

SUPPORTING INFORMATION
FOR

Towards the Total Synthesis of Alpinkidine: Michael Addition to Isoquinolinetriene CE Ring-System Synthons

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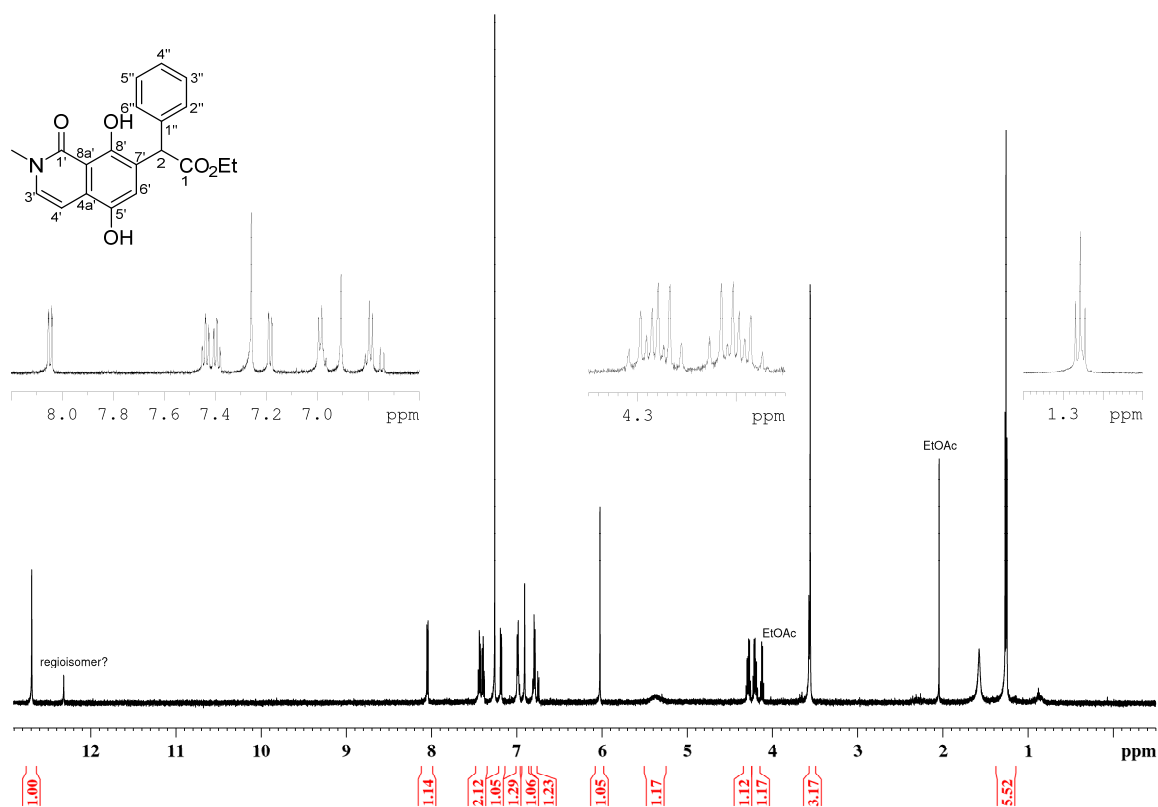
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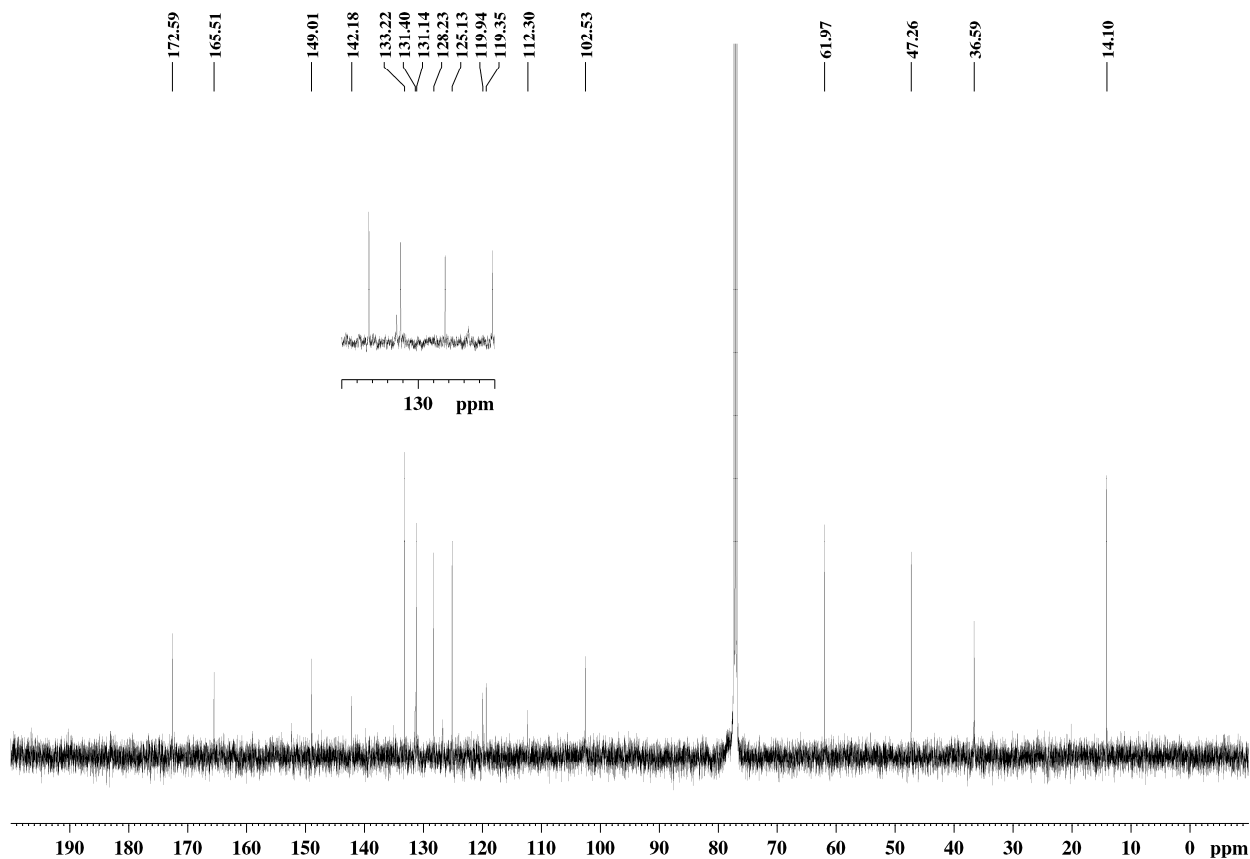
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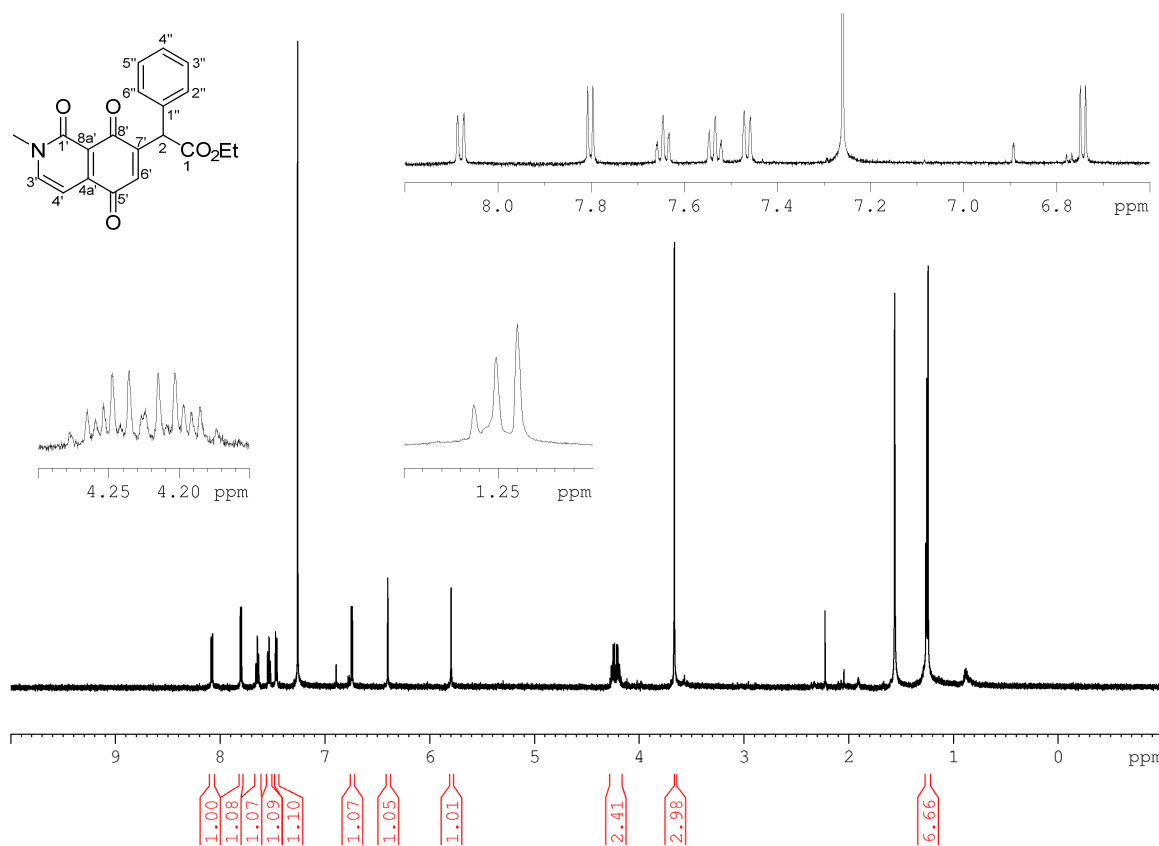
600 MHz ^1H NMR spectrum of **16** in CDCl_3



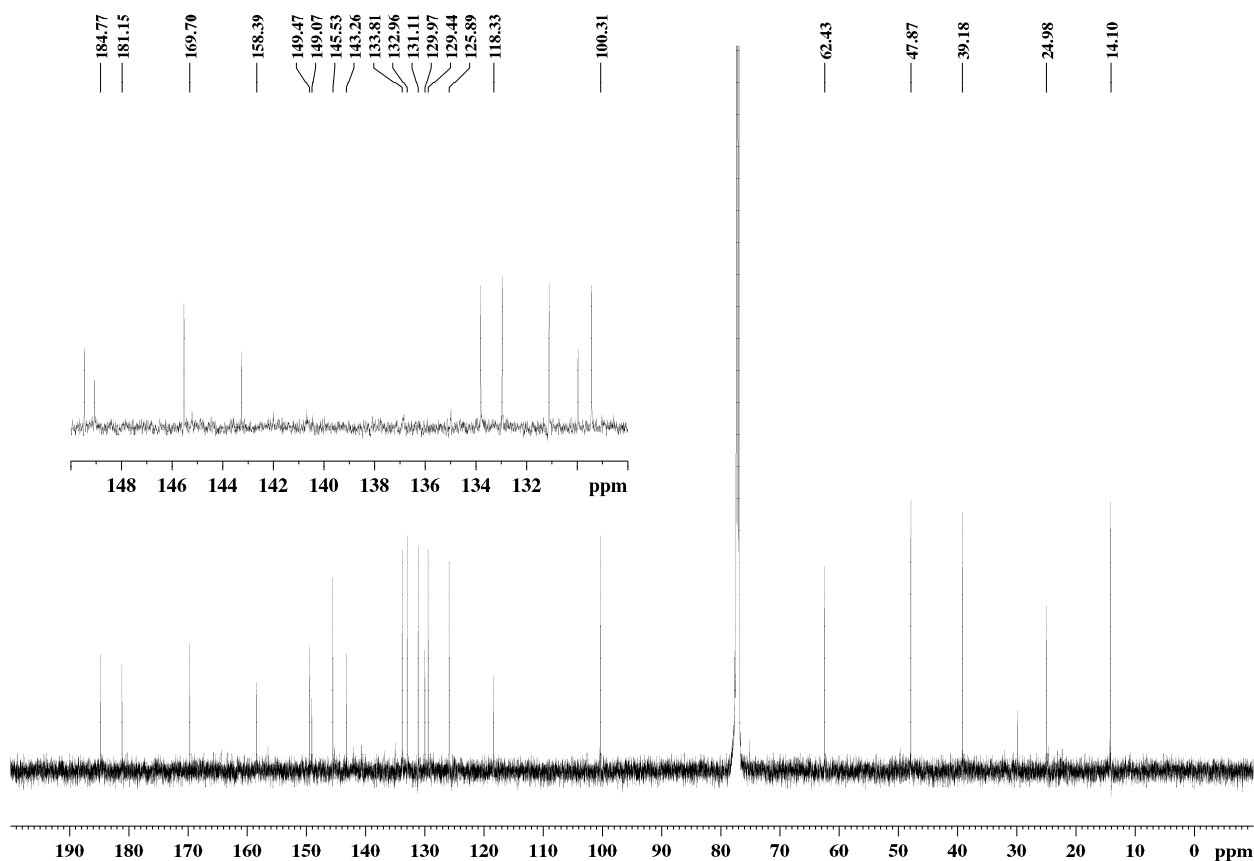
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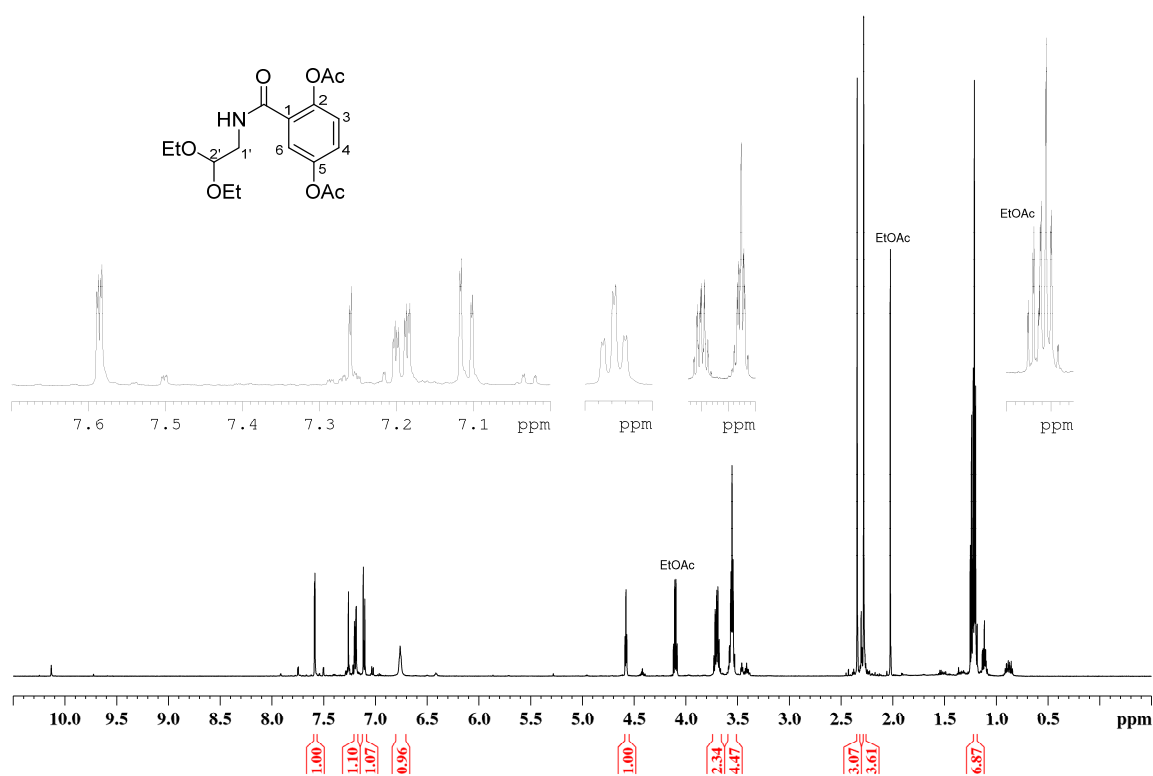
600 MHz ^1H NMR spectrum of **17** in CDCl_3



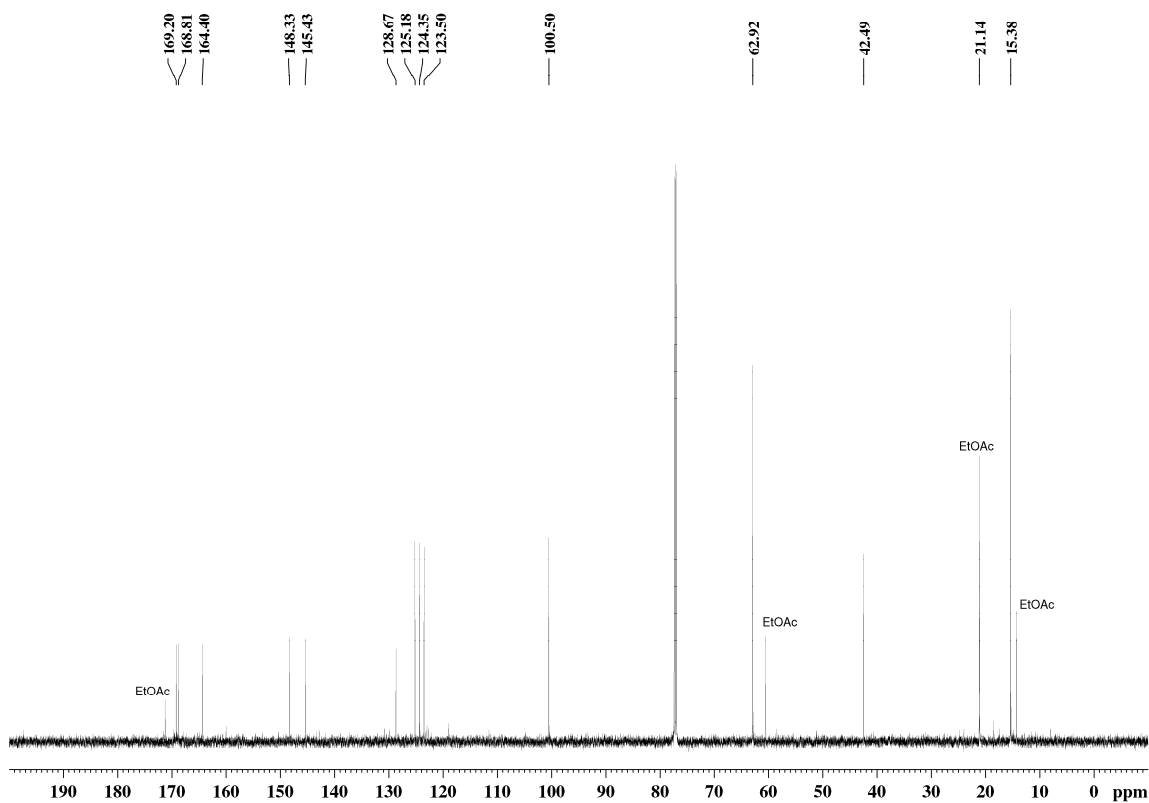
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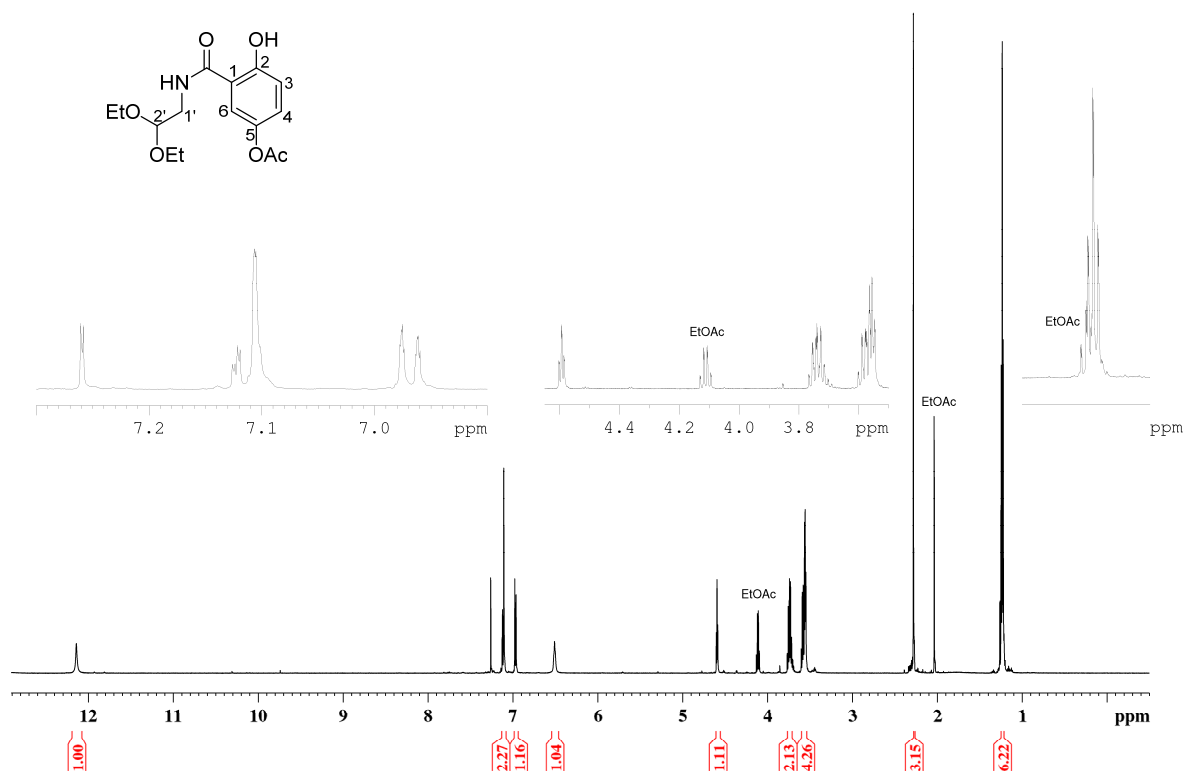
600 MHz ^1H NMR spectrum of **22** in CDCl_3



