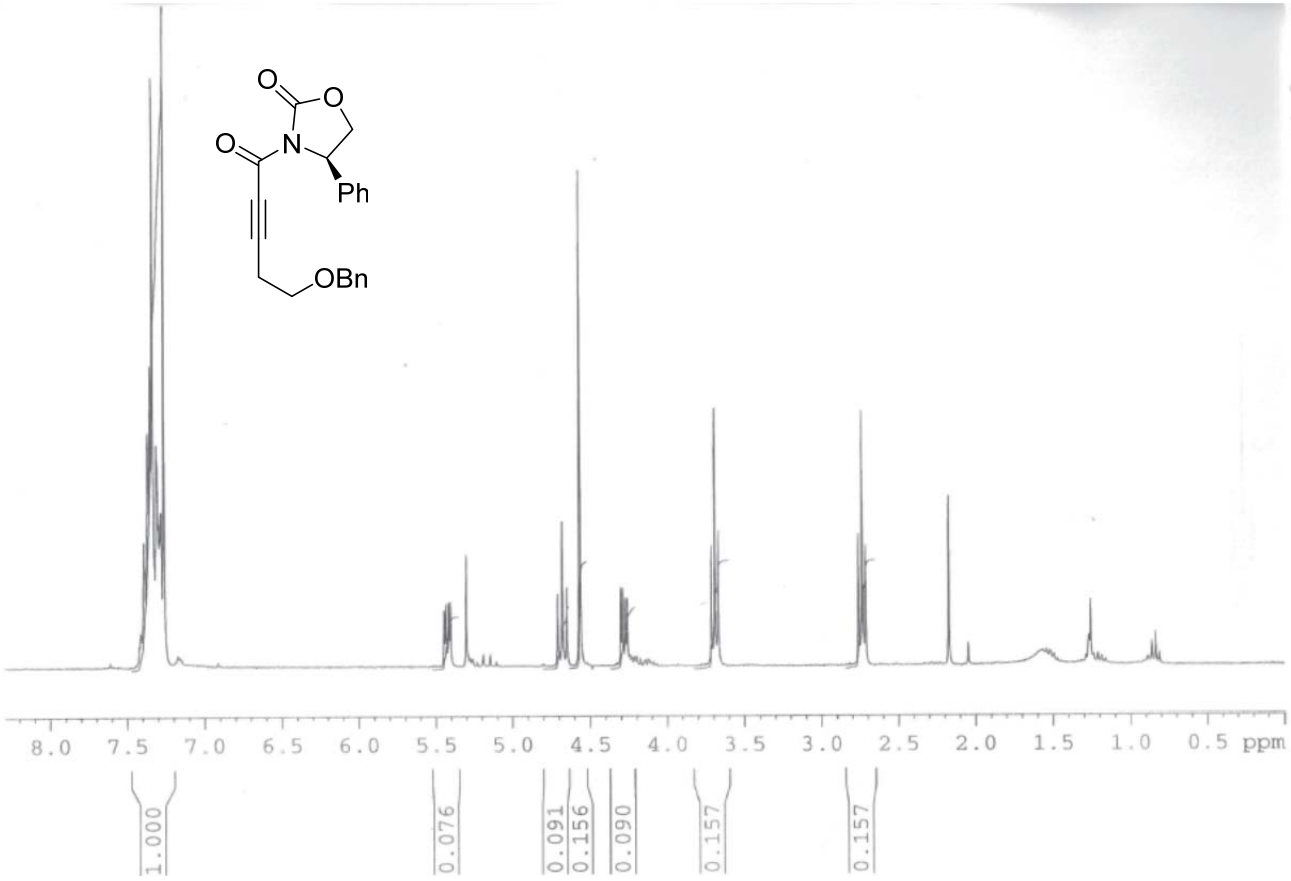

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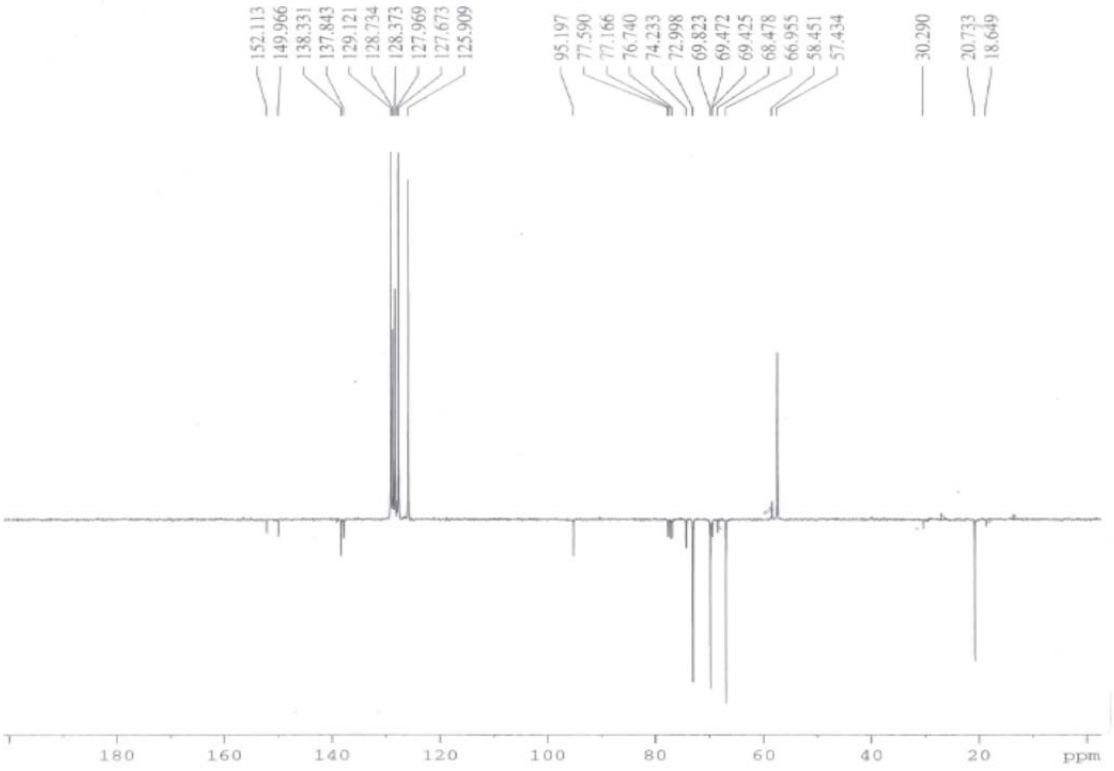