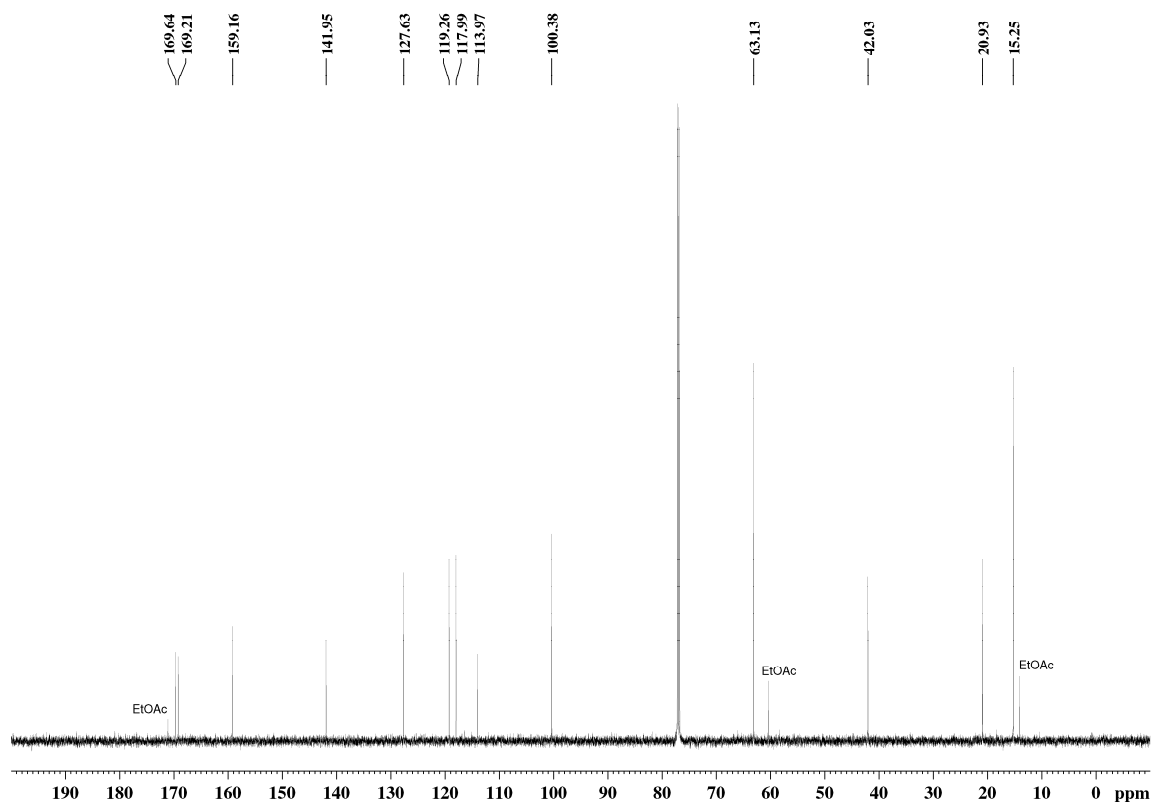
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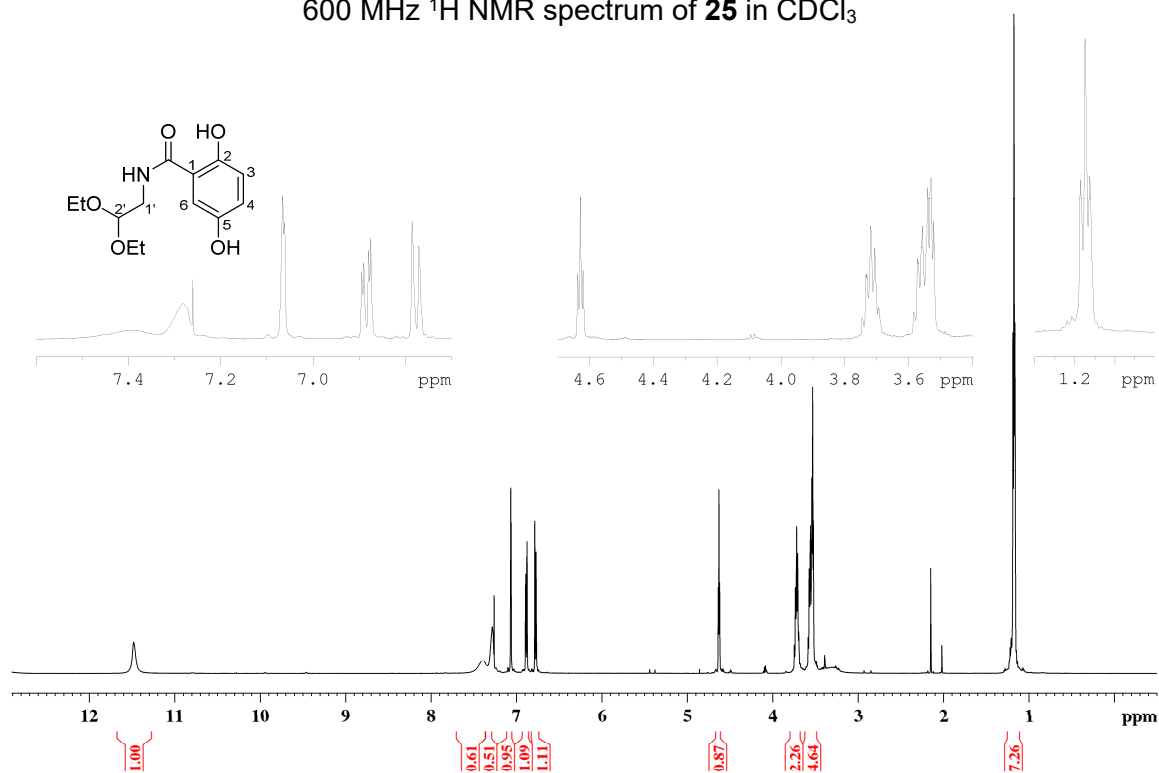
600 MHz ^1H NMR spectrum of **23** in CDCl_3



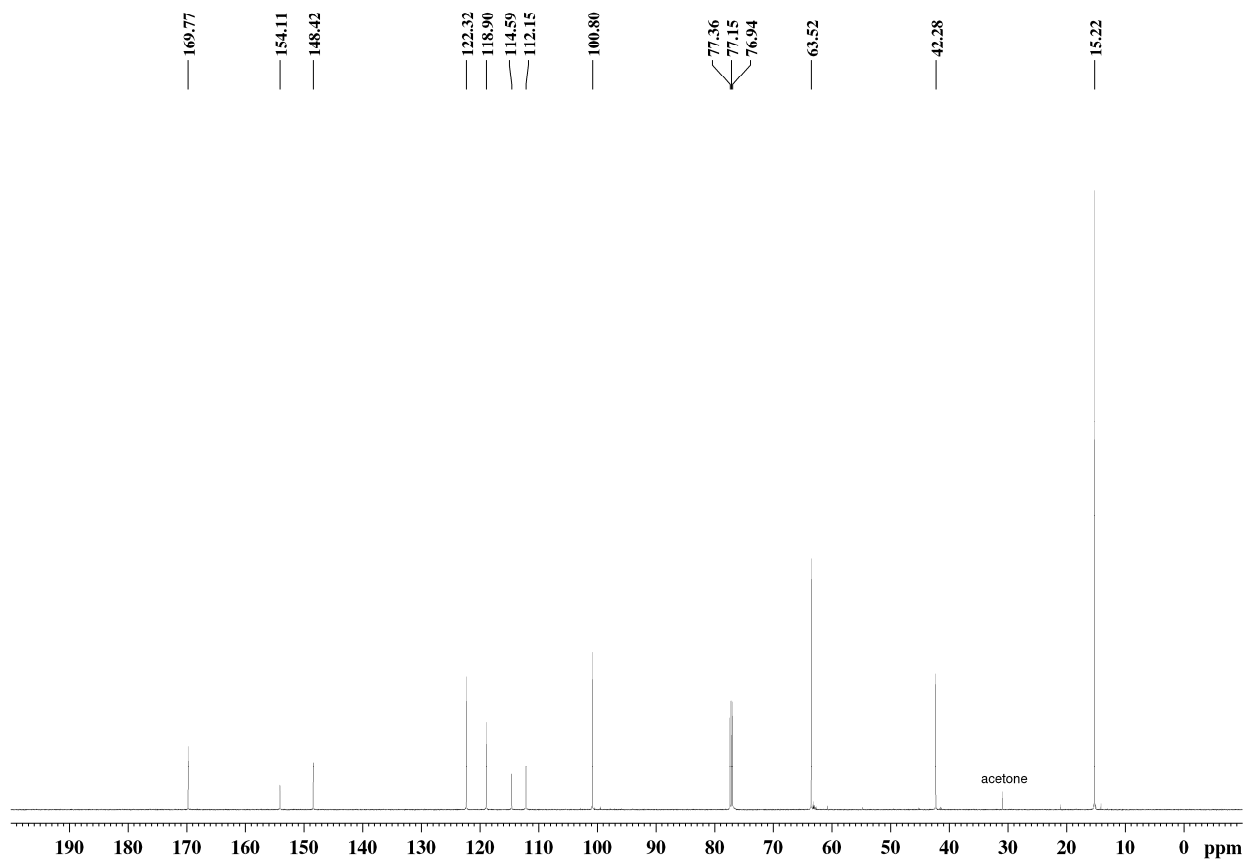
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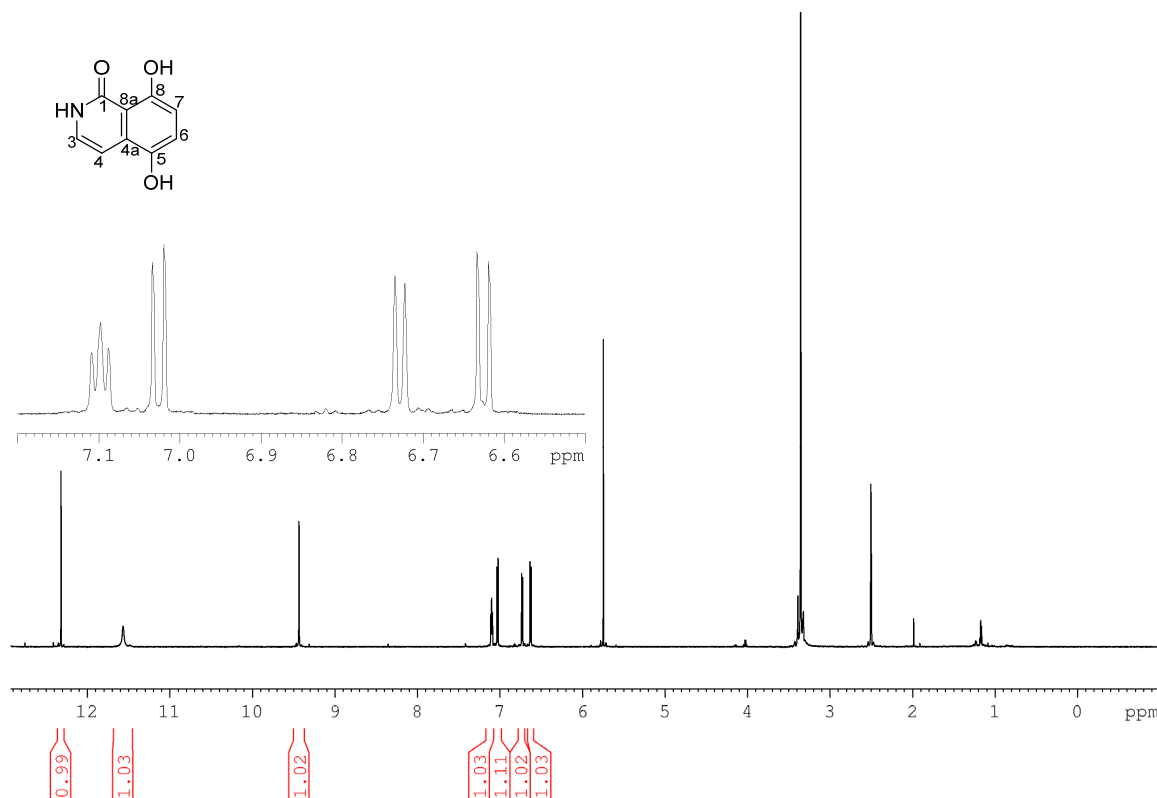
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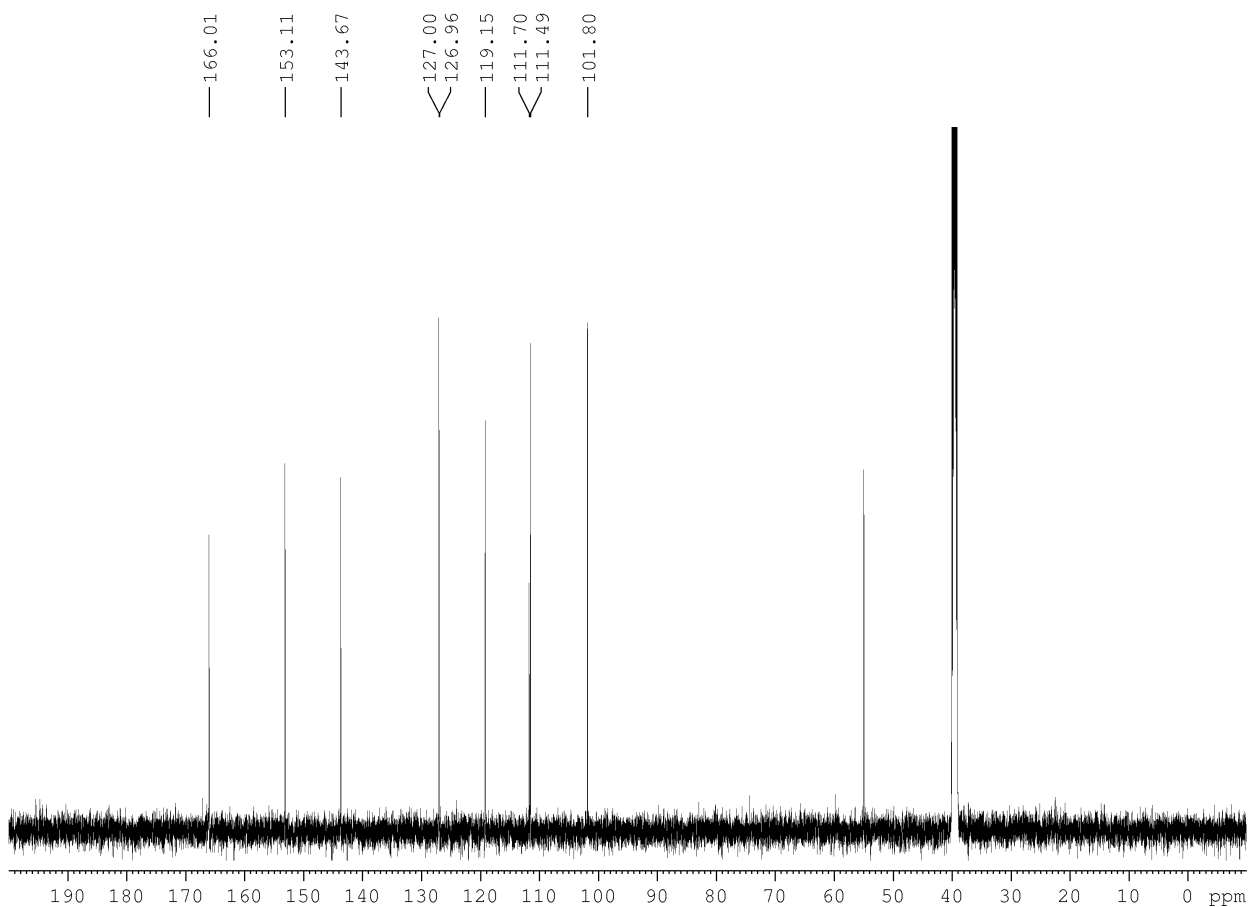
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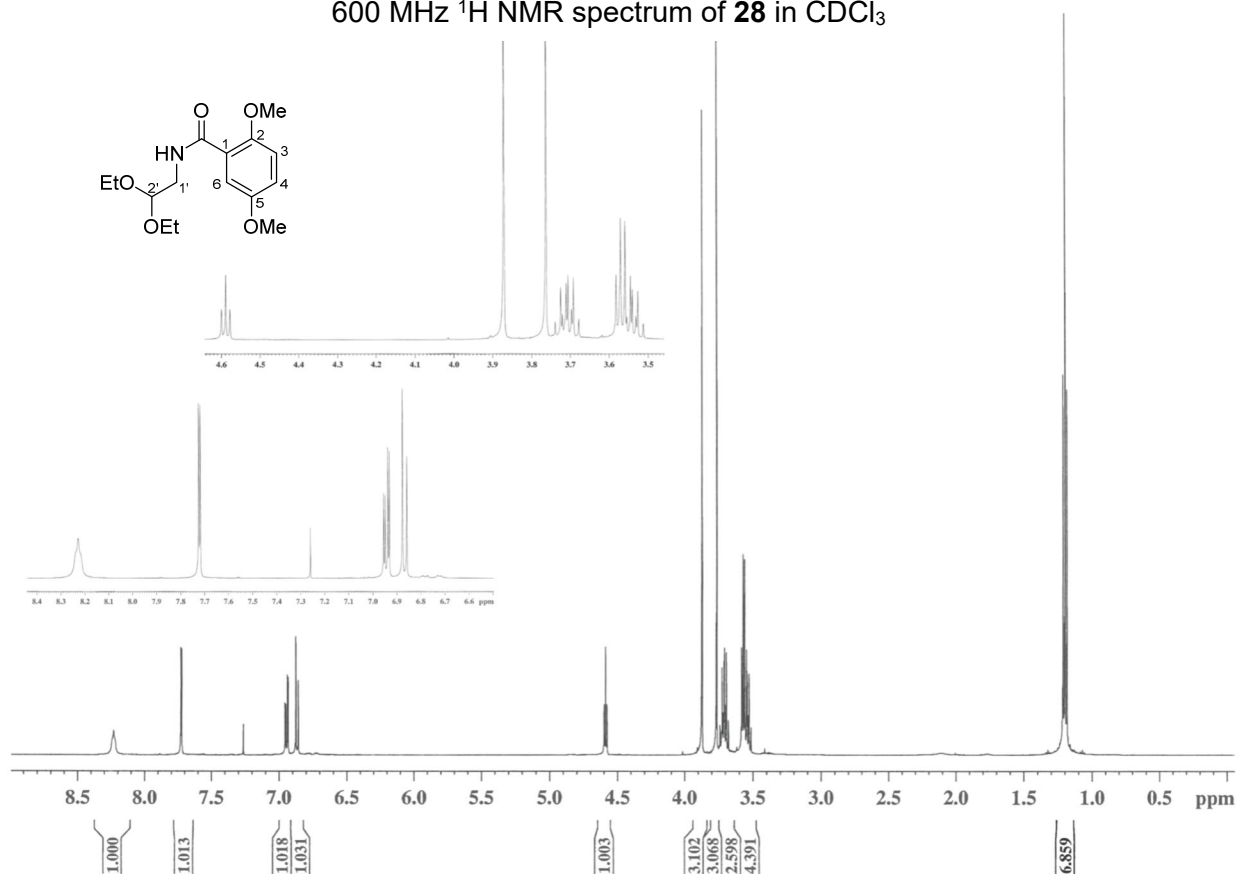
600 MHz ^1H NMR spectrum of **26** in d_6 -DMSO



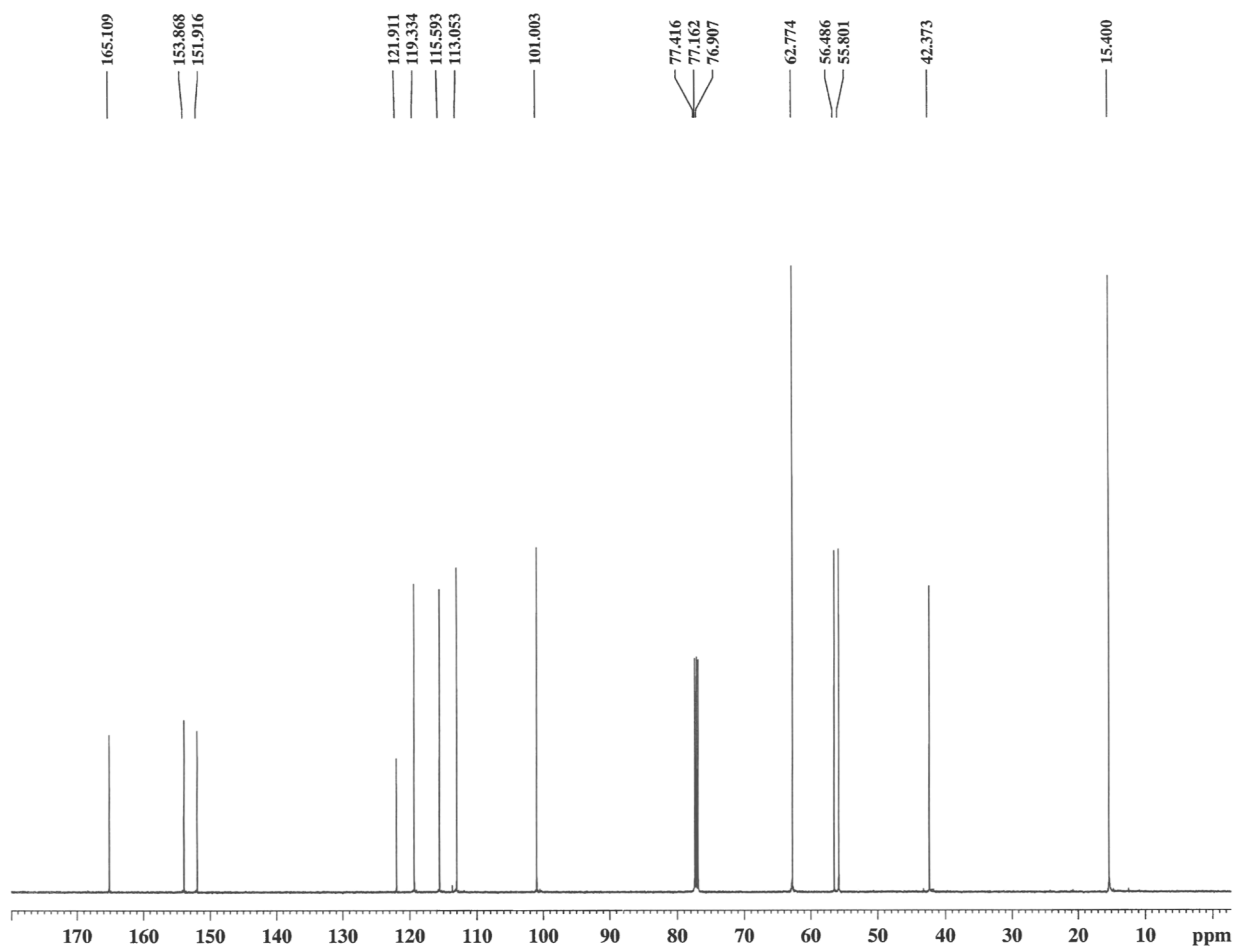
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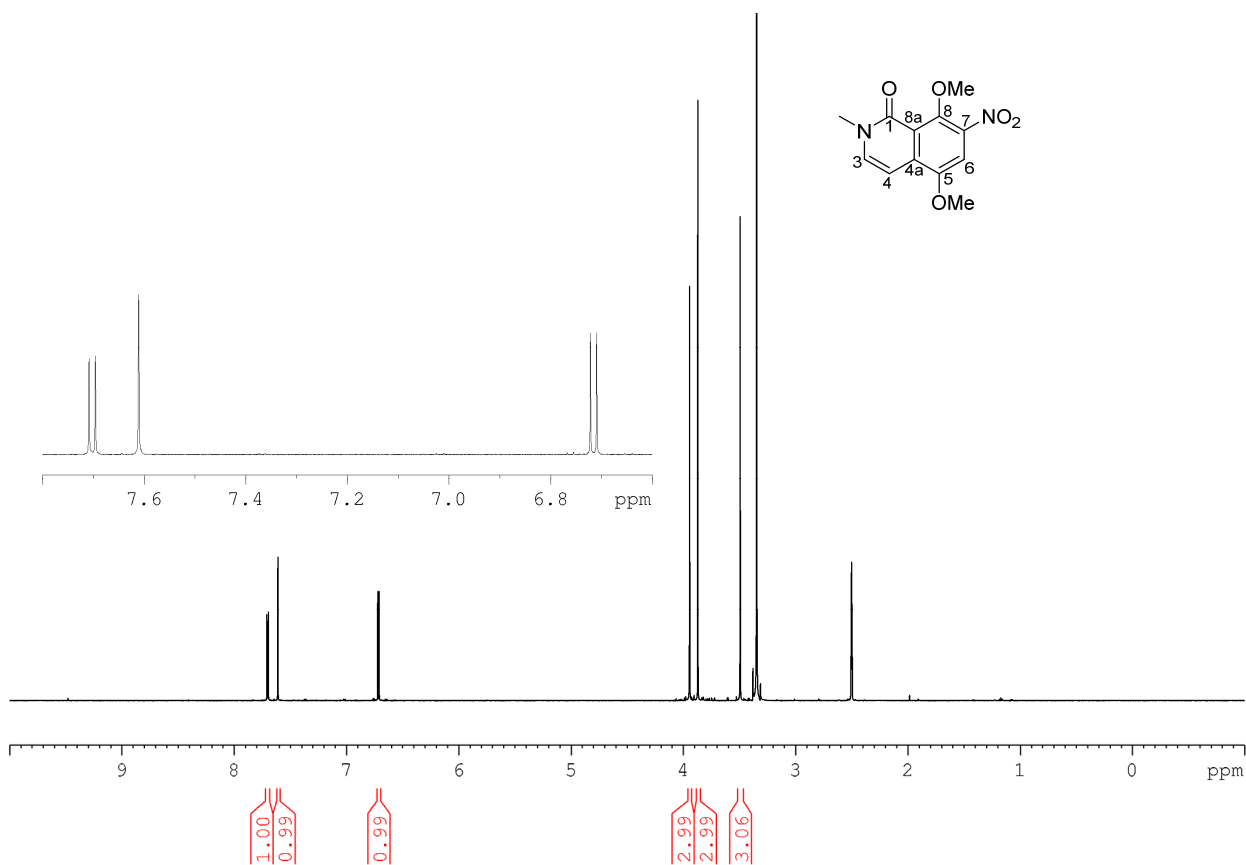
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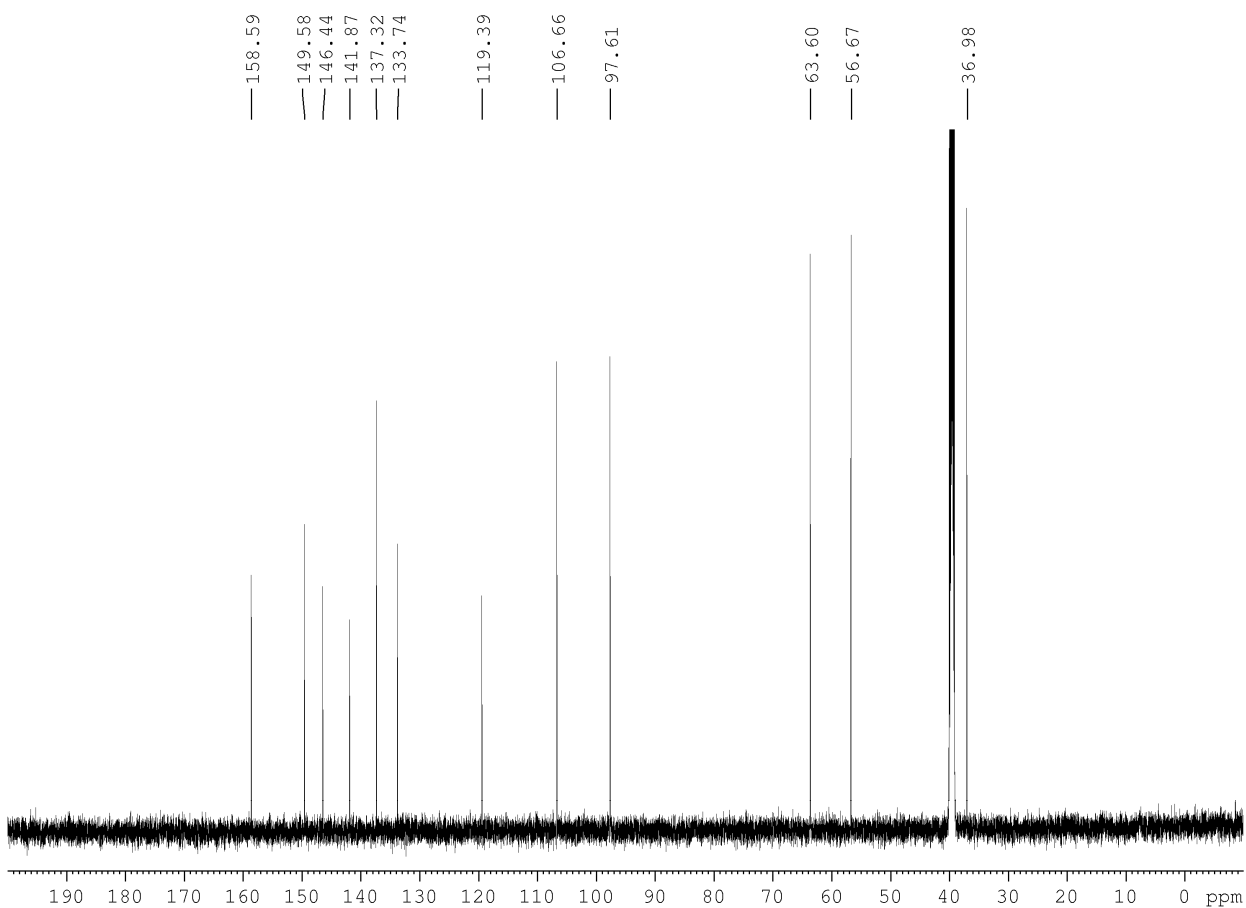
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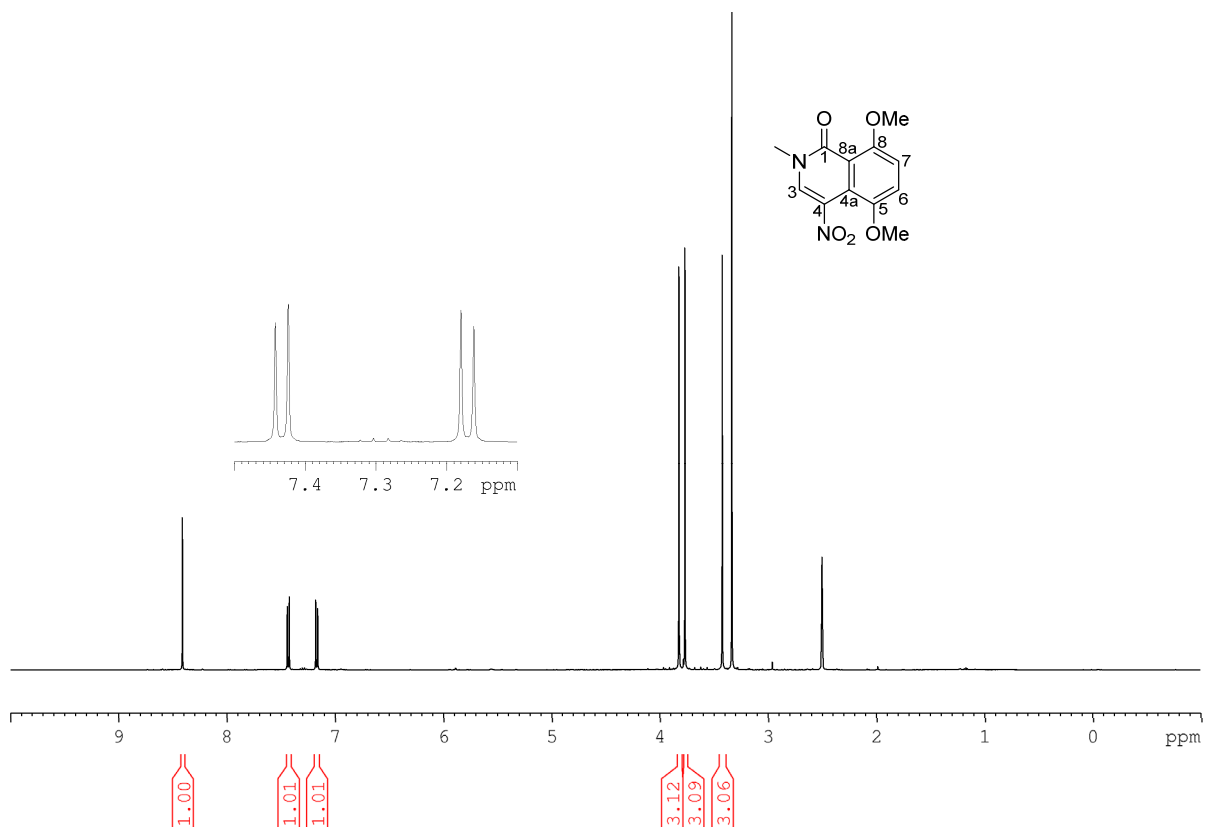
600 MHz ^1H NMR spectrum of **36** in d_6 -DMSO