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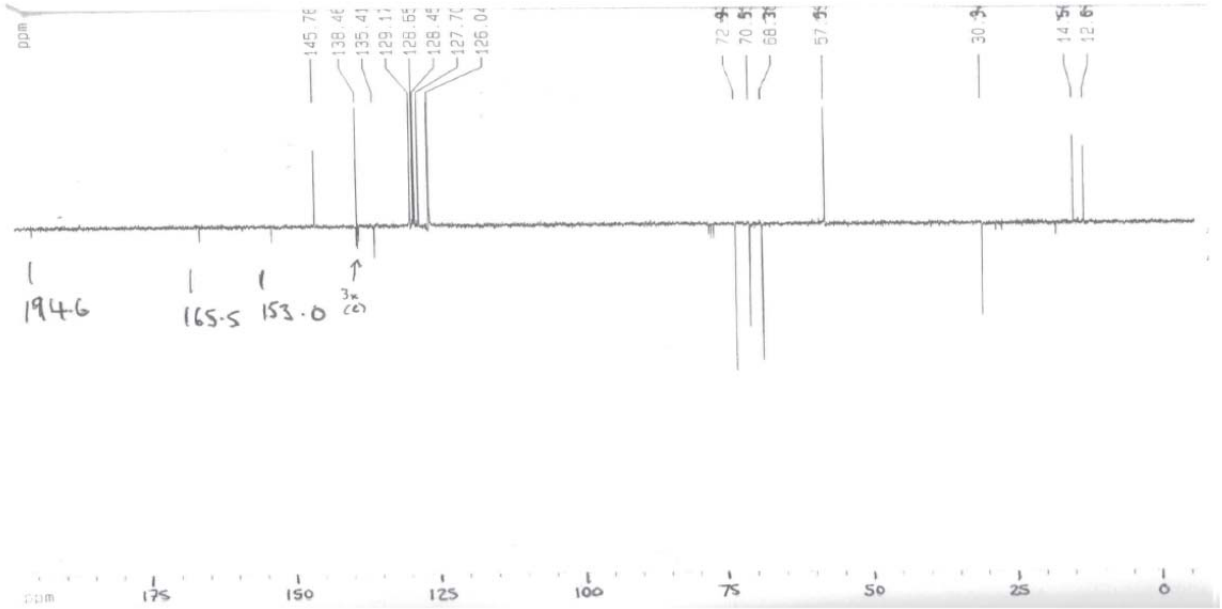
^{13}C NMR ($CDCl_3$) 75 MHz Compound 22