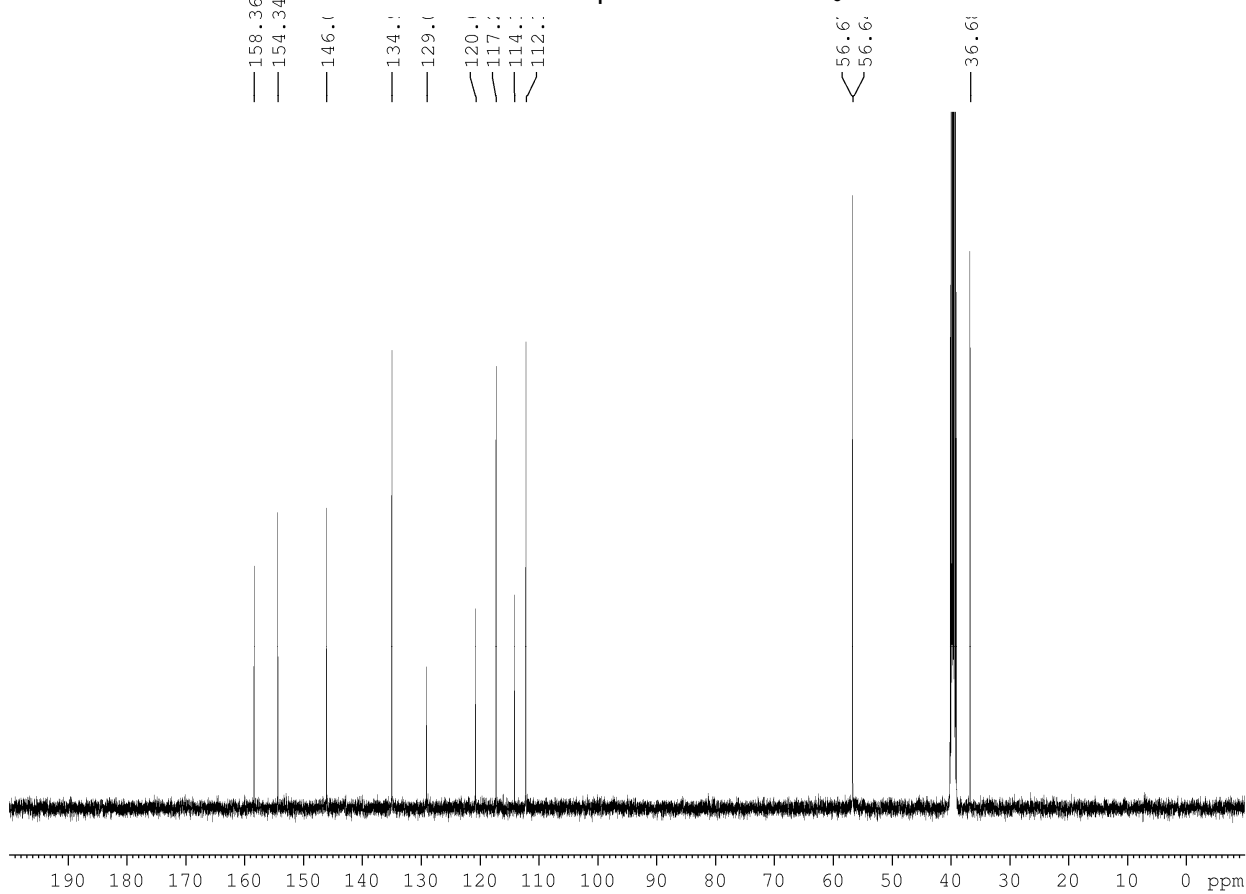
150 MHz ^{13}C NMR spectrum of **36** in d_6 -DMSO



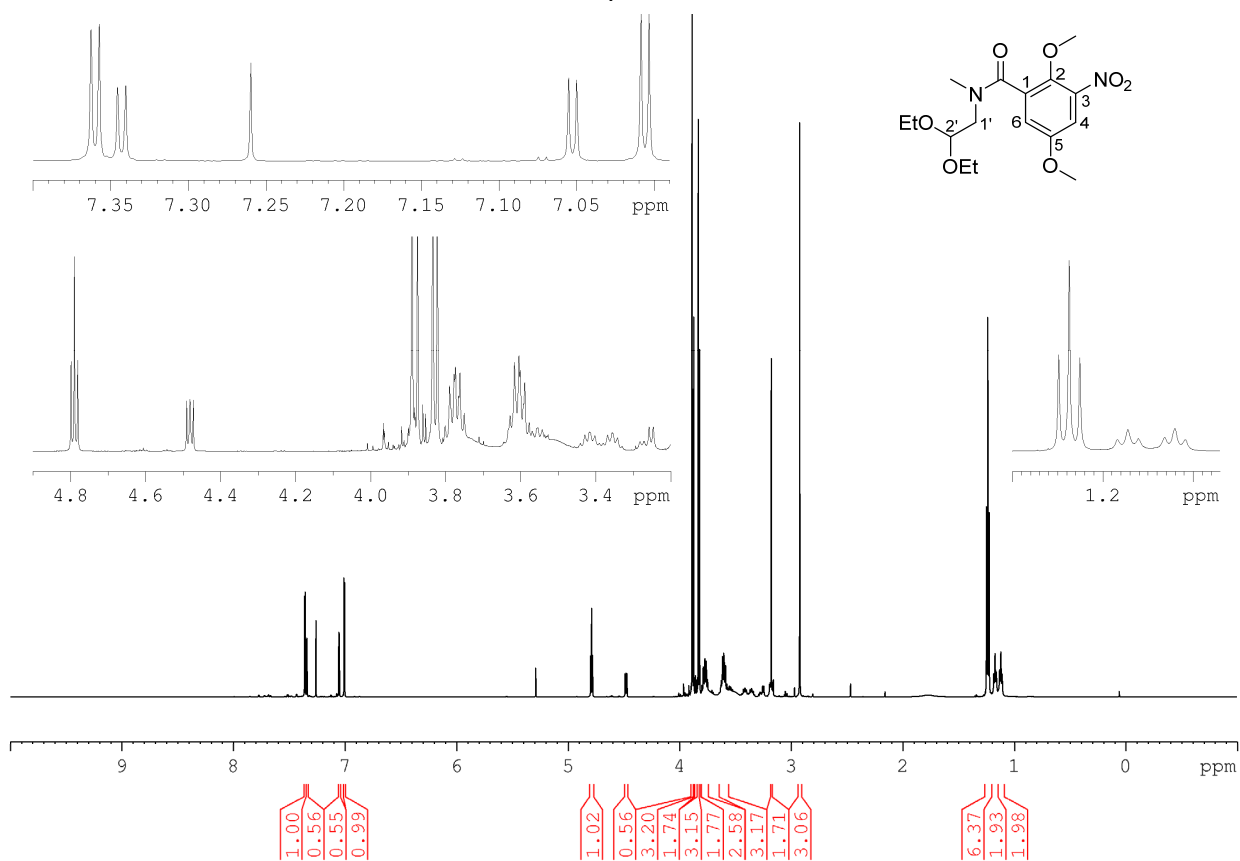
500 MHz ^1H NMR spectrum of **37** in d_6 -DMSO



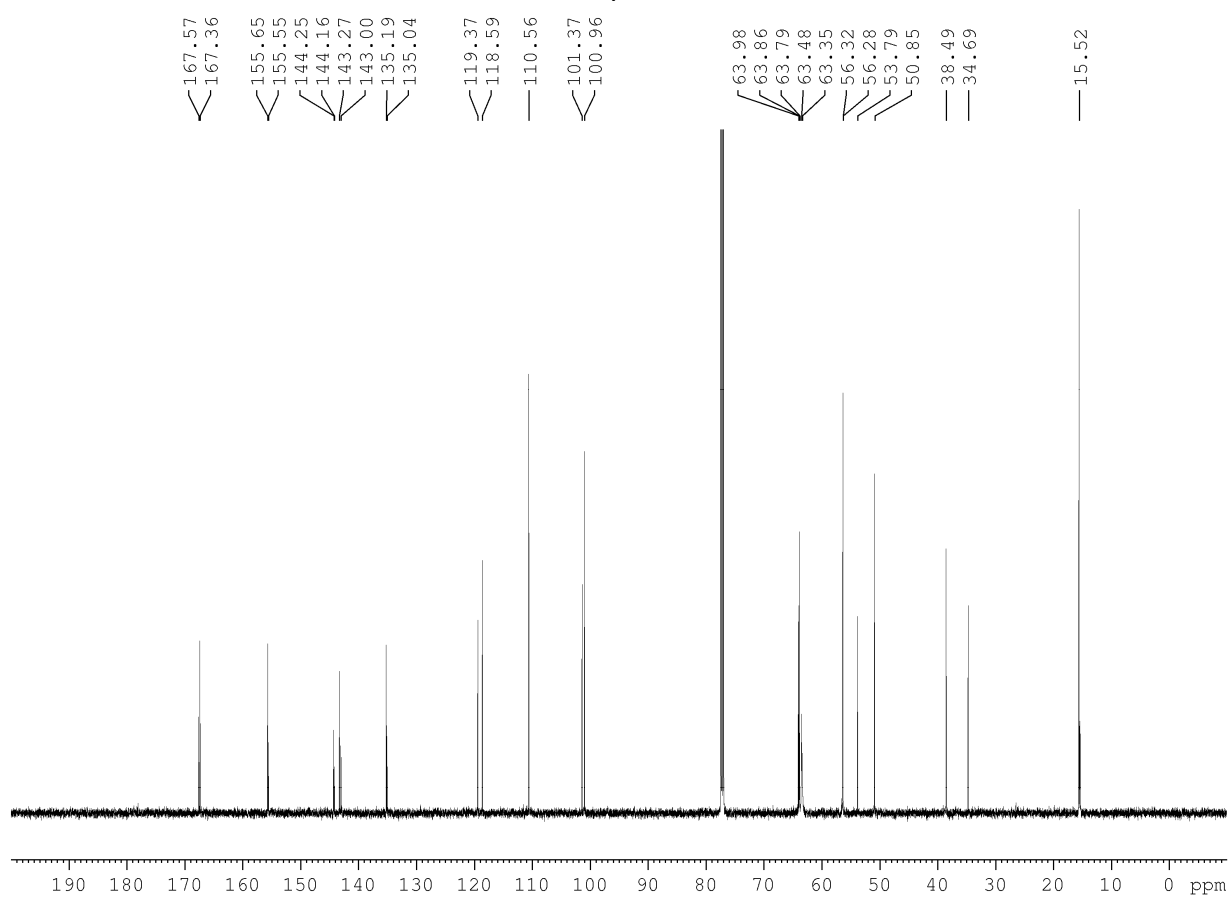
125 MHz ^{13}C NMR spectrum of **37** in d_6 -DMSO



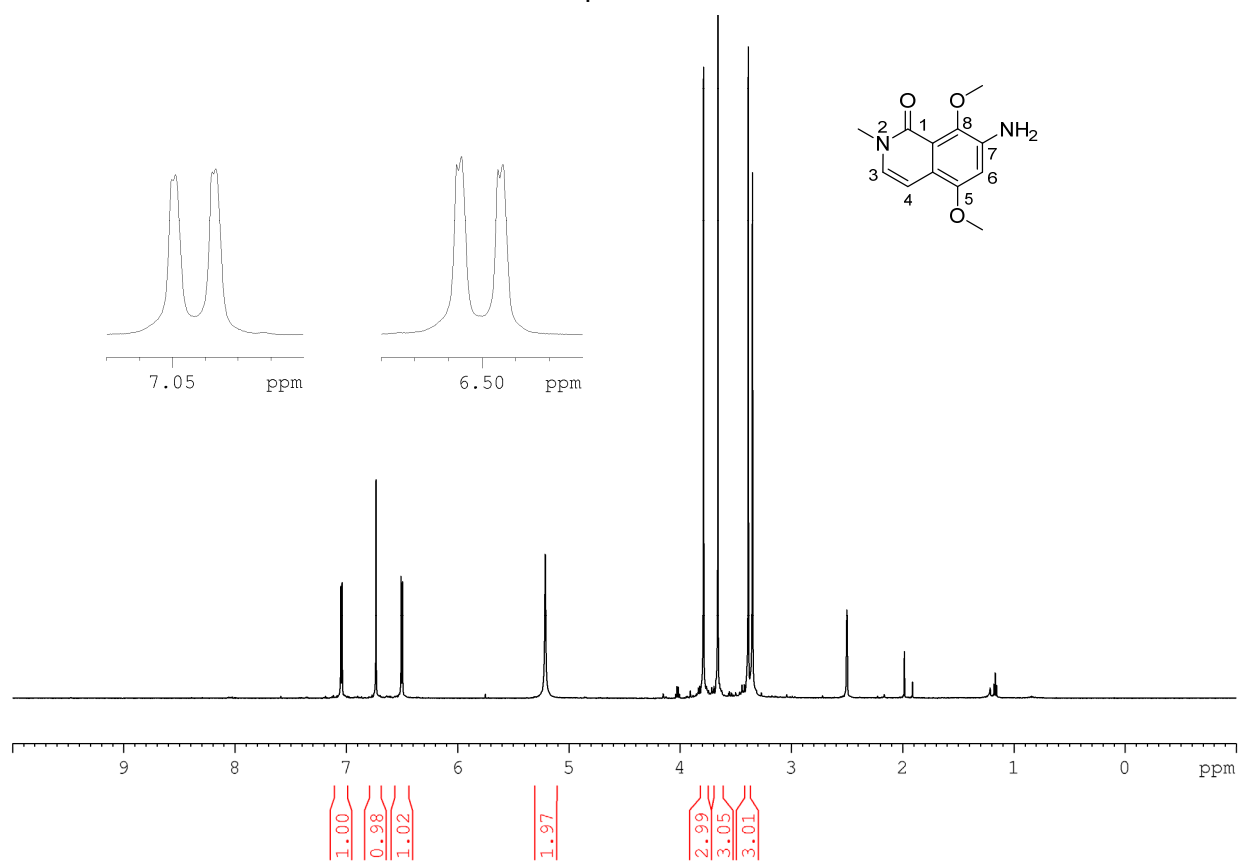
500 MHz ^1H NMR spectrum of **44** in CDCl_3



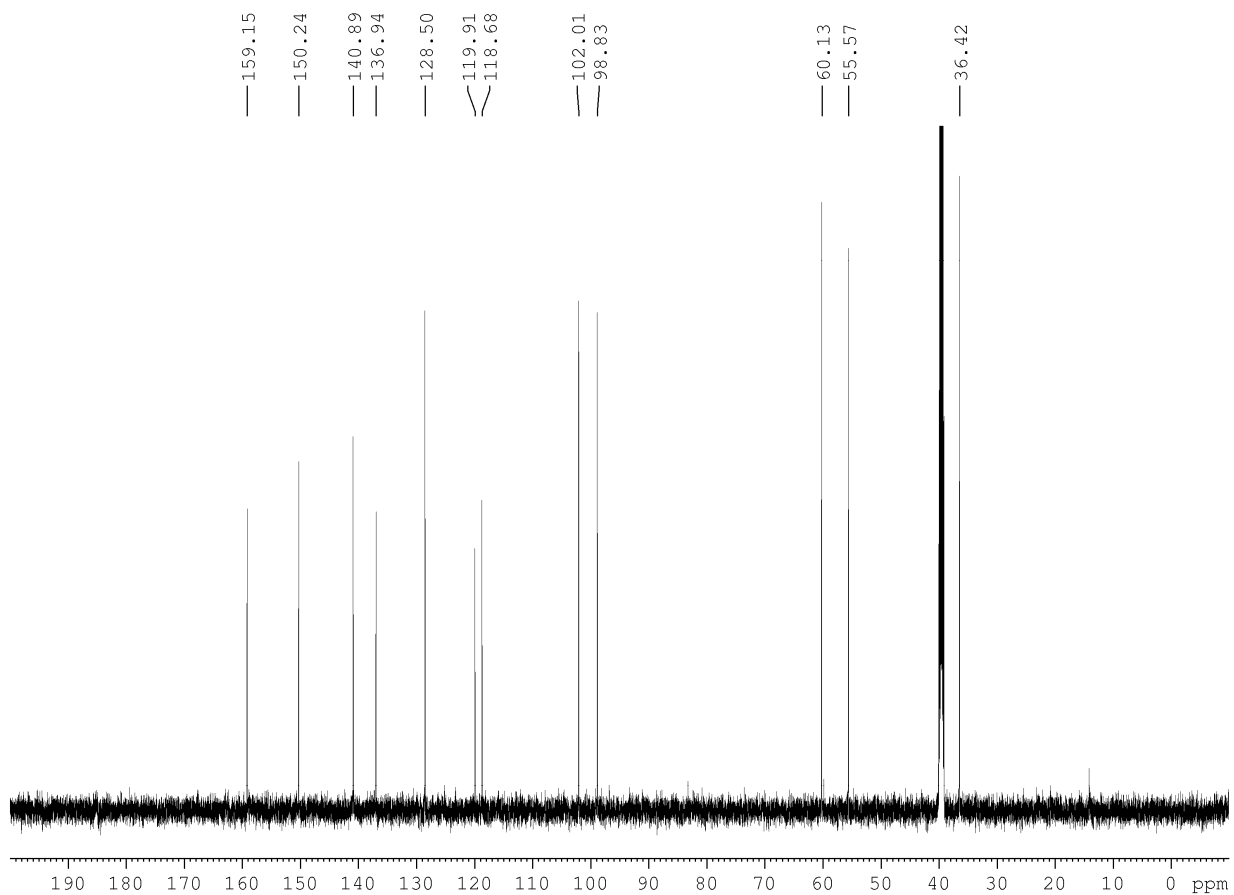
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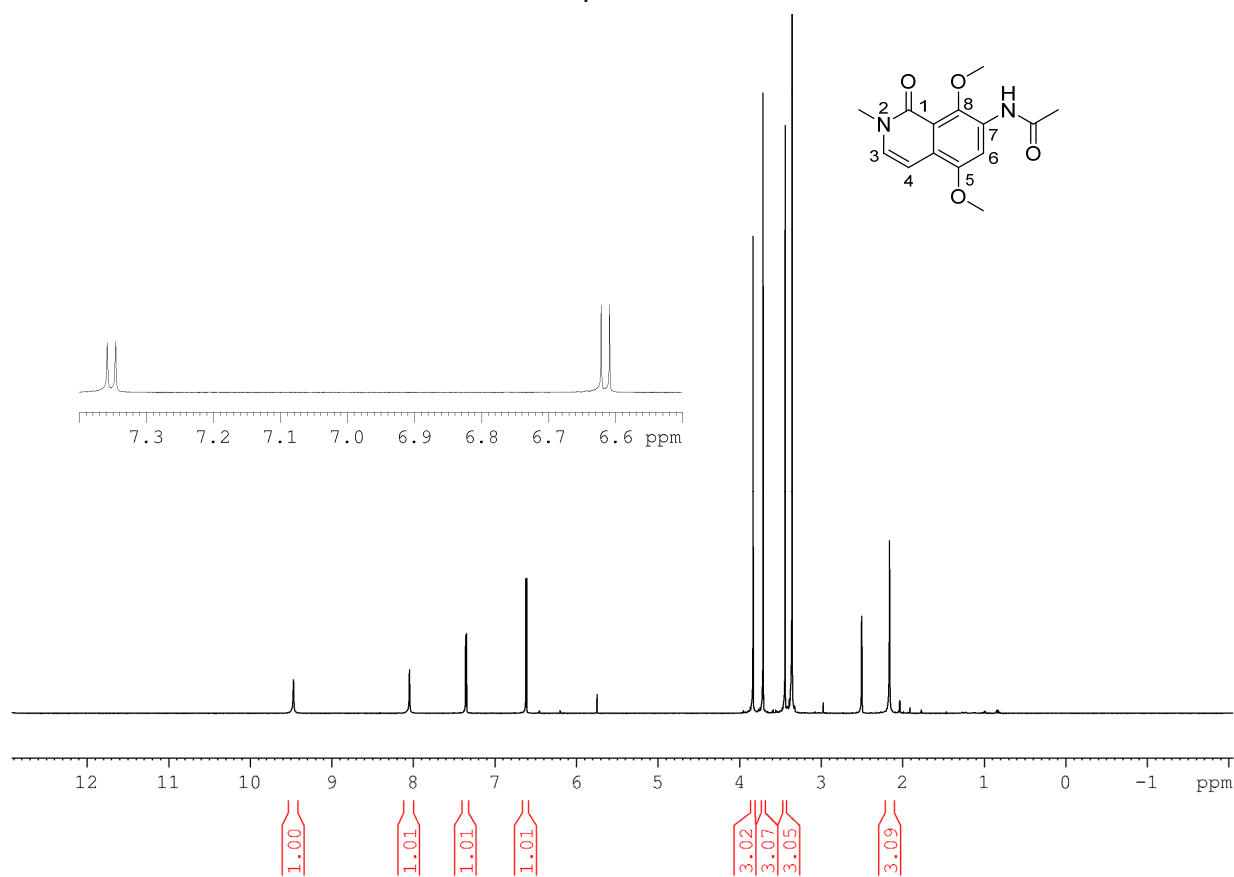
600 MHz ^1H NMR spectrum of **45** in d_6 -DMSO