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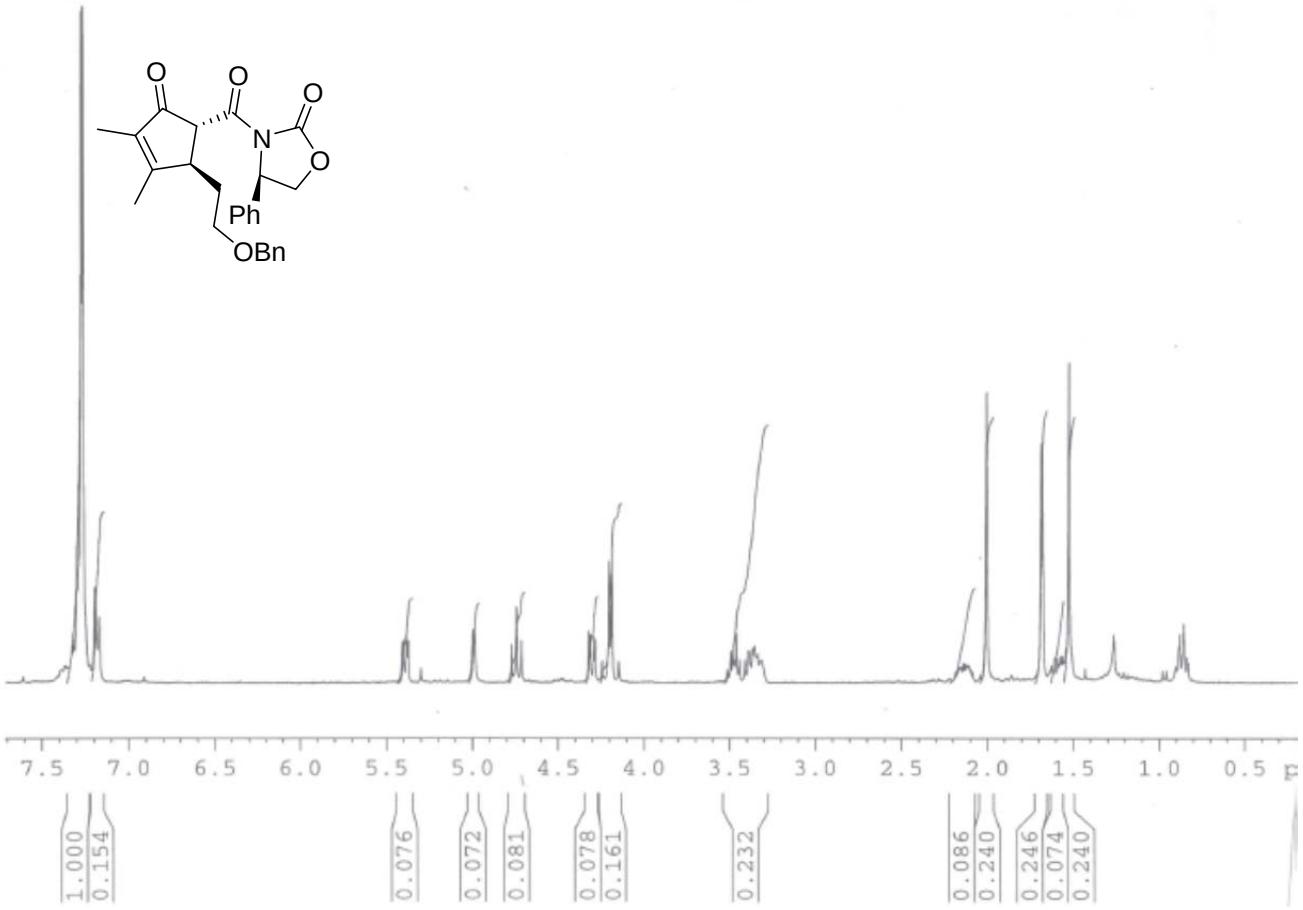

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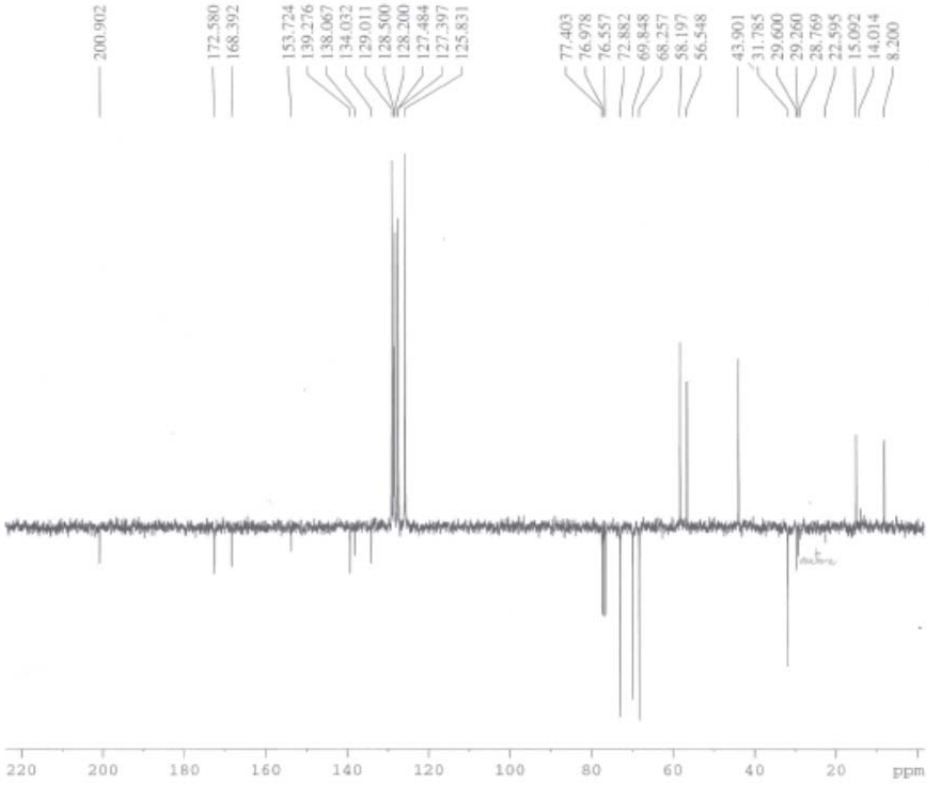

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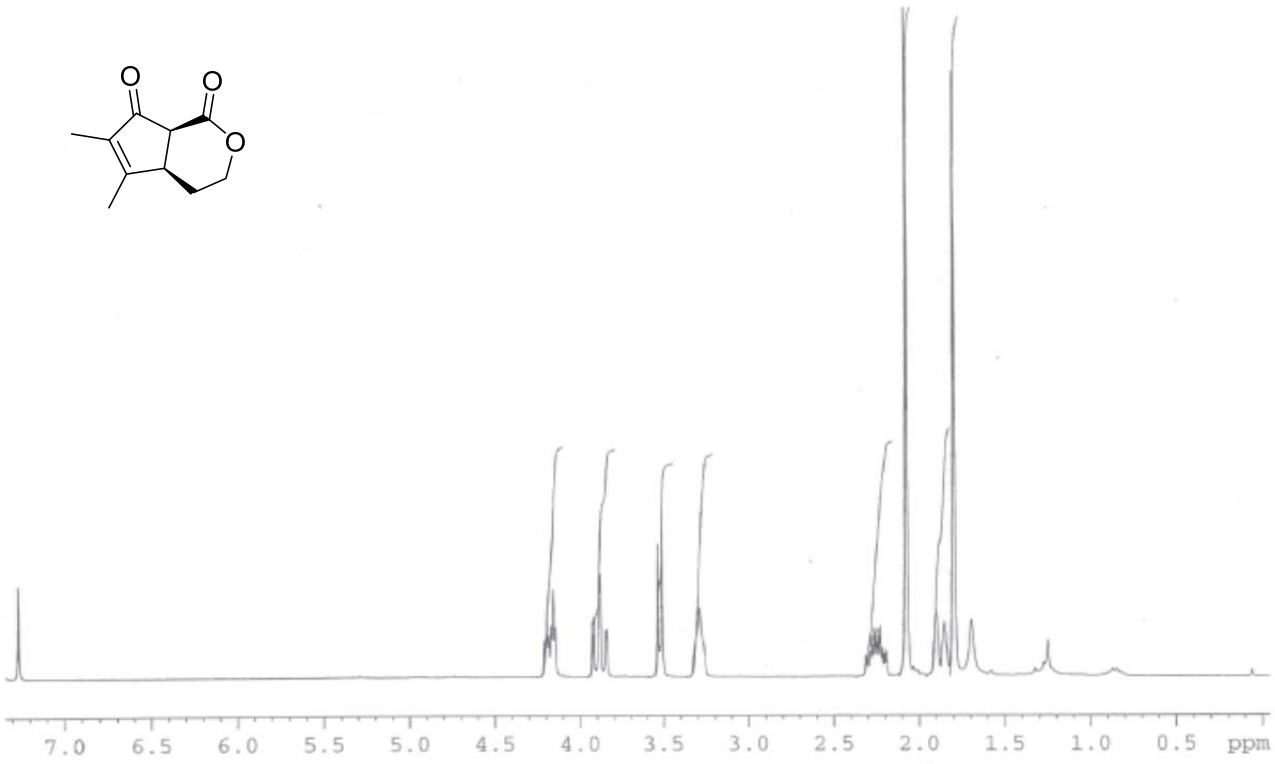

^{13}C NMR ($CDCl_3$) 75 MHz Compound 24