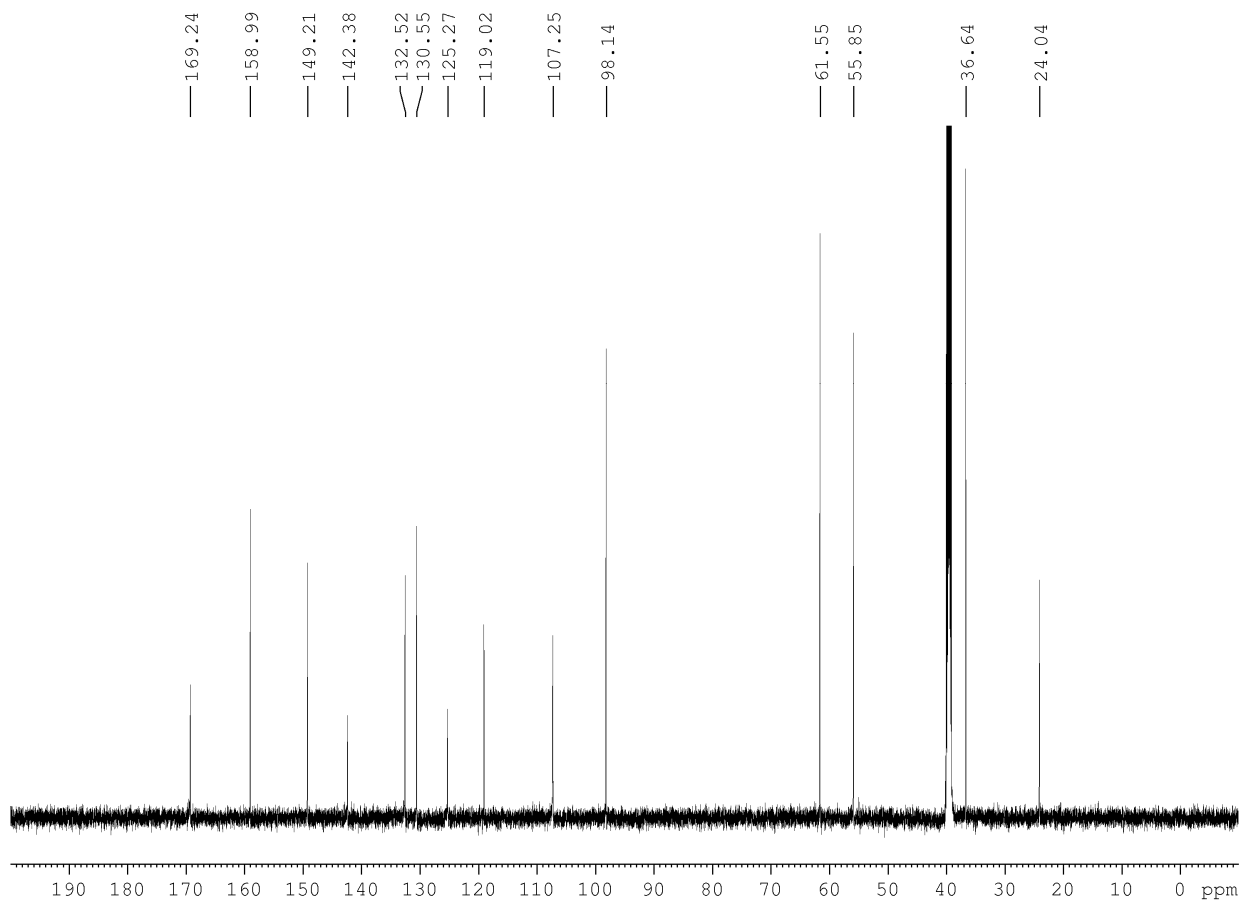
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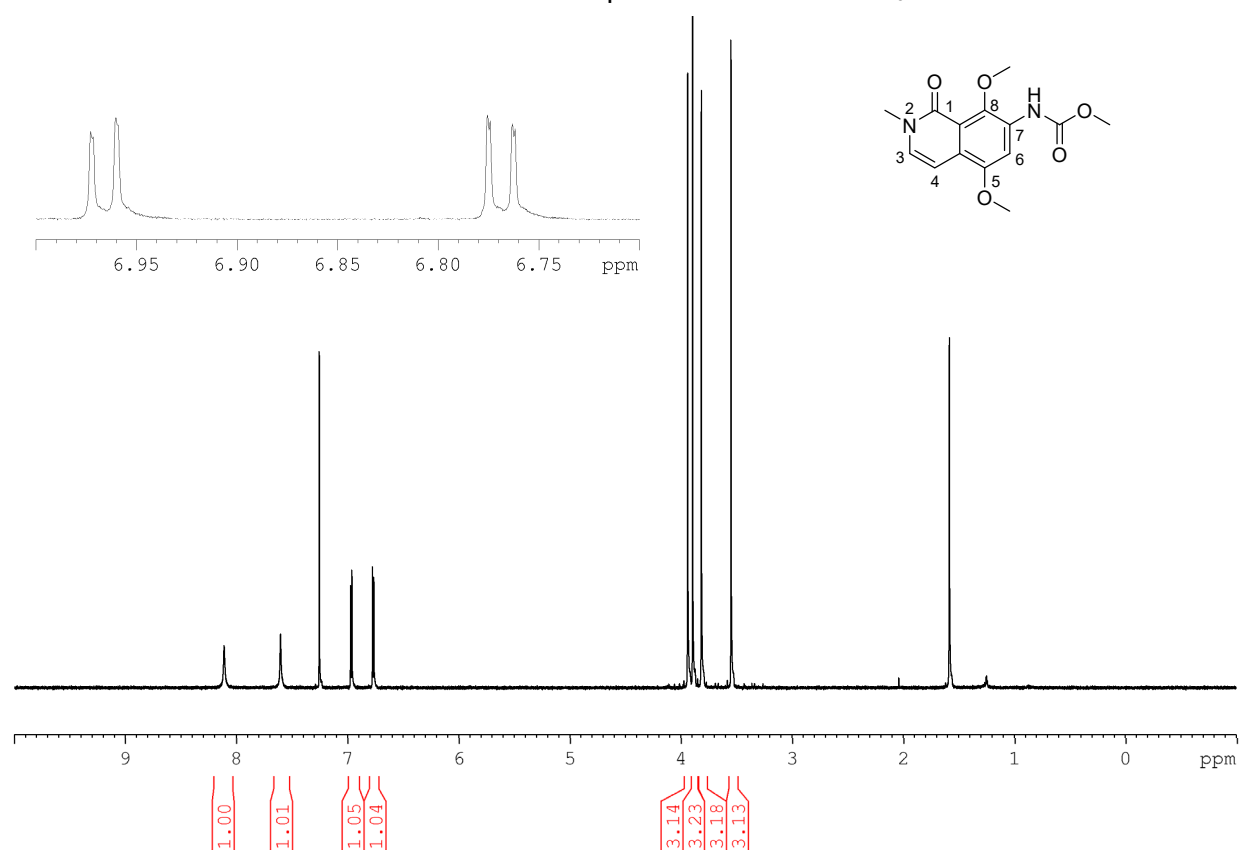
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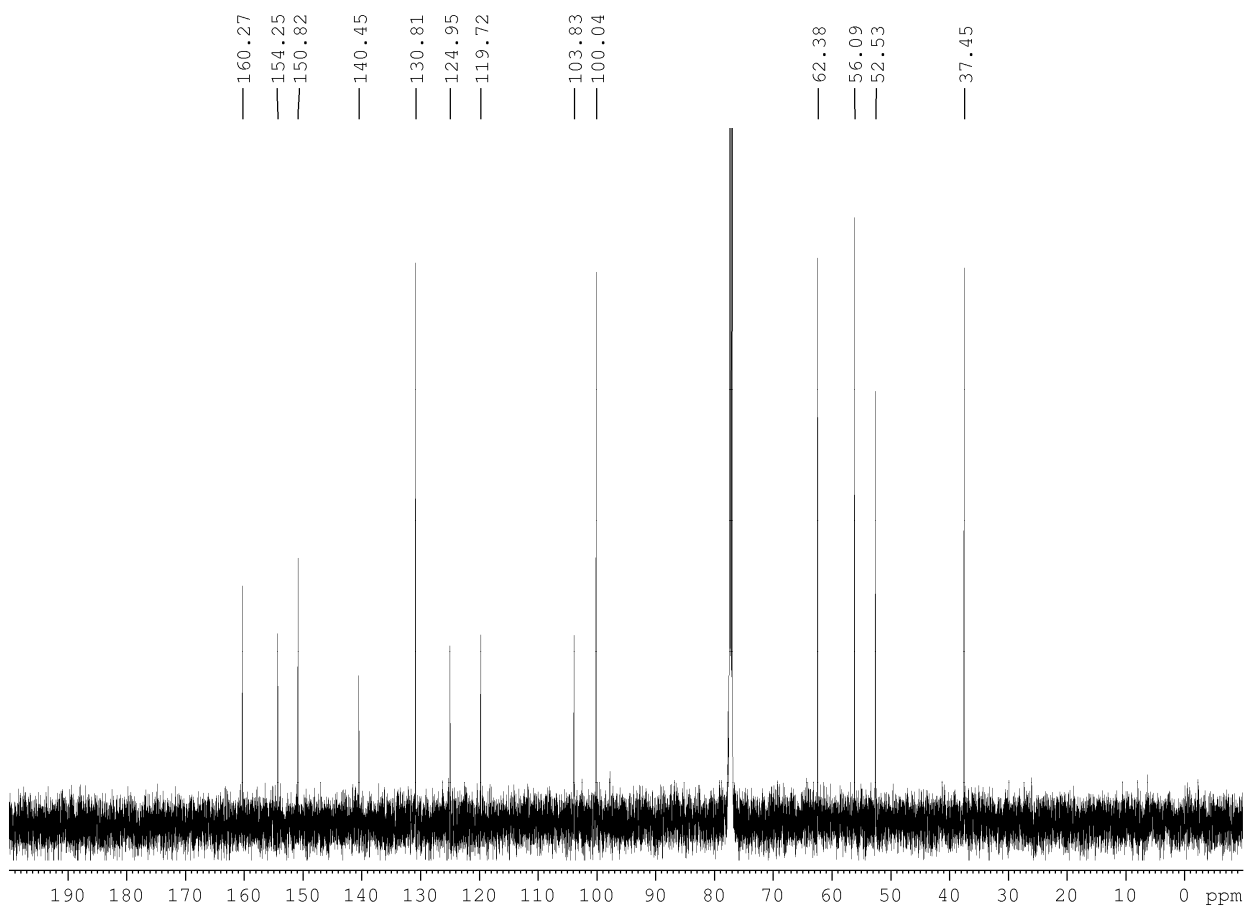
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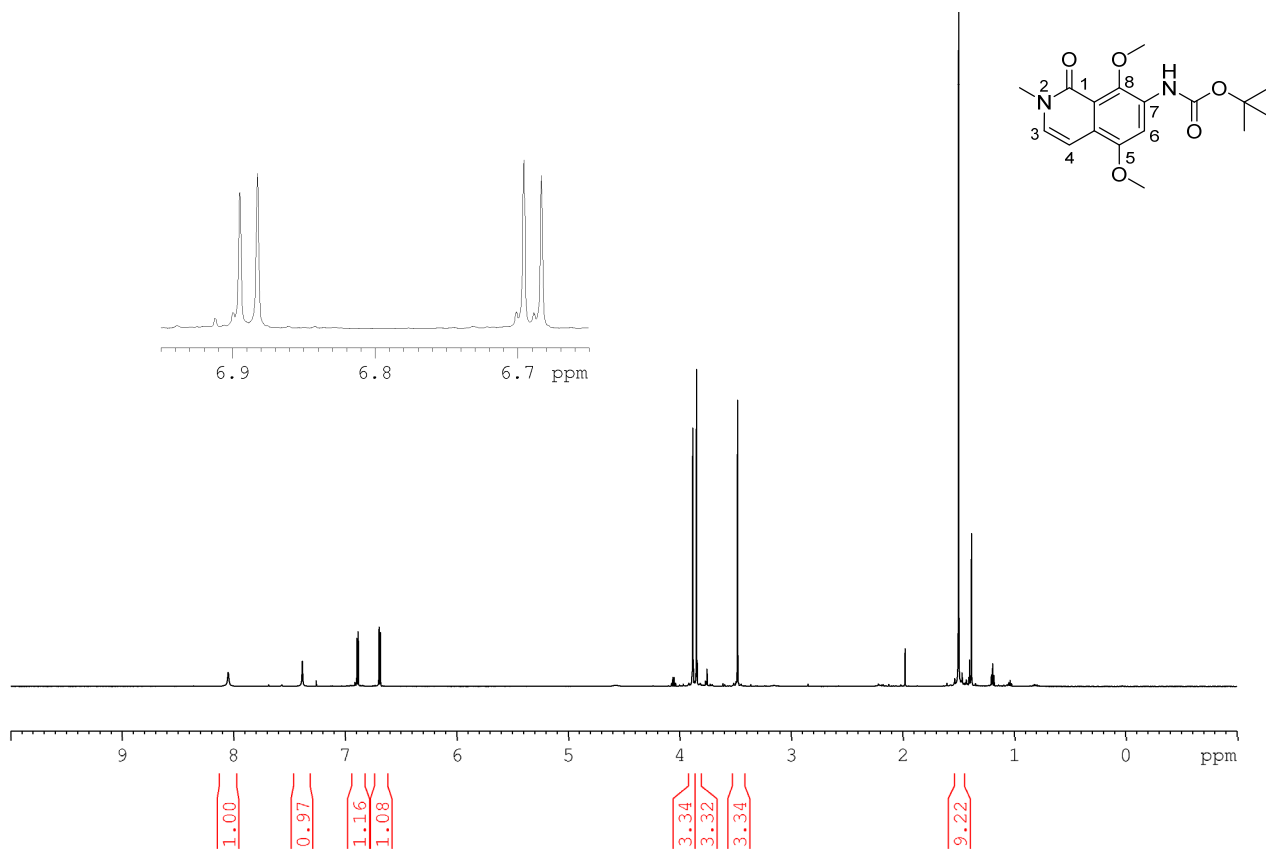
600 MHz ^1H NMR spectrum of **47** in CDCl_3



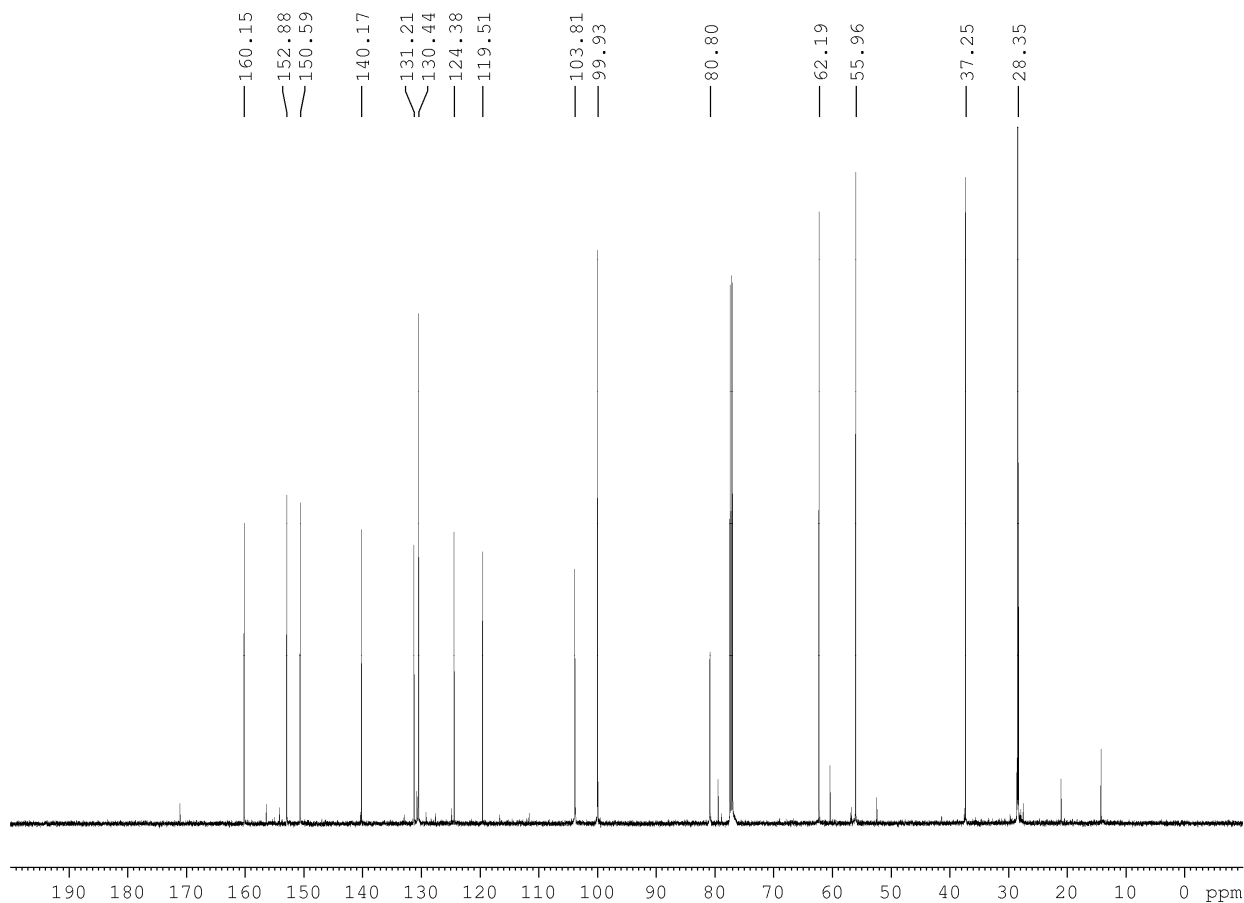
150 MHz ^{13}C NMR spectrum of **47** in CDCl_3



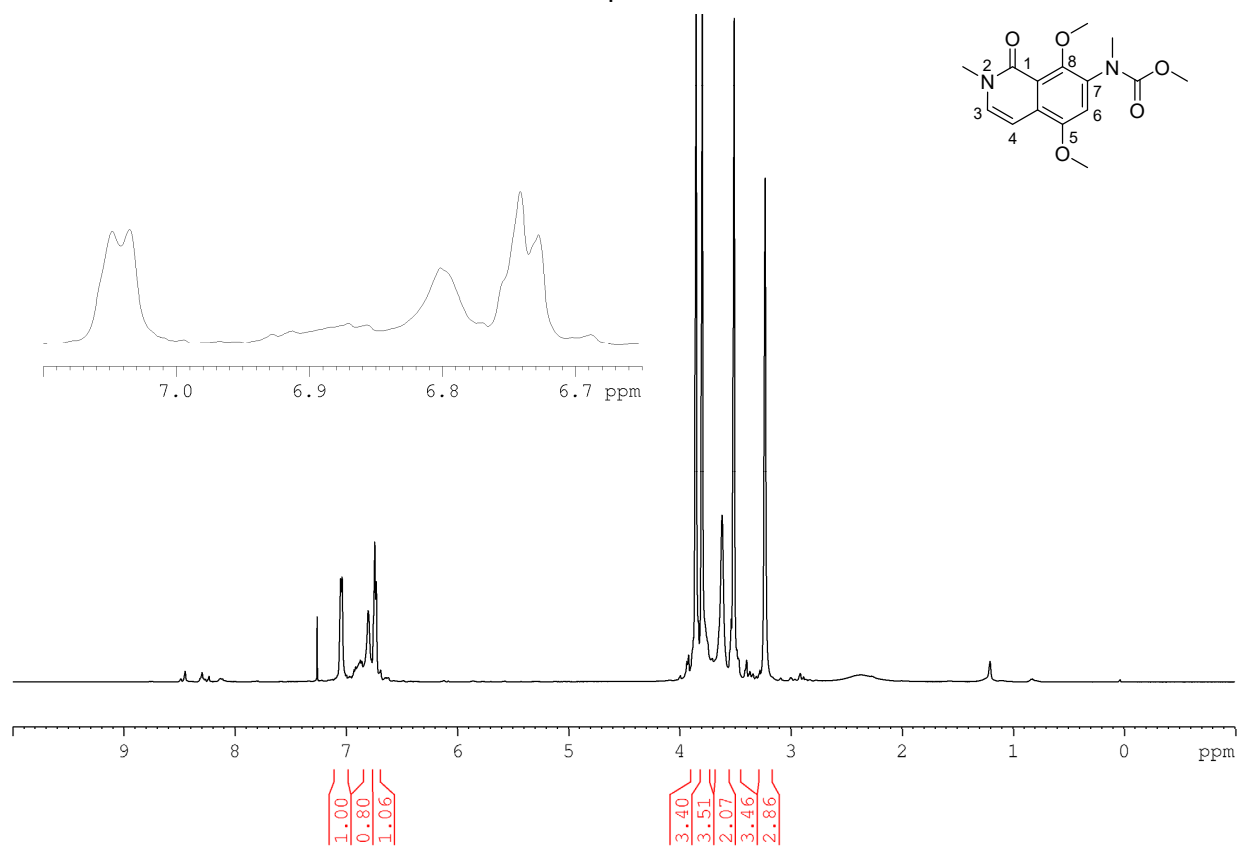
400 MHz ^1H NMR spectrum of **48** in CDCl_3



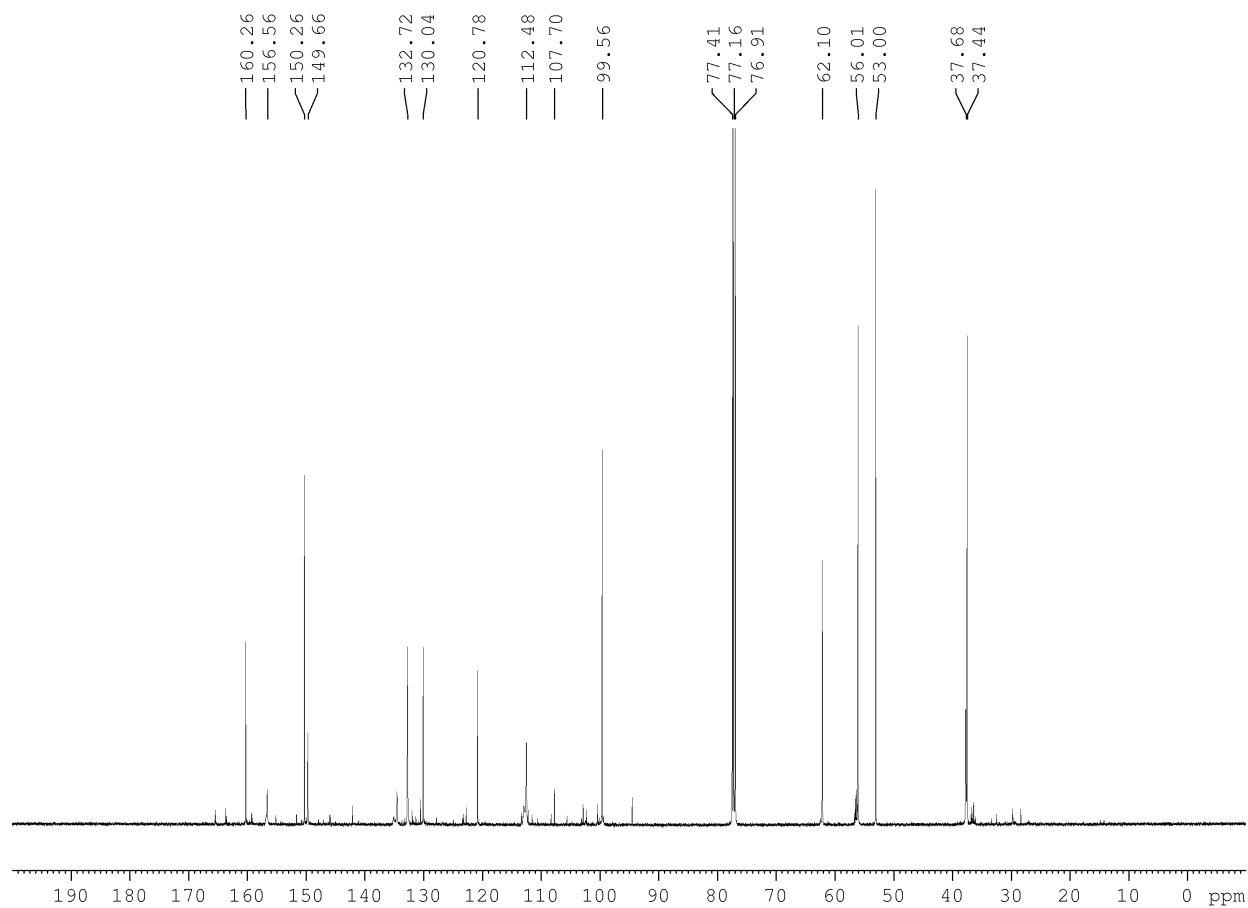
100 MHz ^{13}C NMR spectrum of **48** in CDCl_3



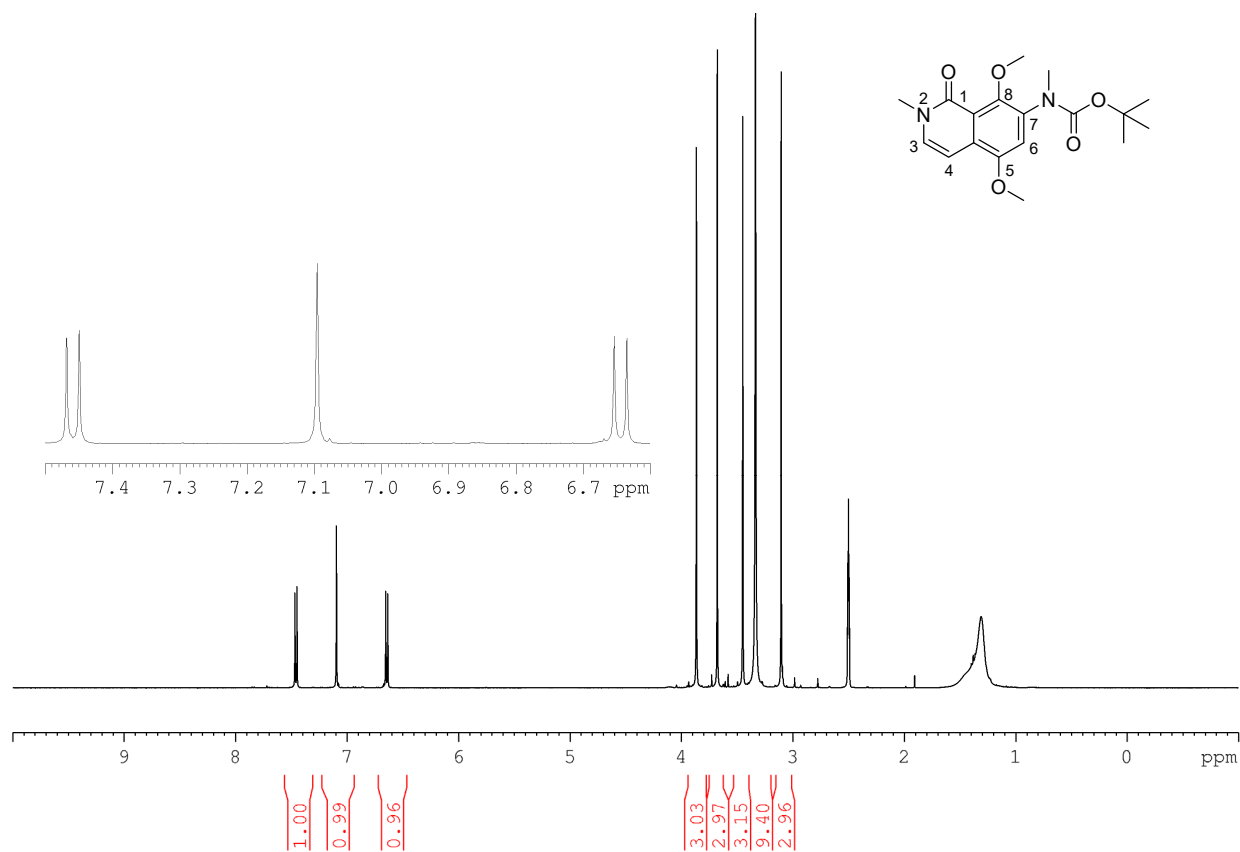
500 MHz ^1H NMR spectrum of **49** in CDCl_3



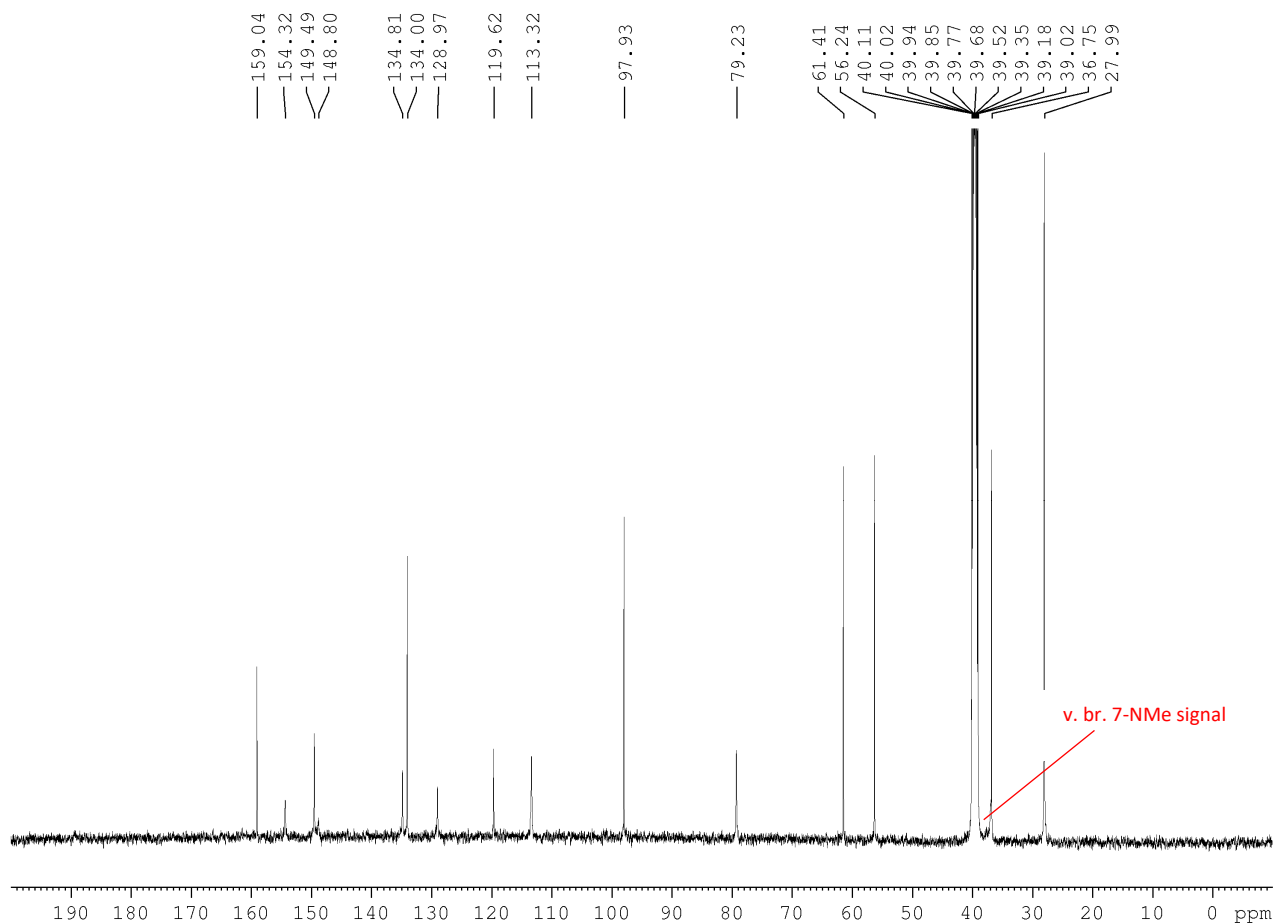
125 MHz ^{13}C NMR spectrum of **49** in CDCl_3



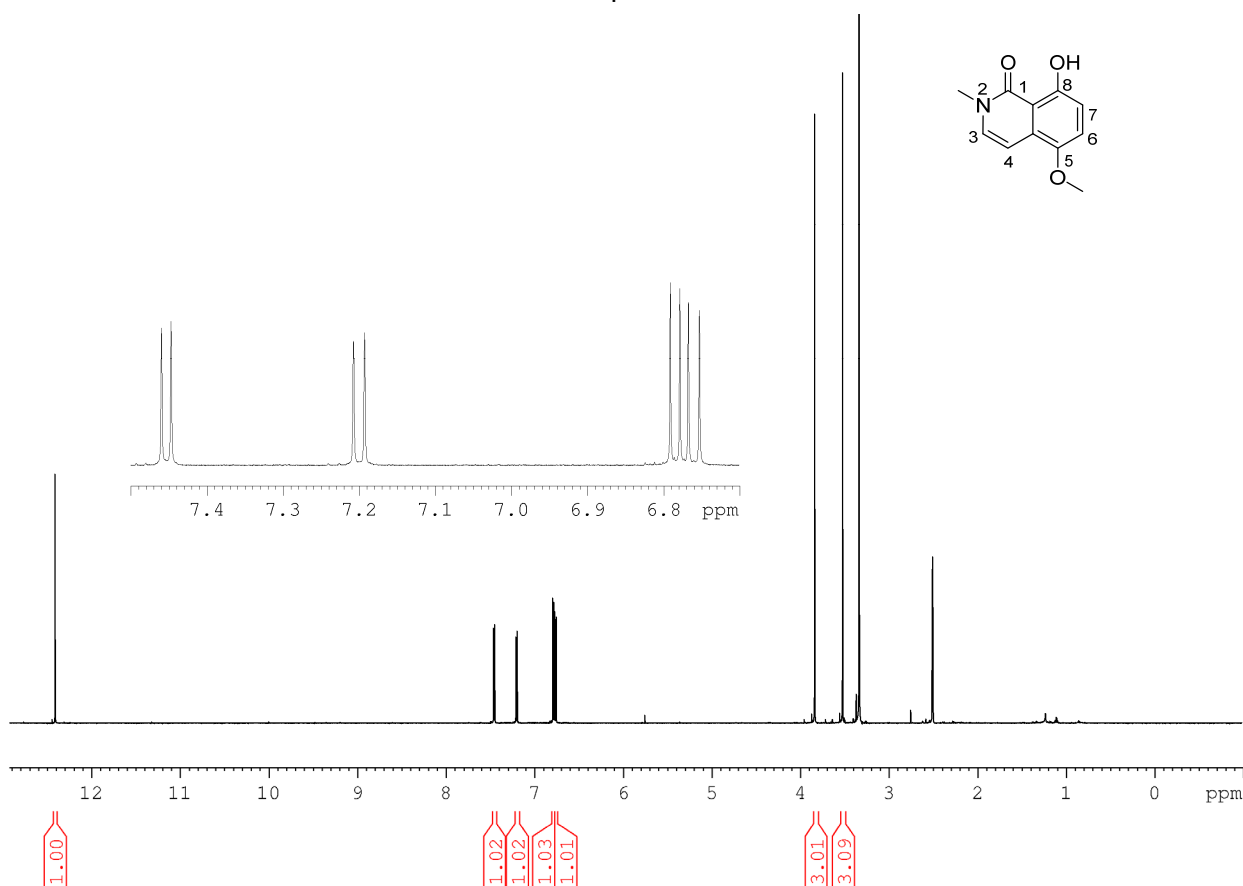
400 MHz ^1H NMR spectrum of **50** in d_6 -DMSO



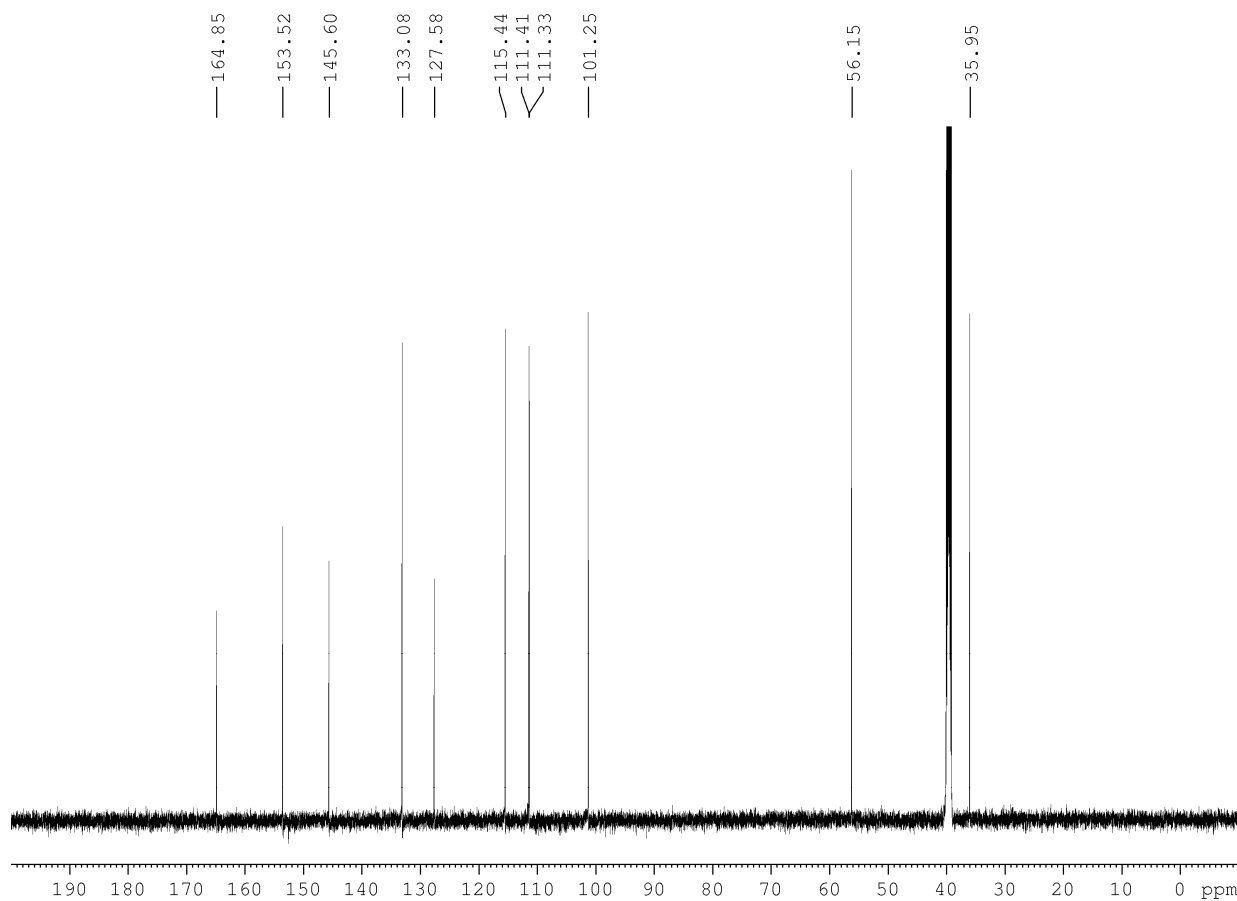
125 MHz ^{13}C NMR spectrum of **50** in d_6 -DMSO



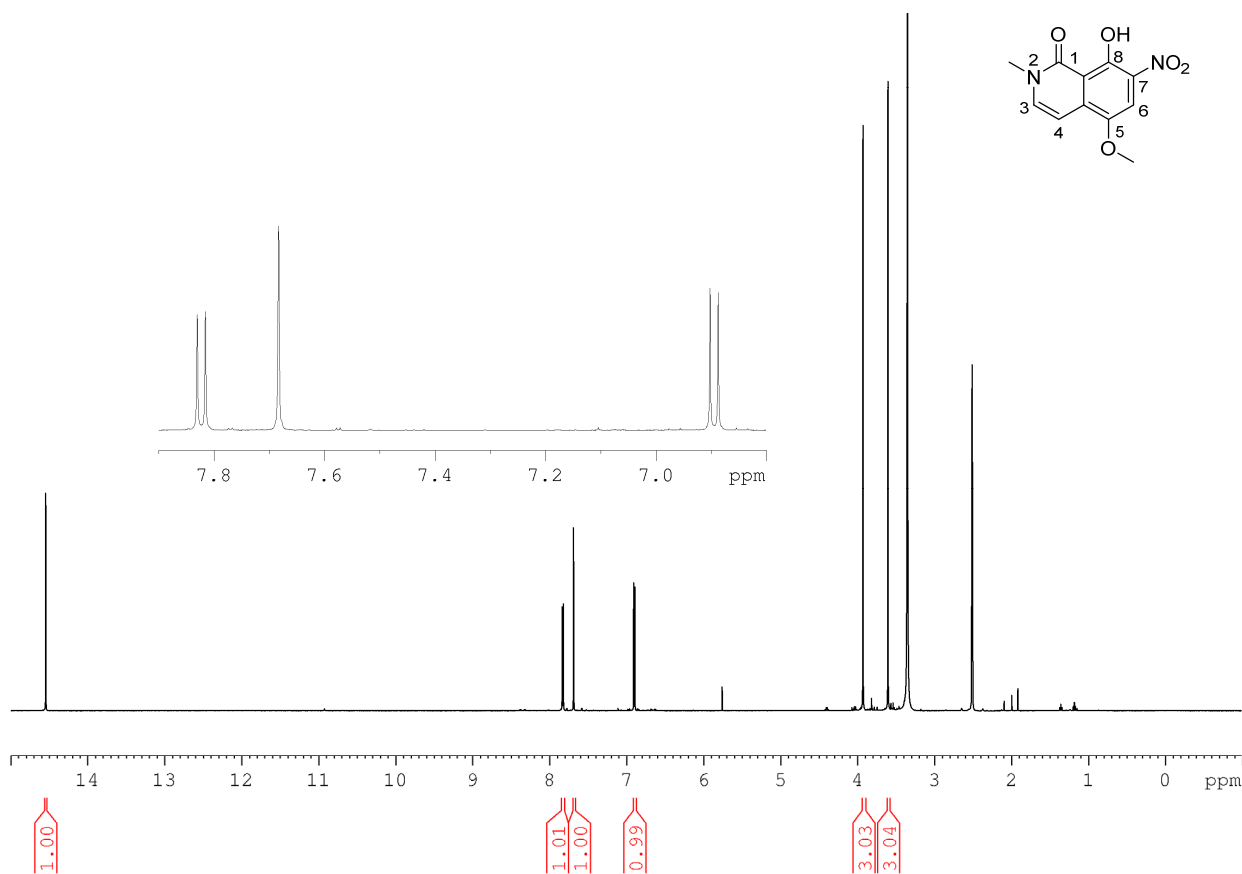
400 MHz ^1H NMR spectrum of **51** in CDCl_3



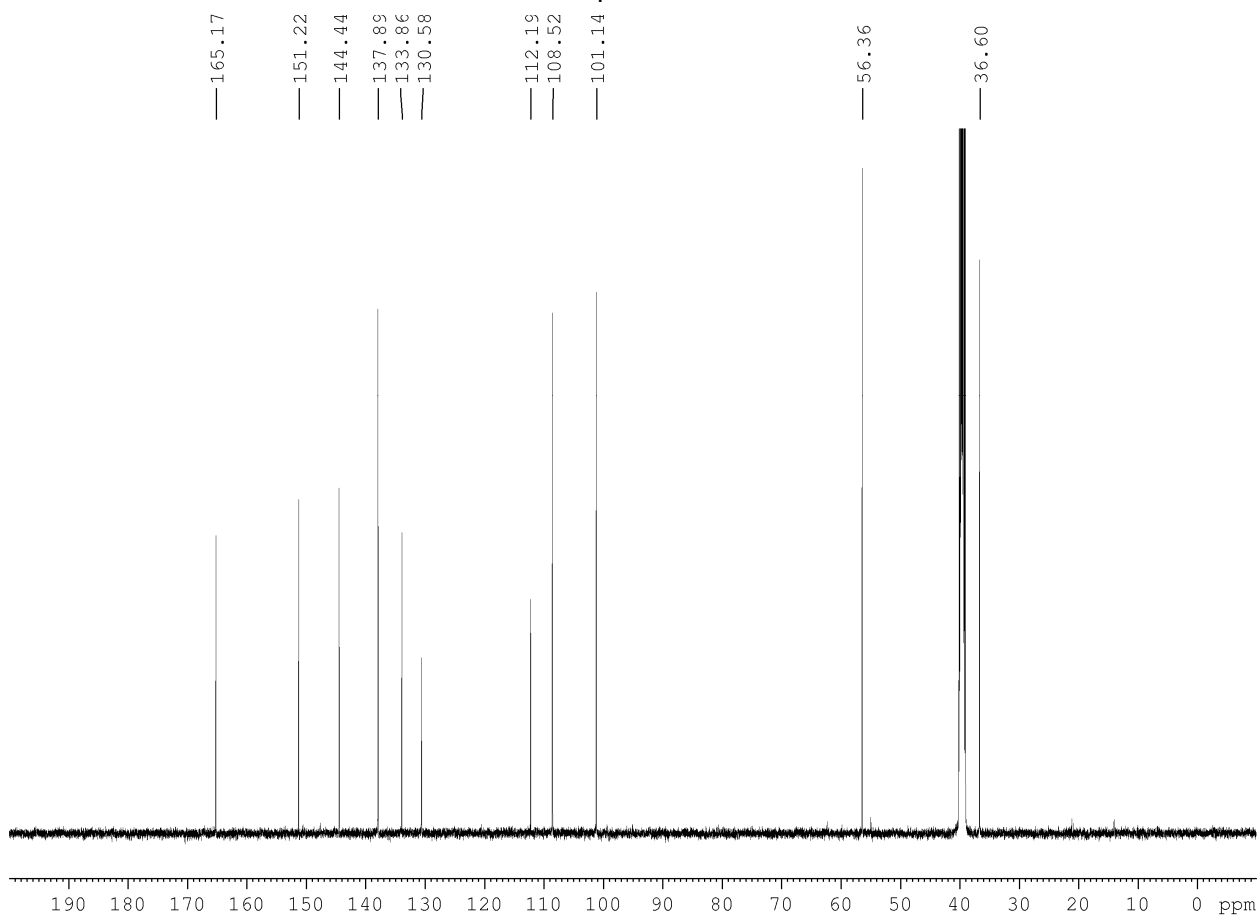
100 MHz ^{13}C NMR spectrum of **51** in CDCl_3



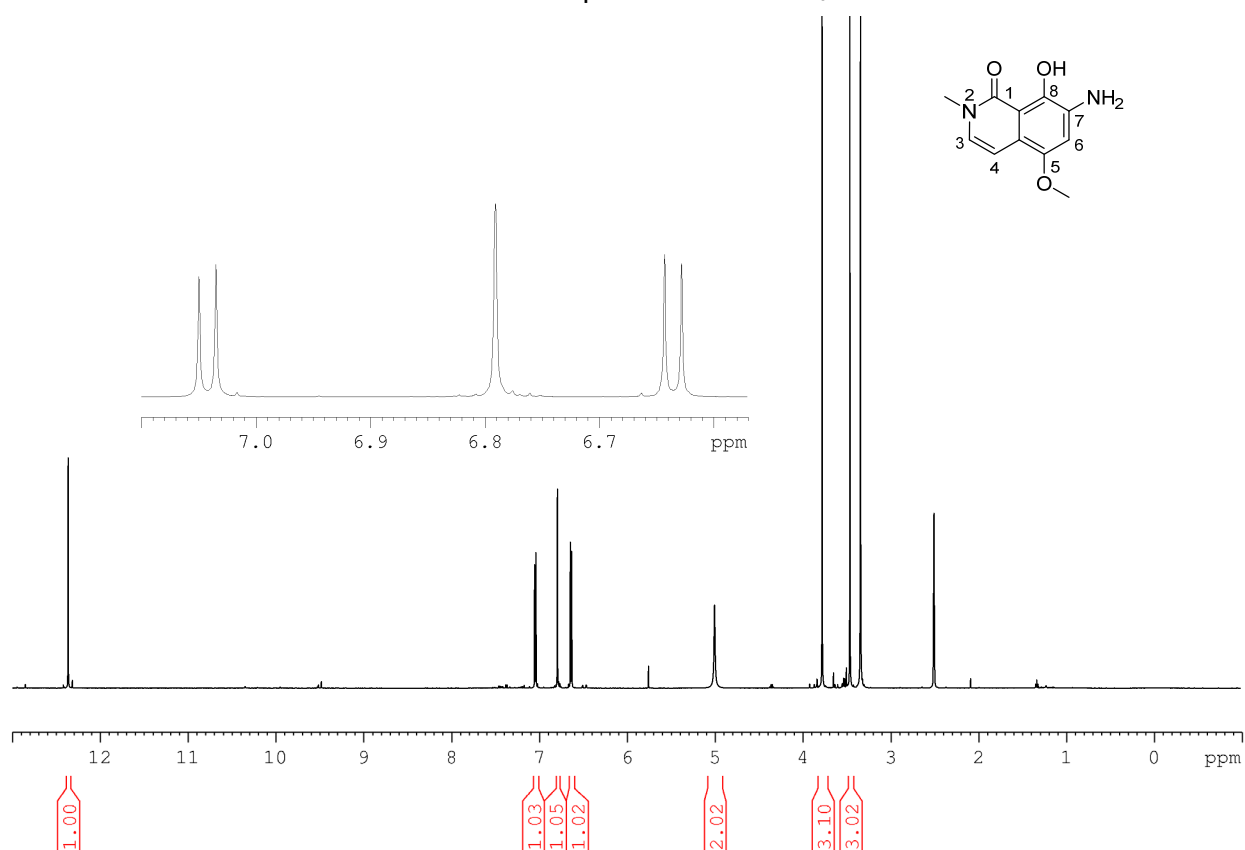
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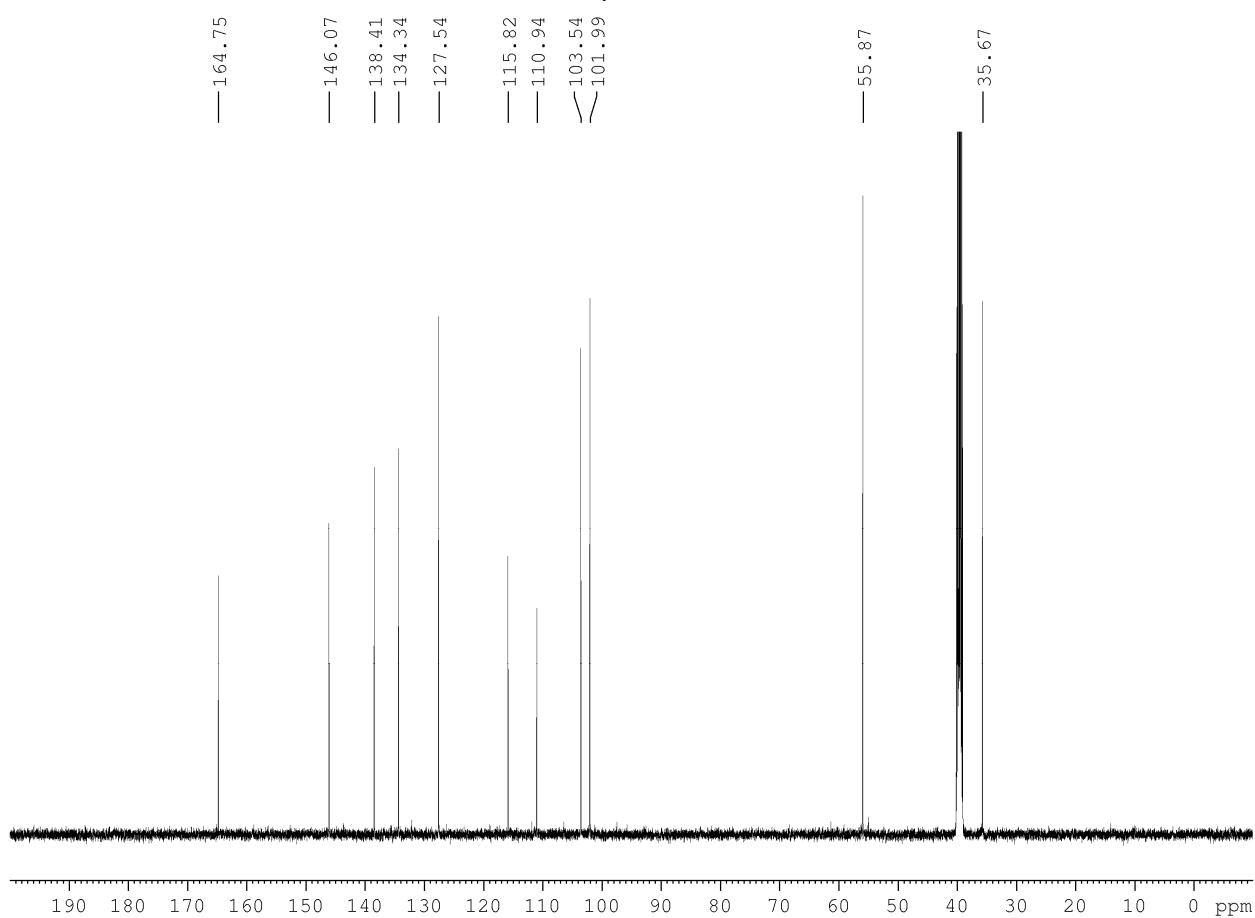
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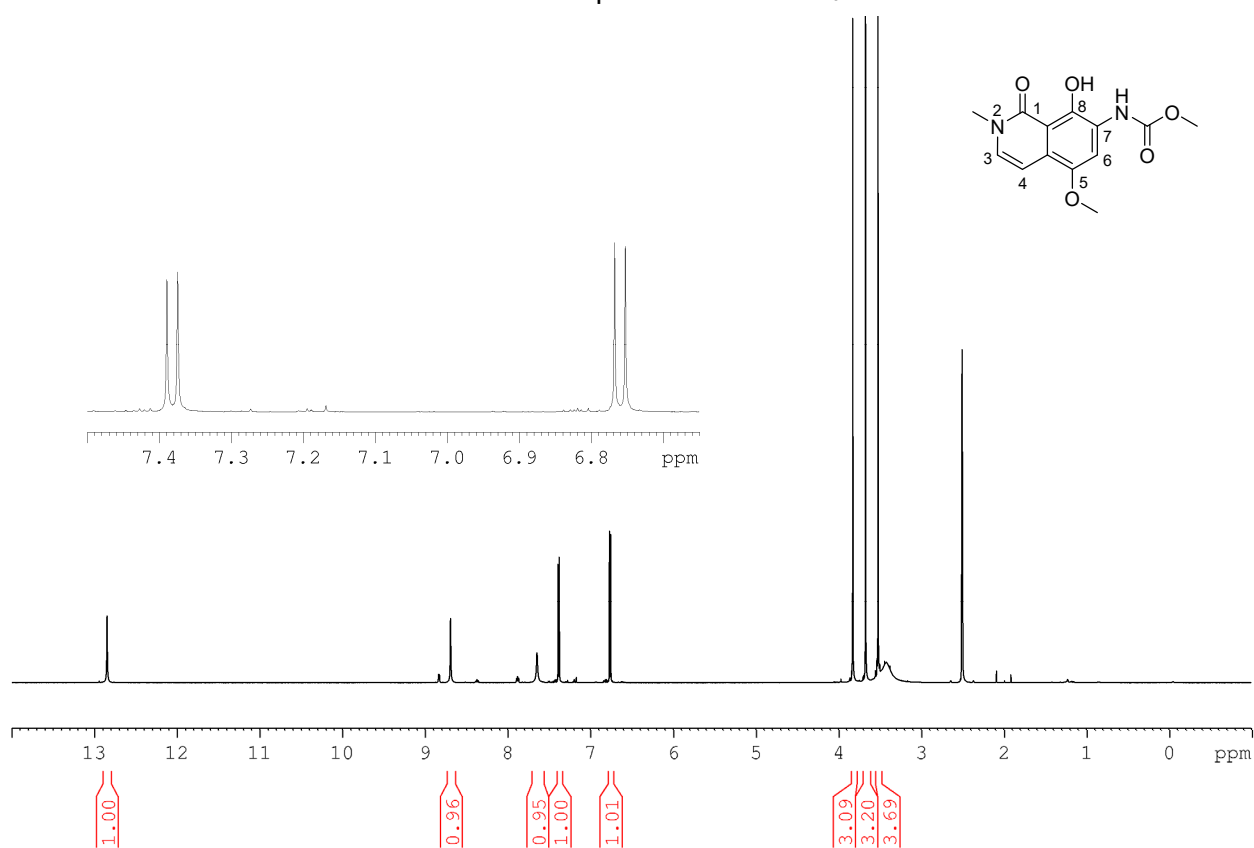
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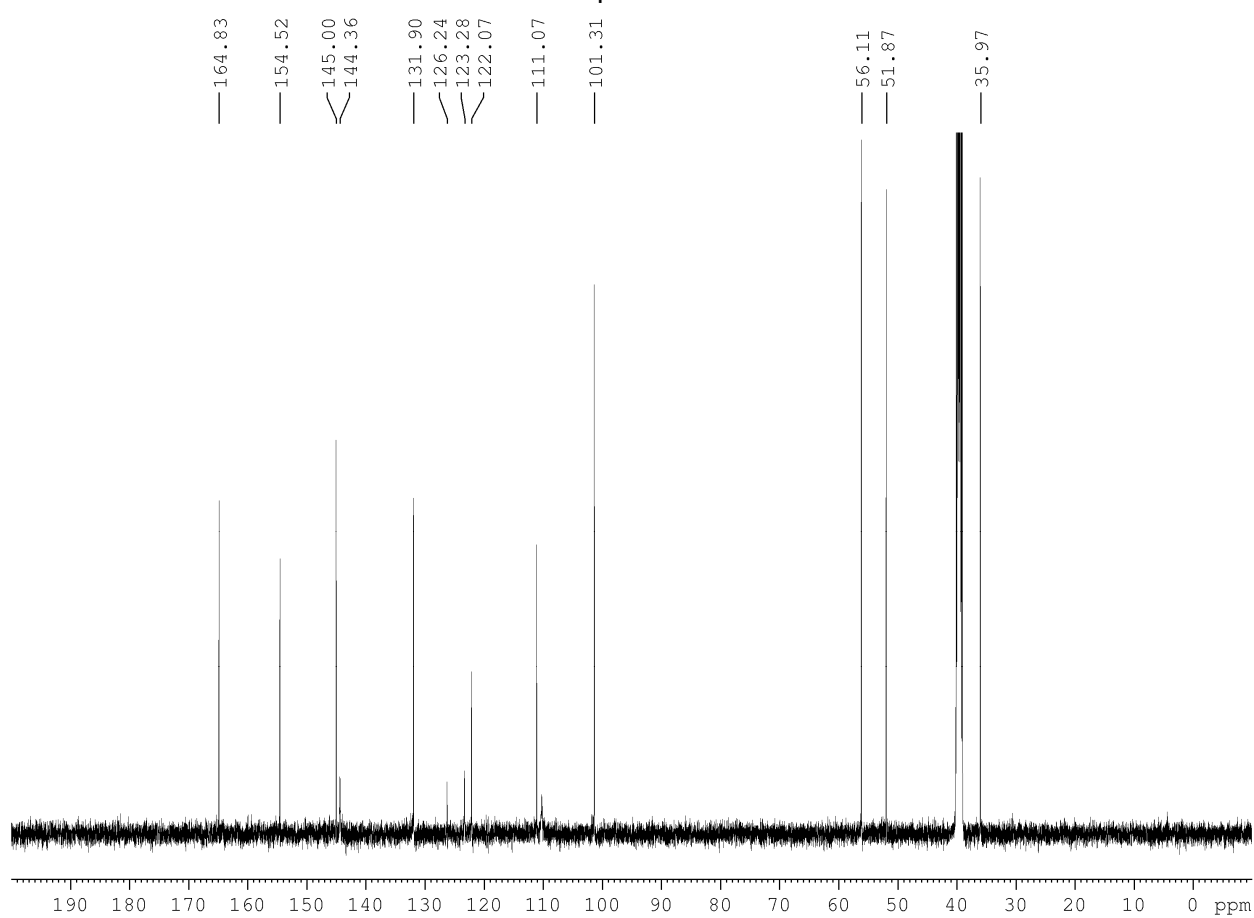
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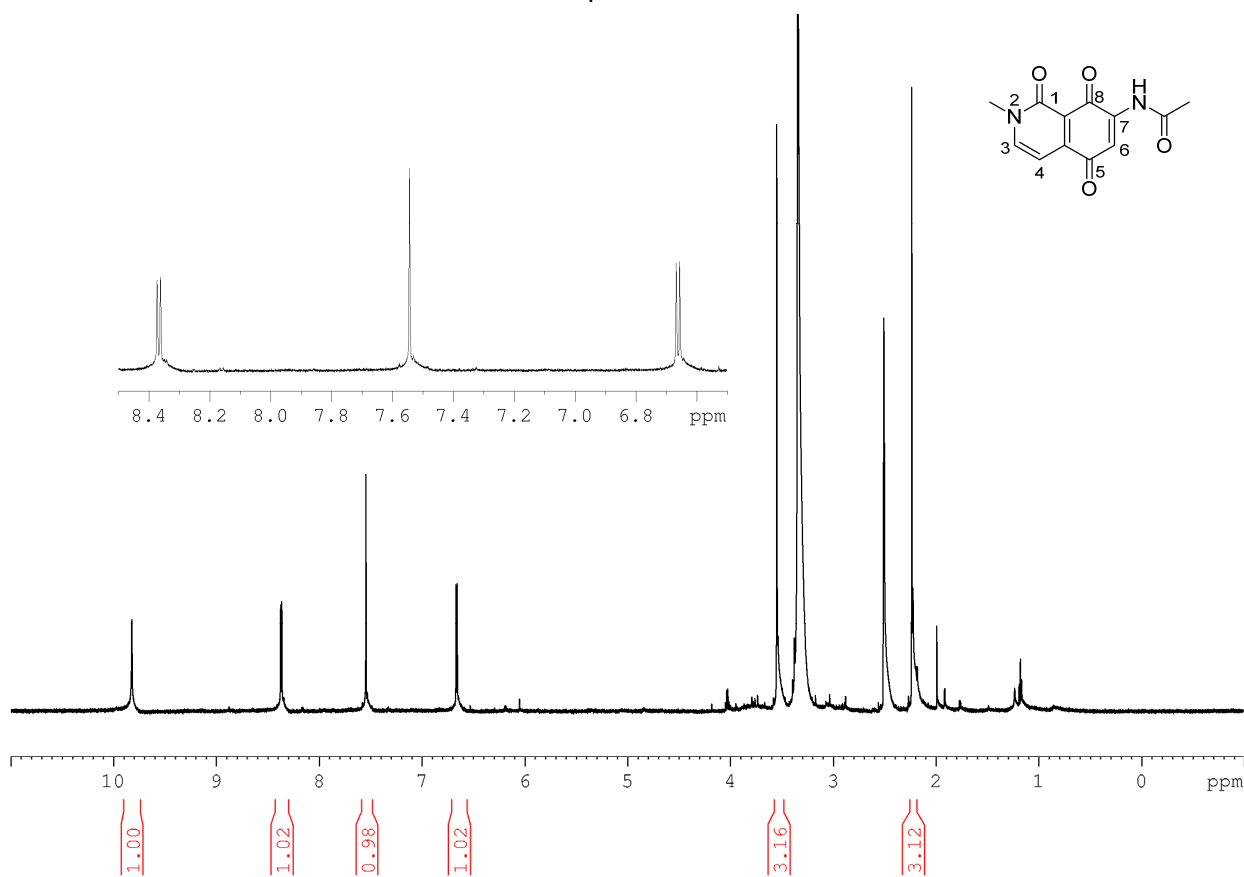
500 MHz ^1H NMR spectrum of **54** in d_6 -DMSO



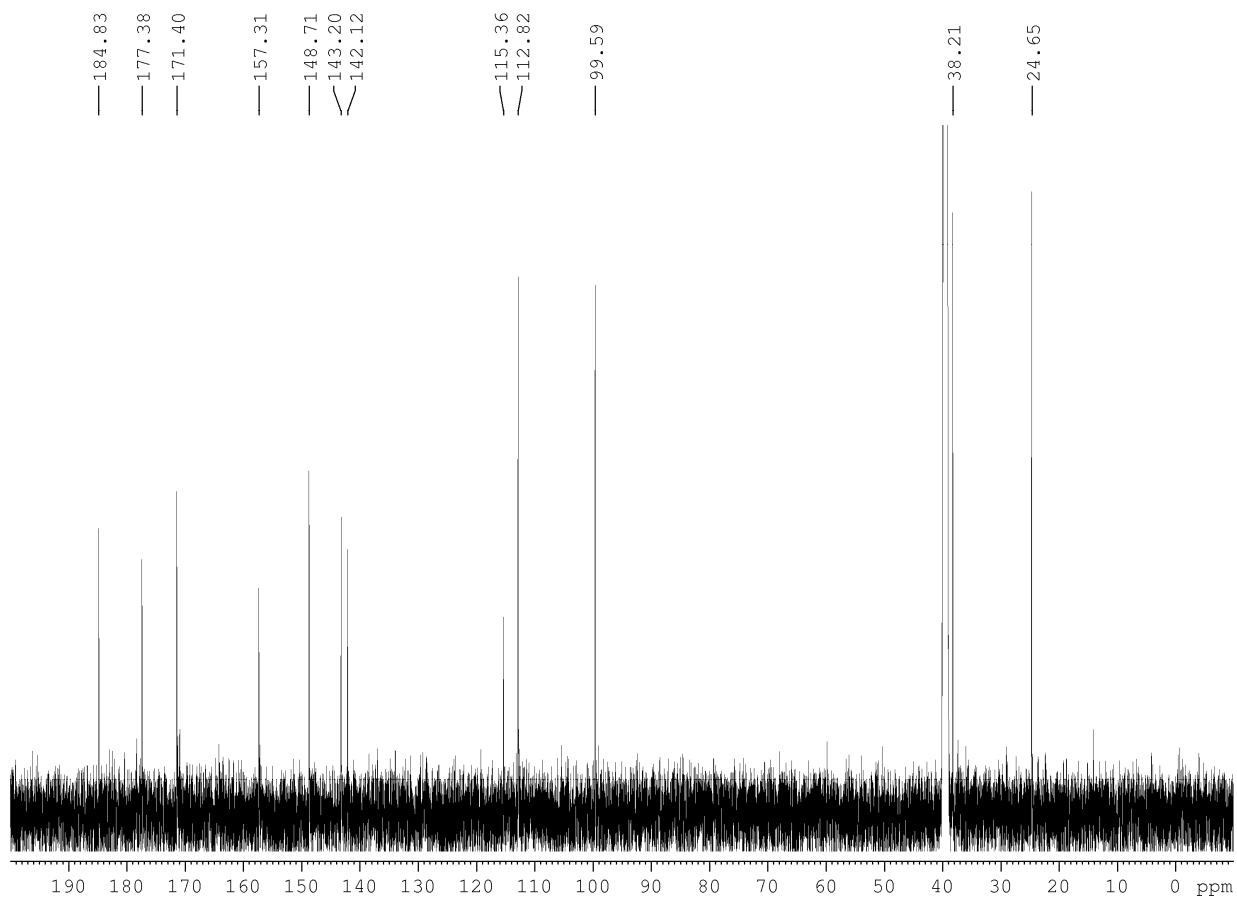
125 MHz ^{13}C NMR spectrum of **54** in d_6 -DMSO



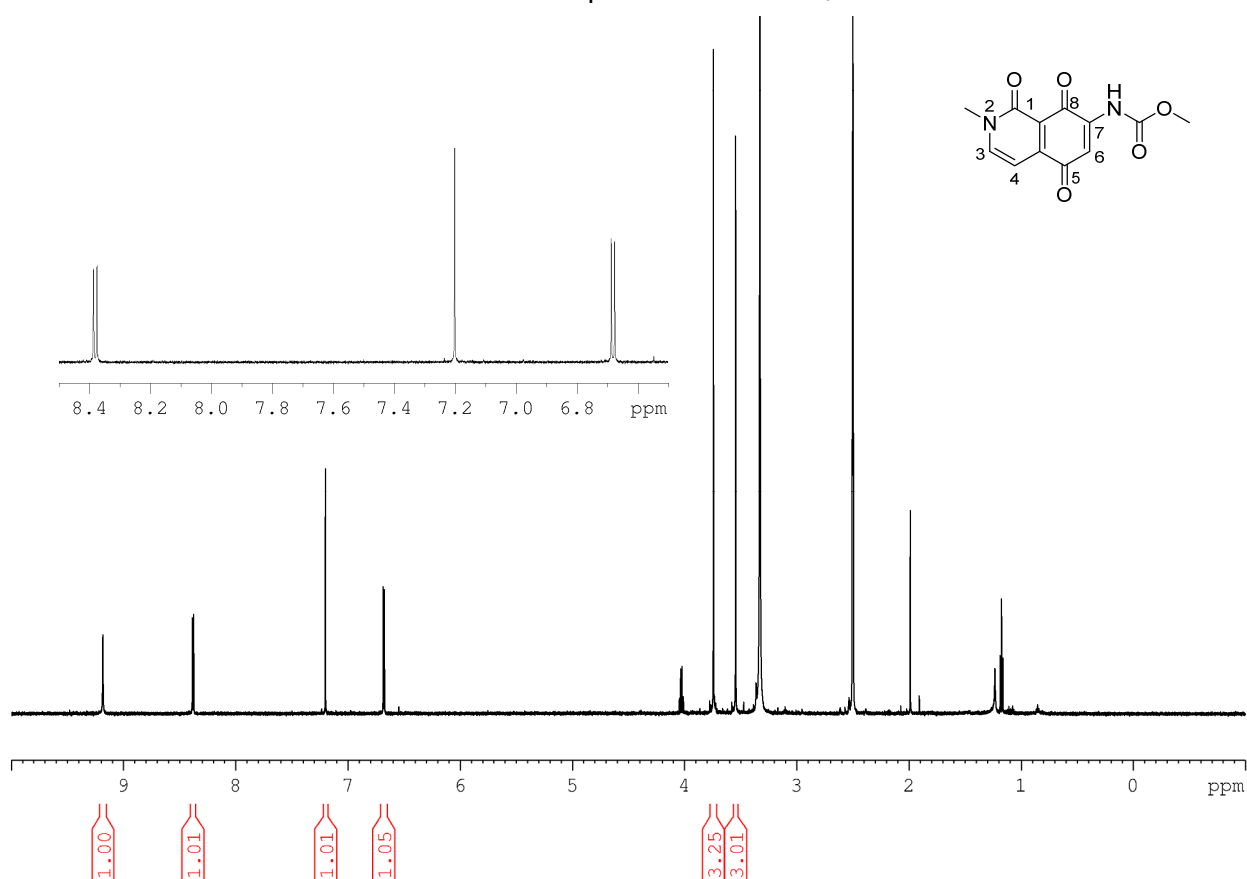
600 MHz ^1H NMR spectrum of **56** in d_6 -DMSO



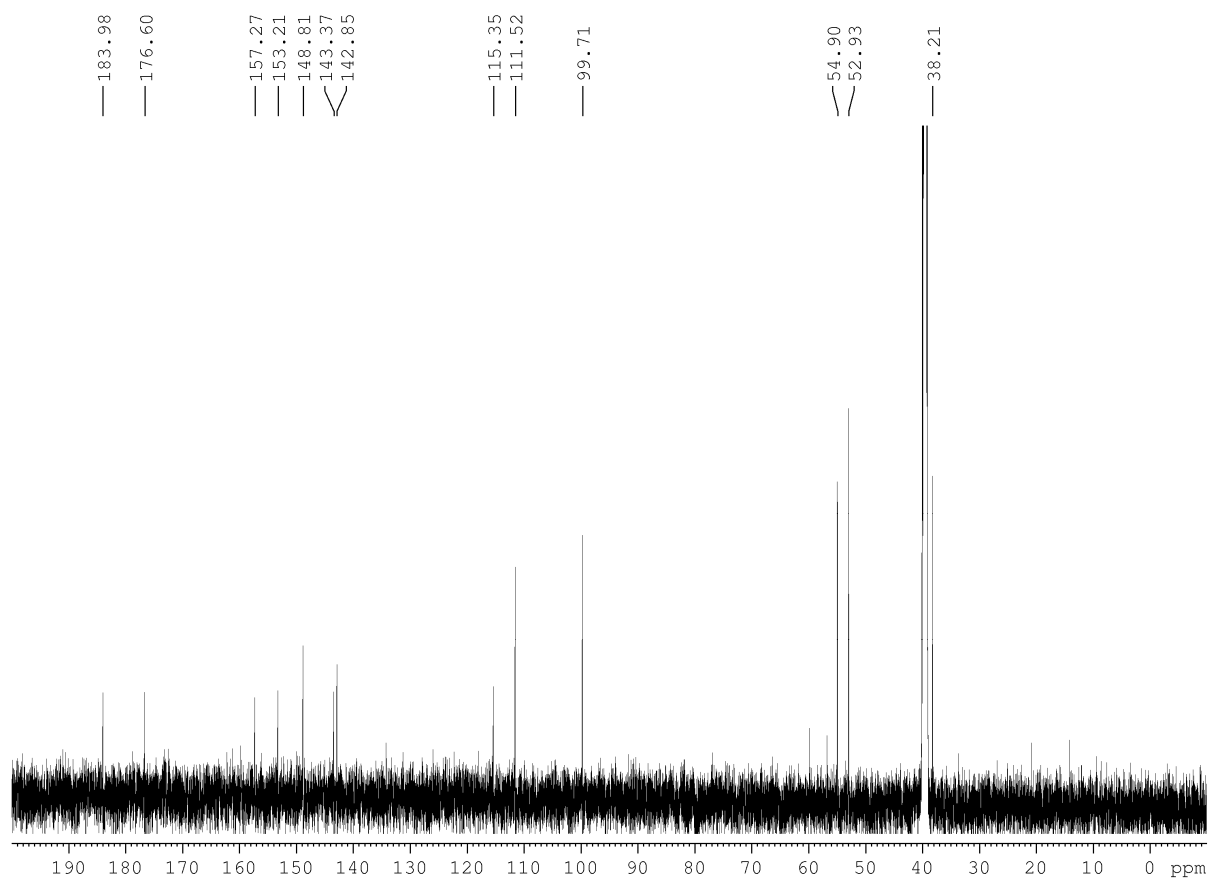
150 MHz ^{13}C NMR spectrum of **56** in d_6 -DMSO



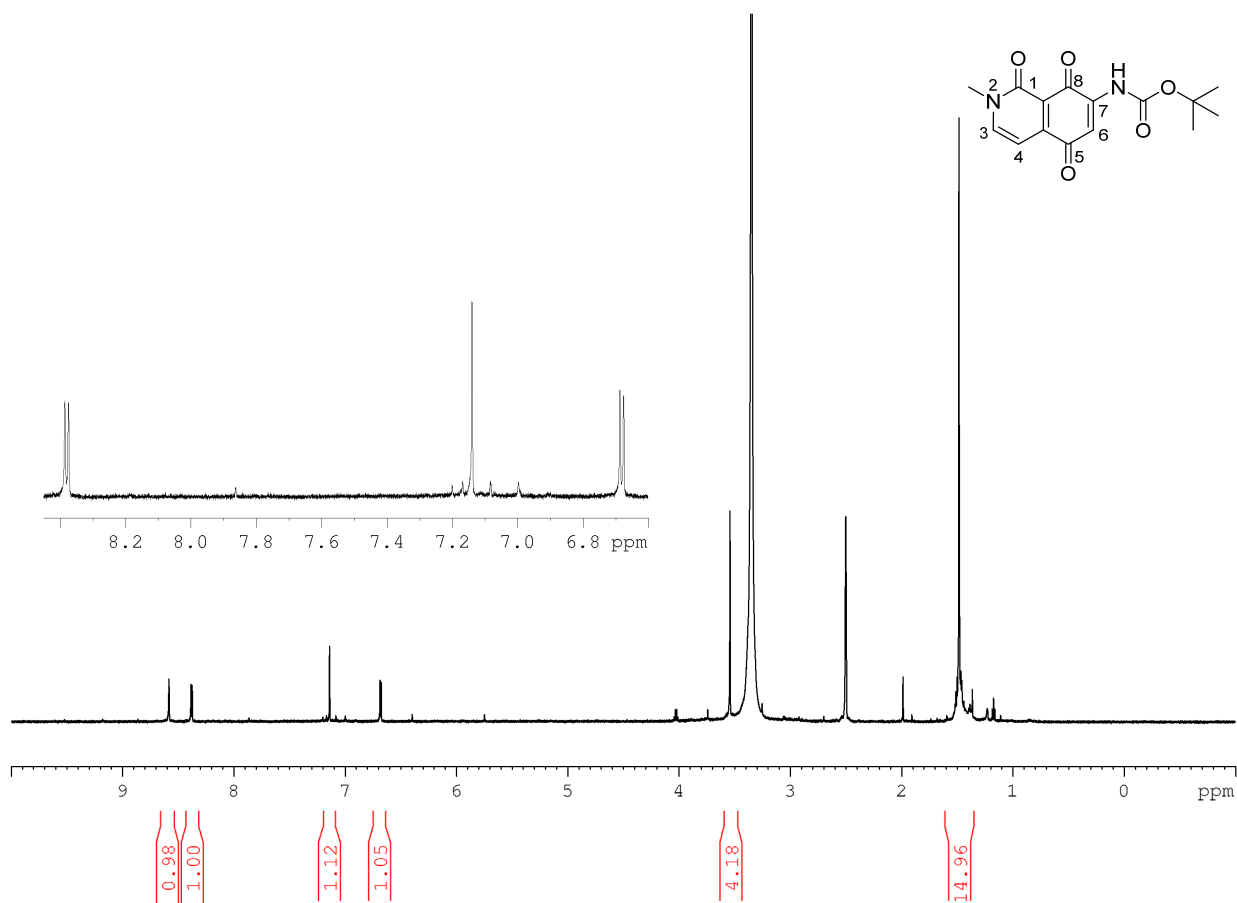
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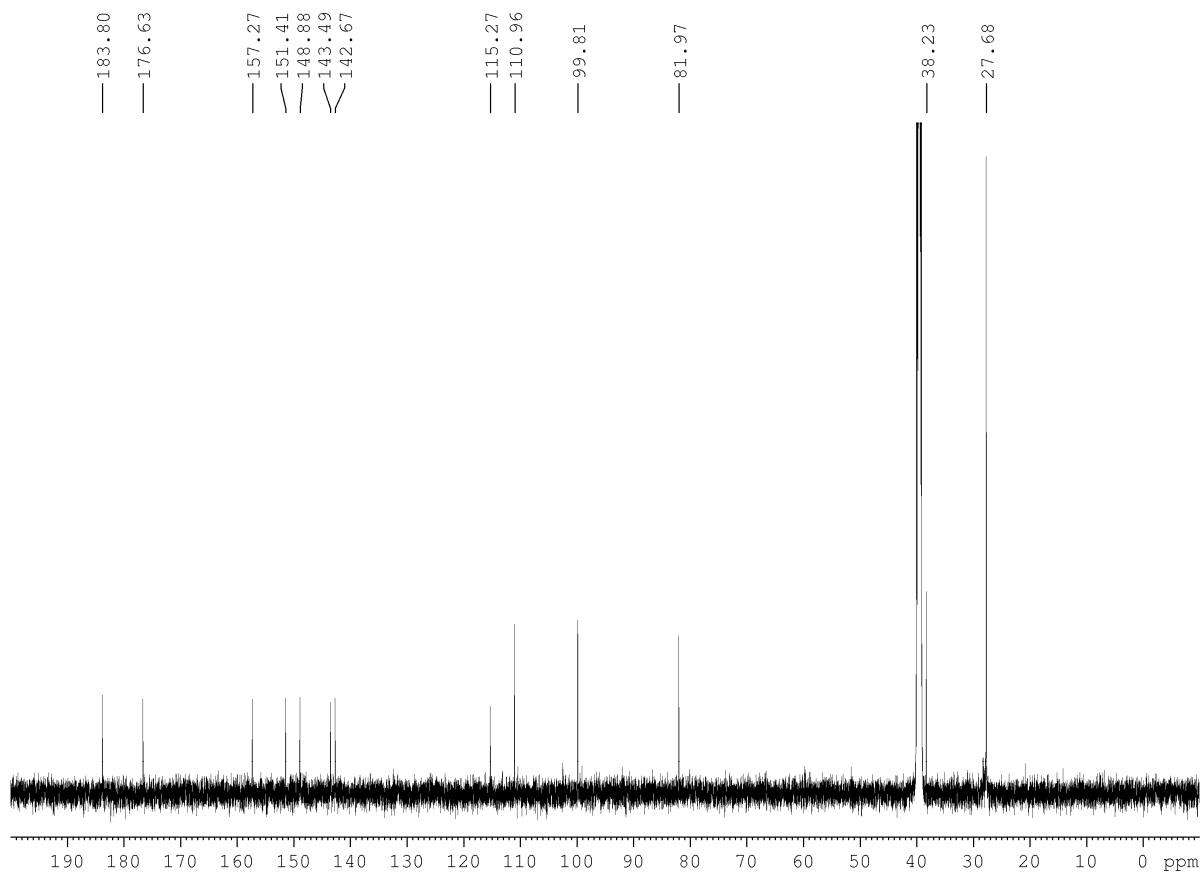
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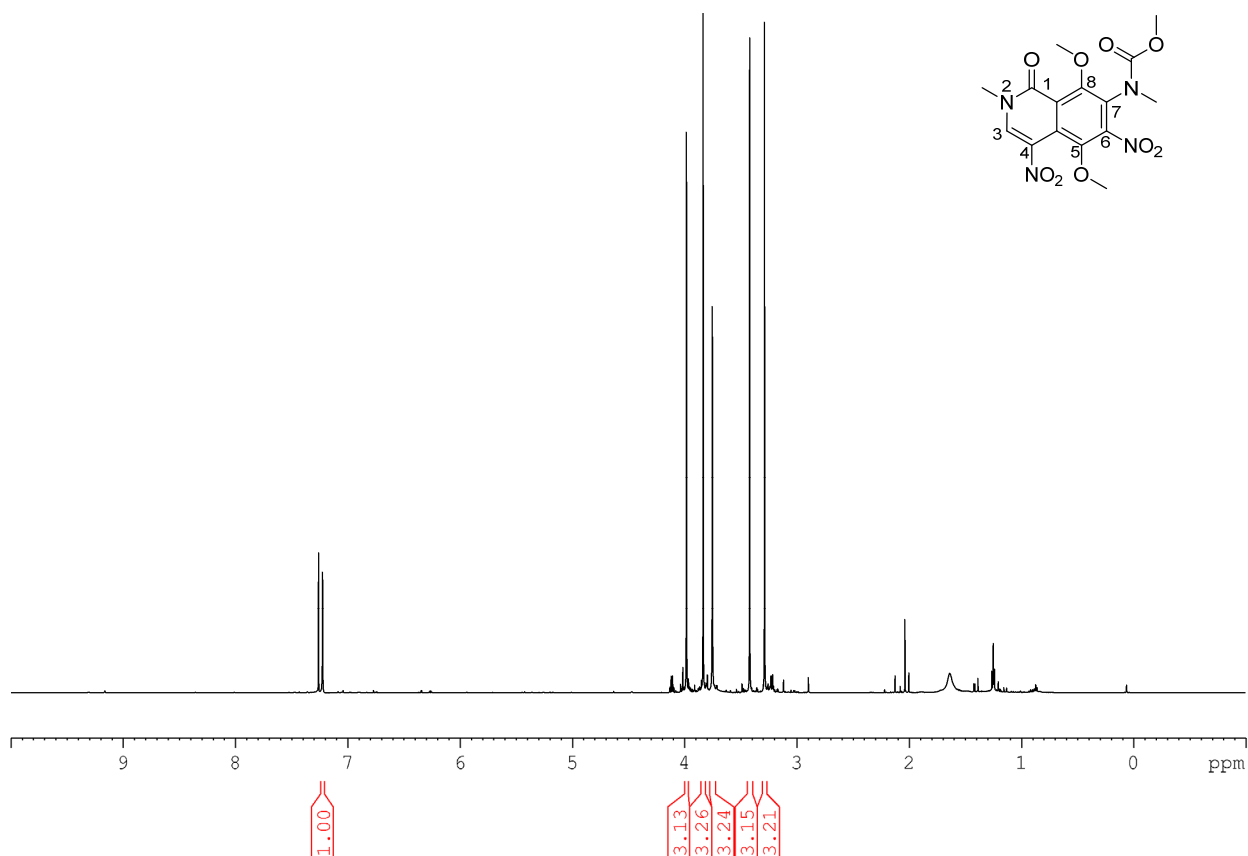
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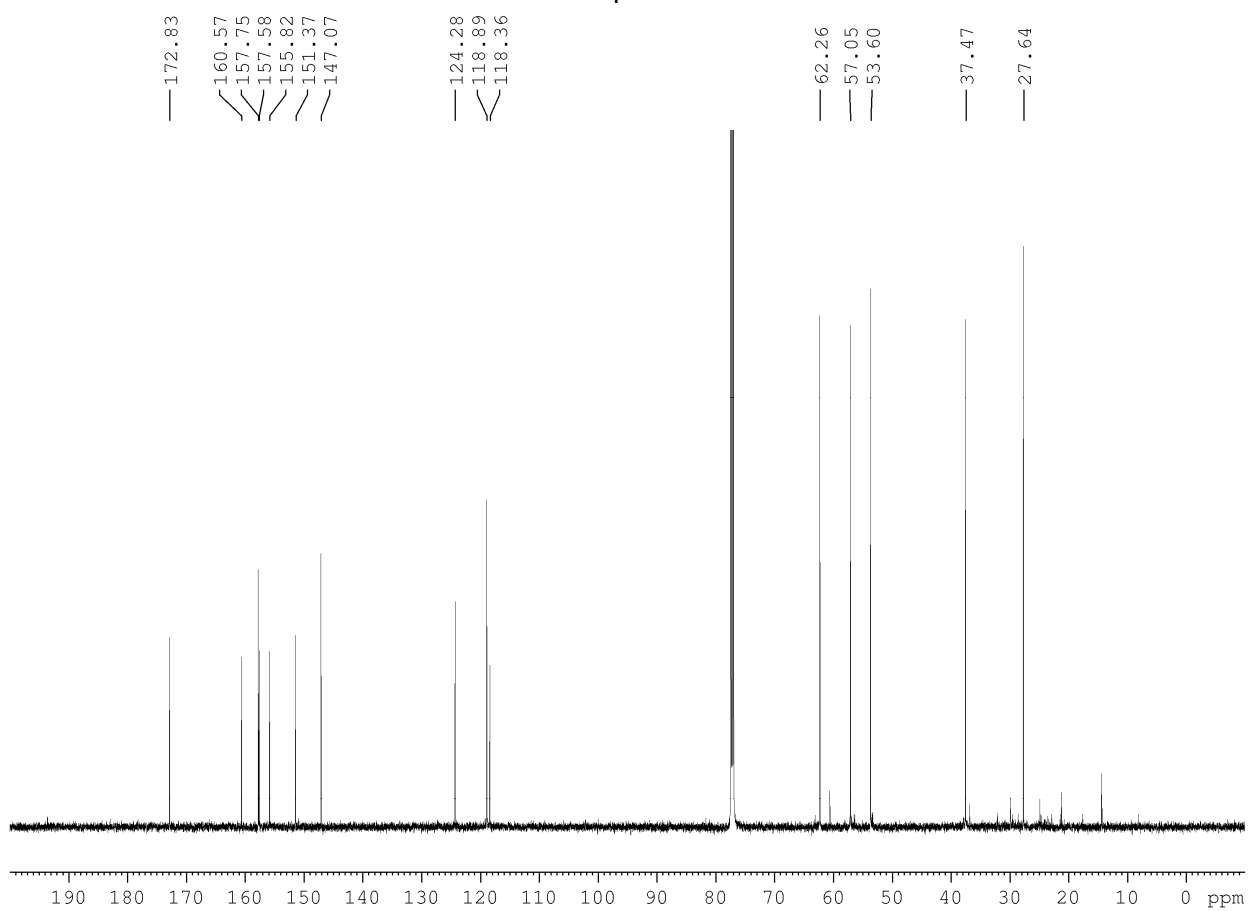
150 MHz ^{13}C NMR spectrum of **58** in d_6 -DMSO



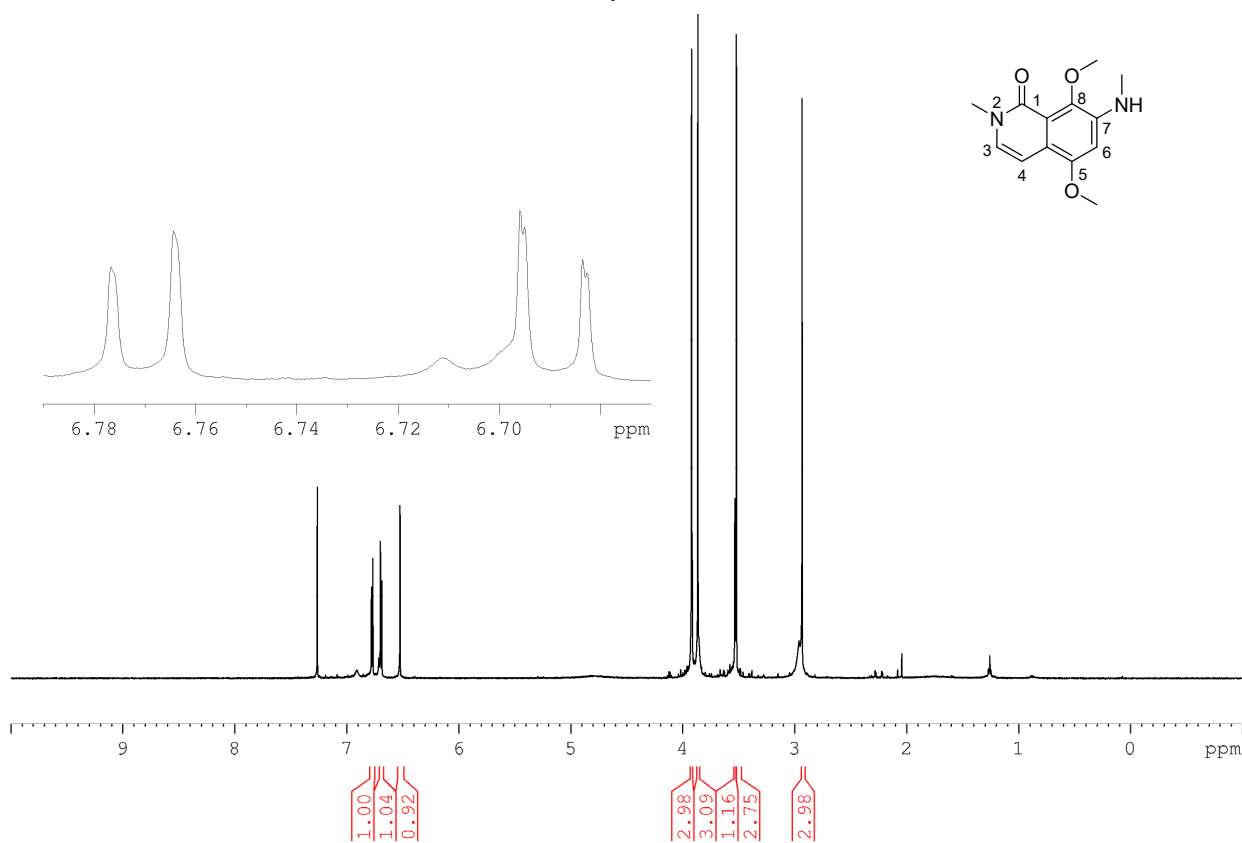
600 MHz ^1H NMR spectrum of **61** in CDCl_3



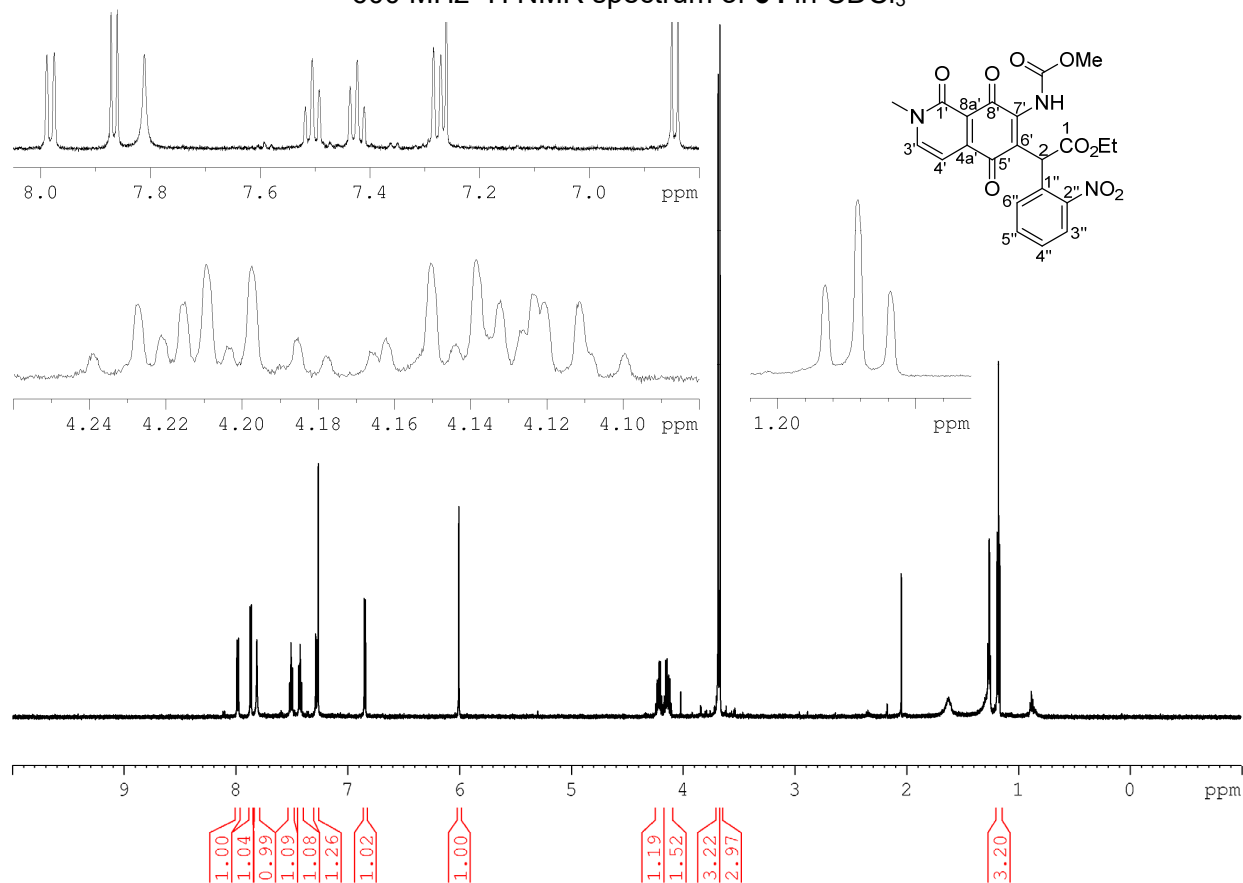
150 MHz ^{13}C NMR spectrum of **61** in CDCl_3



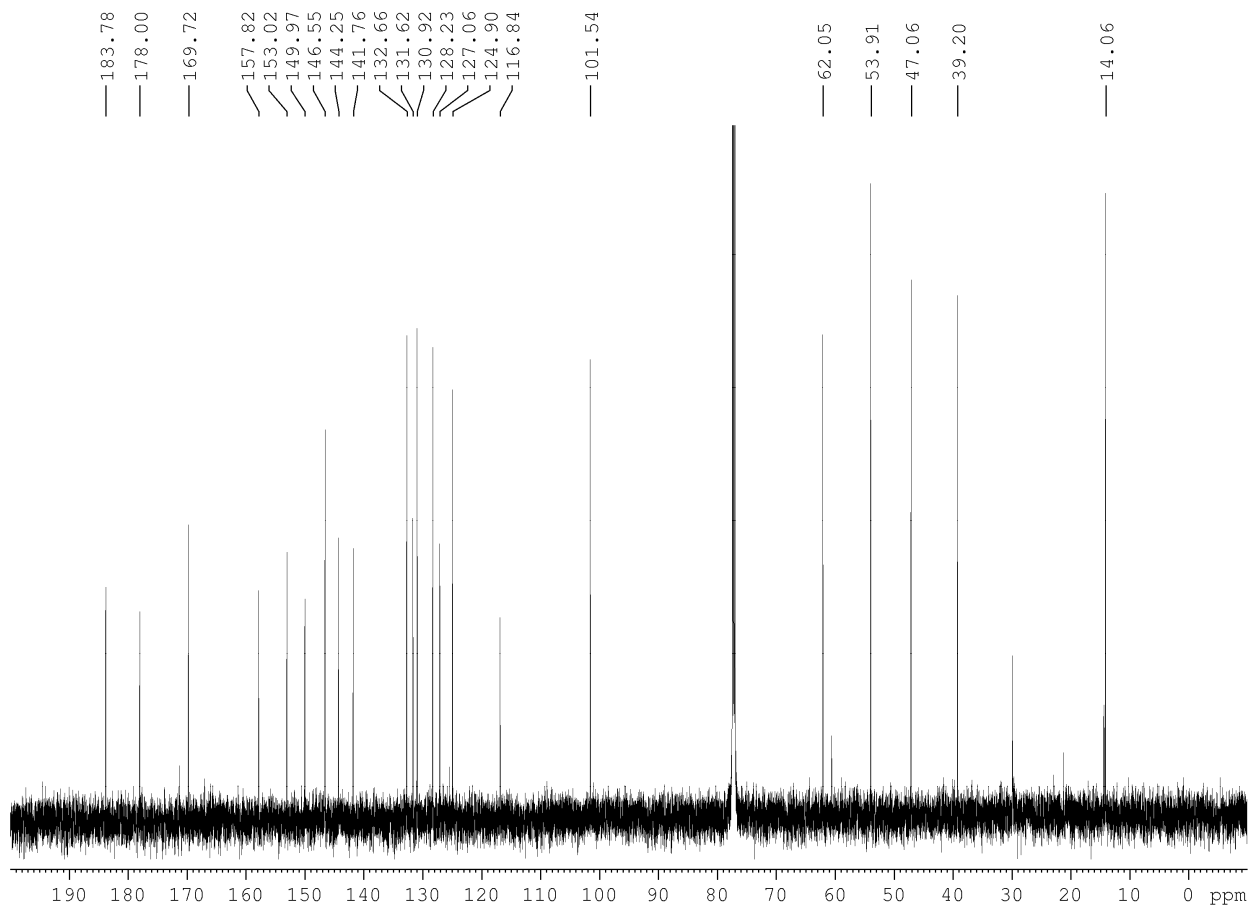
600 MHz ^1H NMR spectrum of **62** in CDCl_3



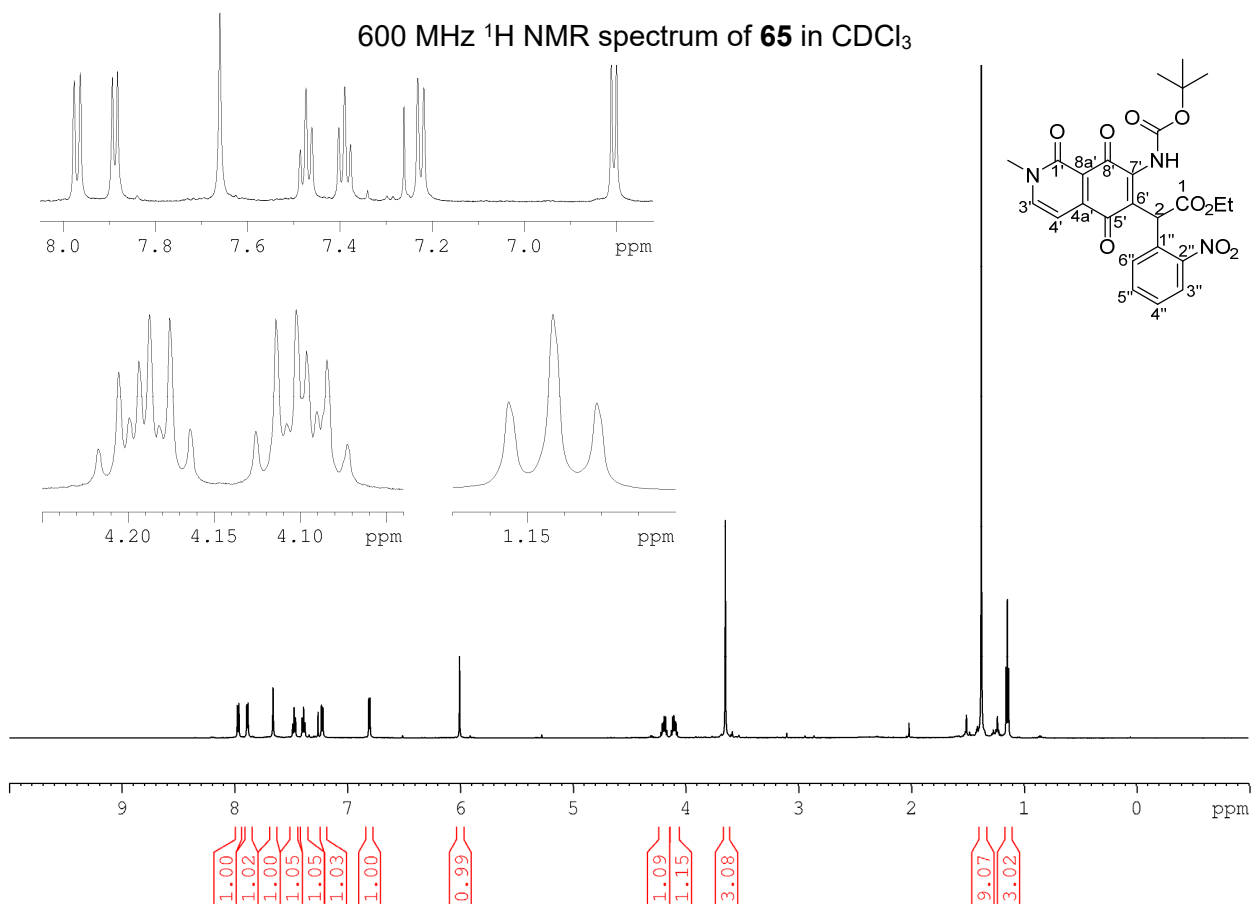
600 MHz ^1H NMR spectrum of **64** in CDCl_3



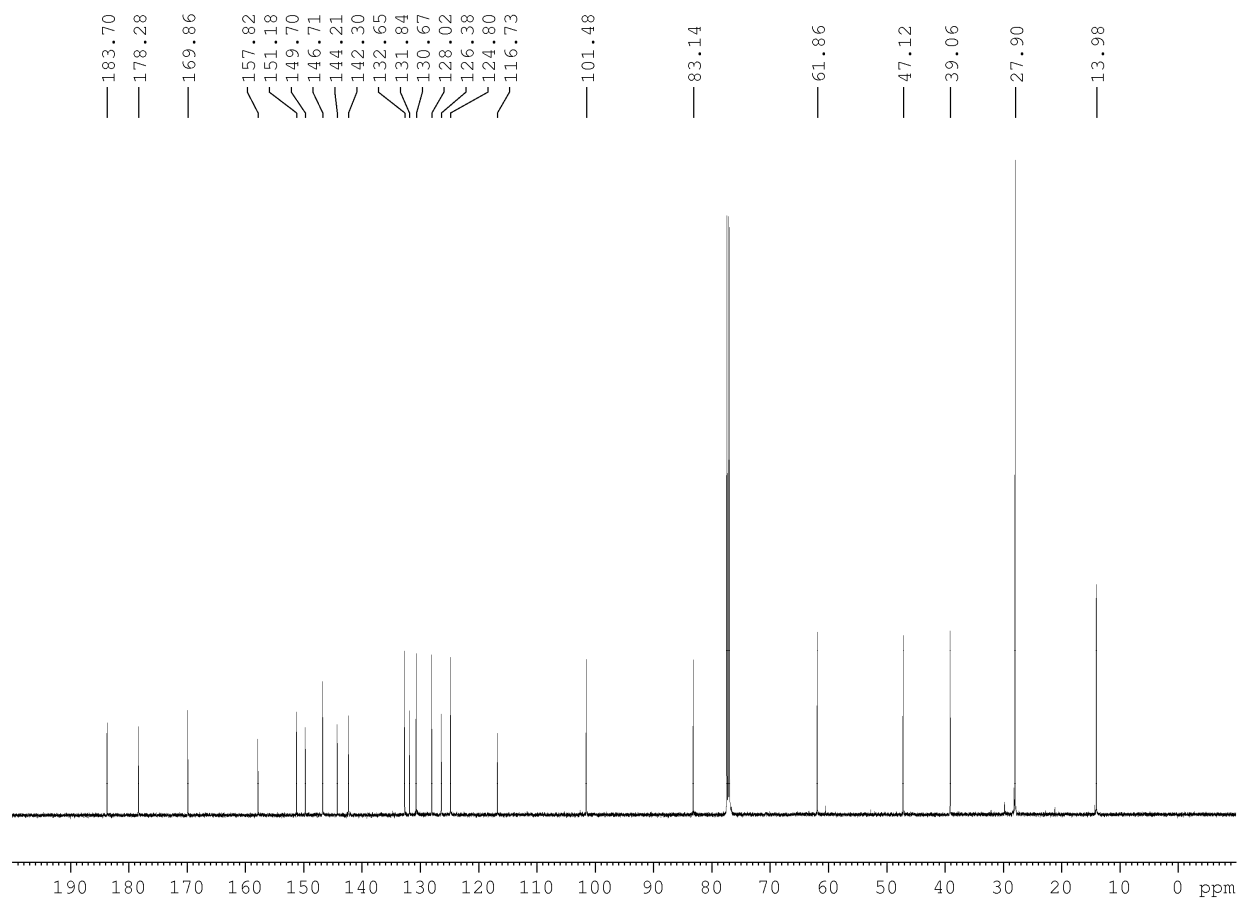
150 MHz ^{13}C NMR spectrum of **64** in CDCl_3



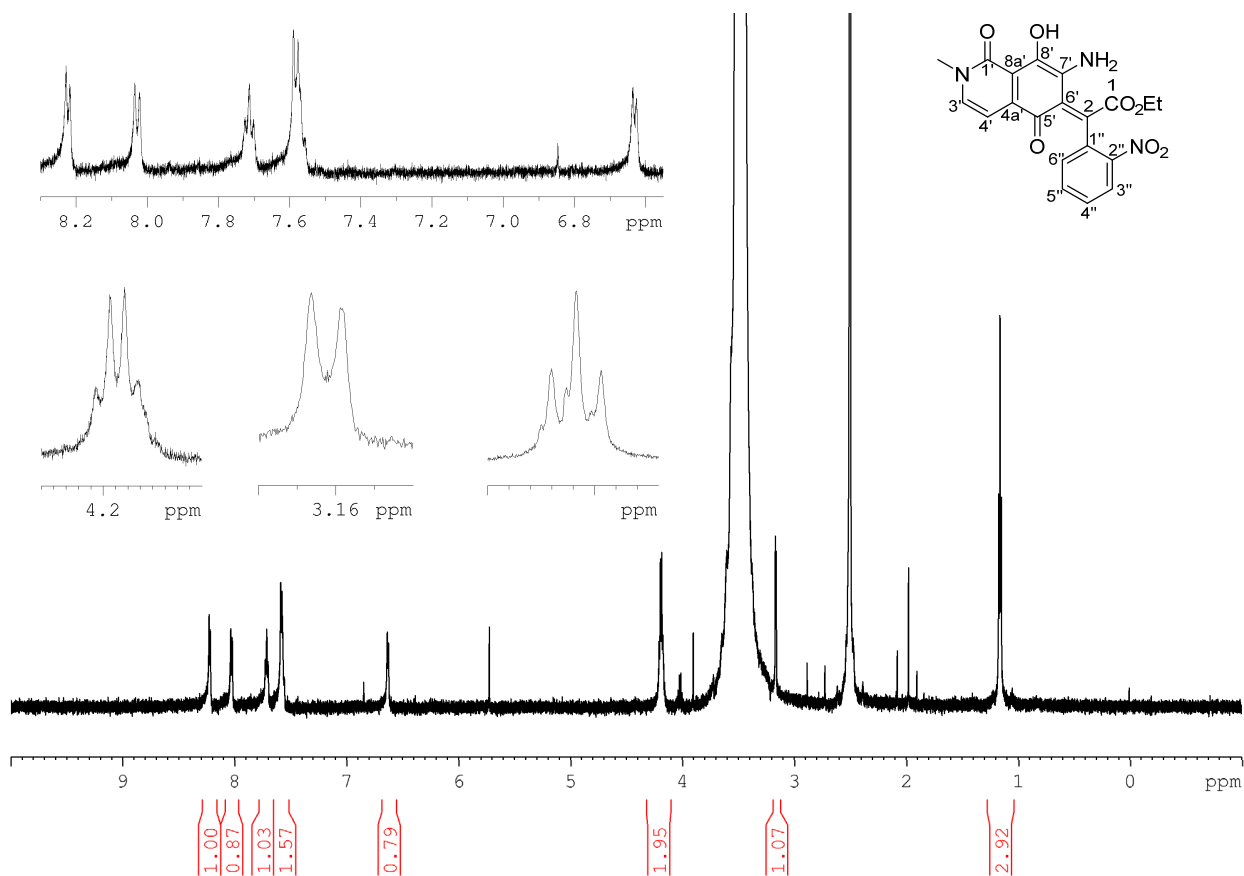
600 MHz ^1H NMR spectrum of **65** in CDCl_3



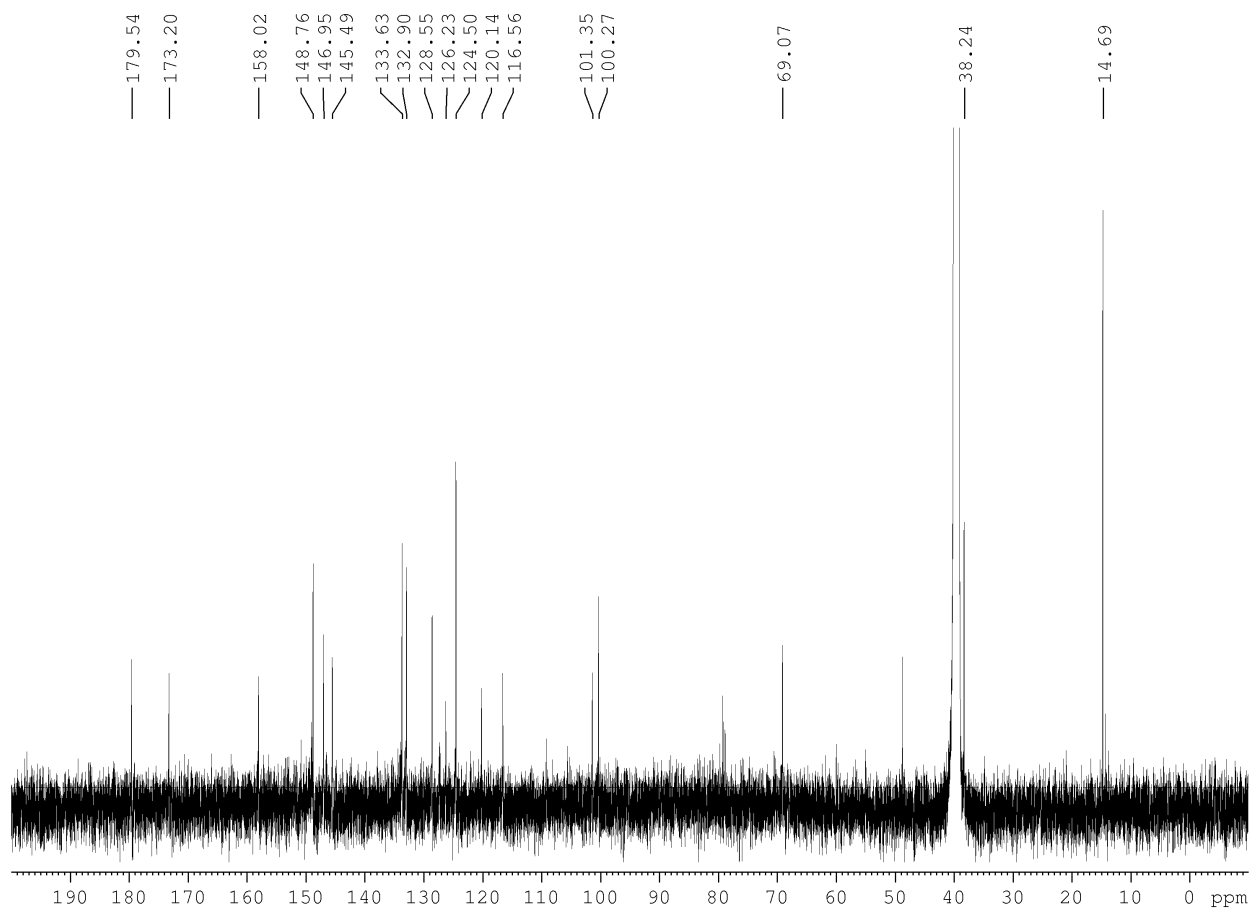
150 MHz ^{13}C NMR spectrum of **65** in CDCl_3



600 MHz ^1H NMR spectrum of **66** in d_6 -DMSO

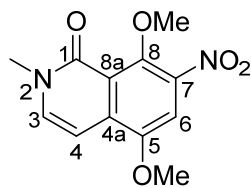


150 MHz ^{13}C NMR spectrum of **66** in d_6 -DMSO

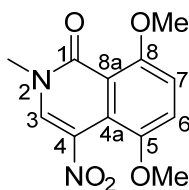


II. X-RAY CRYSTALLOGRAPHIC DATA

Crystallographic data were collected at 100(2) K on an Oxford Diffraction Xcalibur diffractometer using Mo K α radiation ($\lambda = 0.71073$ Å). Following multi-scan absorption corrections and solution by direct methods, the structures were refined against F^2 with full-matrix least-squares using the program SHELXL-2017. All hydrogen atoms were added at calculated positions and refined by use of a riding model with isotropic displacement parameters based on those of the parent atoms. Anisotropic displacement parameters were employed for the non-hydrogen atoms.

Table S1. Crystallographic data for 5,8-dimethoxy-2-methyl-7-nitroisoquinolin-1(2*H*)-one (**36**)

Empirical formula	C ₁₂ H ₁₂ N ₂ O ₅
Formula weight	264.24
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>
Unit cell dimensions	<i>a</i> = 15.7383(9) Å <i>b</i> = 10.5862(4) Å <i>c</i> = 7.0845(4) Å β = 90.023(5)°
Volume	1180.34(10) Å ³
<i>Z</i>	4
Density (calculated)	1.487 Mg/m ³
μ	0.118 mm ⁻¹
Crystal size	0.34 x 0.17 x 0.02 mm ³
θ range for data collection	2.88 to 28.99°.
Index ranges	−19 ≤ <i>h</i> ≤ 21, −14 ≤ <i>k</i> ≤ 13, −9 ≤ <i>l</i> ≤ 9
Reflections collected	10397
Independent reflections	3090 [<i>R</i> (int) = 0.0412]
Completeness to θ = 28.75°	99.1 %
Absorption correction	Semi-empirical from equivalents
Max./min transmission	1.00/0.892
Refinement method	Full-matrix least-squares on <i>F</i> ²
Data / restraints / parameters	3090 / 0 / 176
Goodness-of-fit on <i>F</i> ²	1.179
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0687, <i>wR</i> 2 = 0.1186
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0851, <i>wR</i> 2 = 0.1242
Largest diff. peak and hole	0.307 and −0.314 e.Å ⁻³
CCDC	1825607

Table S2. Crystallographic data for 5,8-dimethoxy-2-methyl-4-nitroisoquinolin-1(2*H*)-one (**37**)

Empirical formula	C ₁₂ H ₁₂ N ₂ O ₅
Formula weight	264.24
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>
Unit cell dimensions	<i>a</i> = 9.8063(3) Å <i>b</i> = 15.7583(3) Å <i>c</i> = 7.4795(2) Å β = 102.000(3)°
Volume	1130.55(5) Å ³
<i>Z</i>	4
Density (calculated)	1.552 Mg/m ³
Absorption coefficient	0.123 mm ⁻¹
Crystal size	0.40 x 0.10 x 0.08 mm ³
θ range for data collection	3.07 to 31.00°.
Index ranges	-14 ≤ <i>h</i> ≤ 14, -22 ≤ <i>k</i> ≤ 22, -10 ≤ <i>l</i> ≤ 7
Reflections collected	12815
Independent reflections	3559 [<i>R</i> (int) = 0.0328]
Completeness to θ = 28.75°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max./min transmission	1.00/0.963
Refinement method	Full-matrix least-squares on <i>F</i> ²
Data / restraints / parameters	3559 / 0 / 175
Goodness-of-fit on <i>F</i> ²	1.107
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0482, <i>wR</i> 2 = 0.1162
<i>R</i> indices (all data)	<i>R</i> 1 = 0.0580, <i>wR</i> 2 = 0.1212
Largest diff. peak and hole	0.492 and -0.244 e.Å ⁻³
CCDC	1825618