

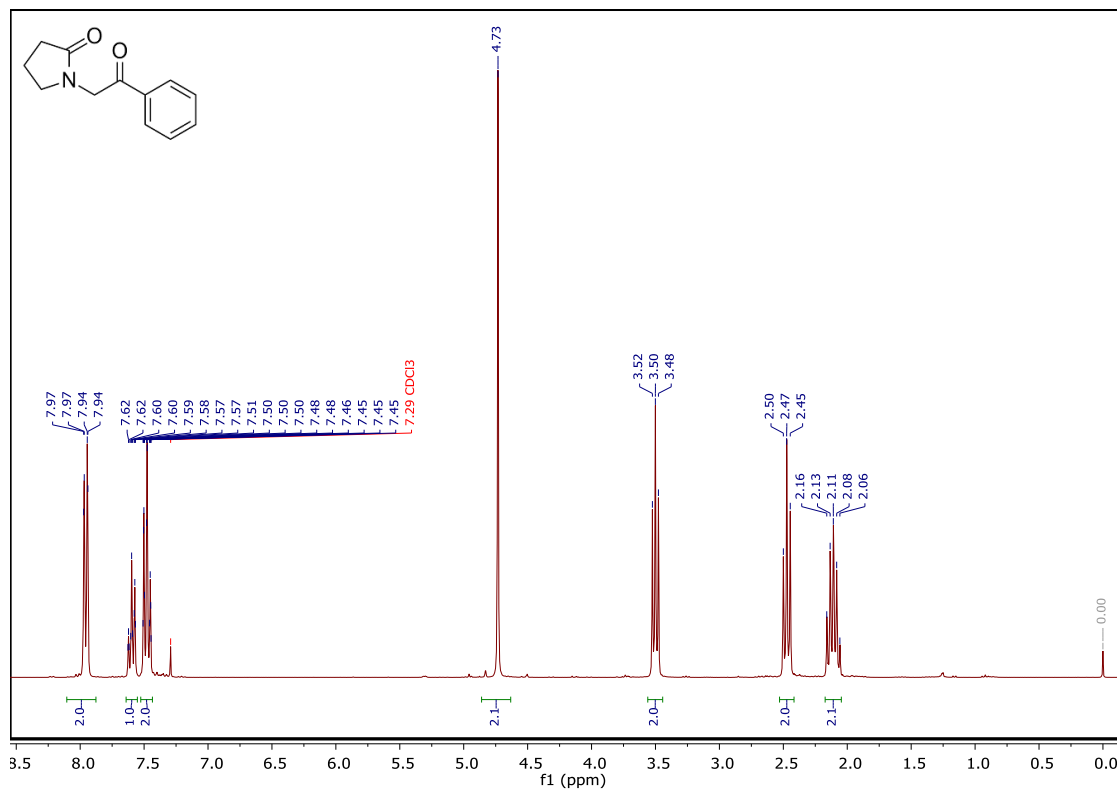
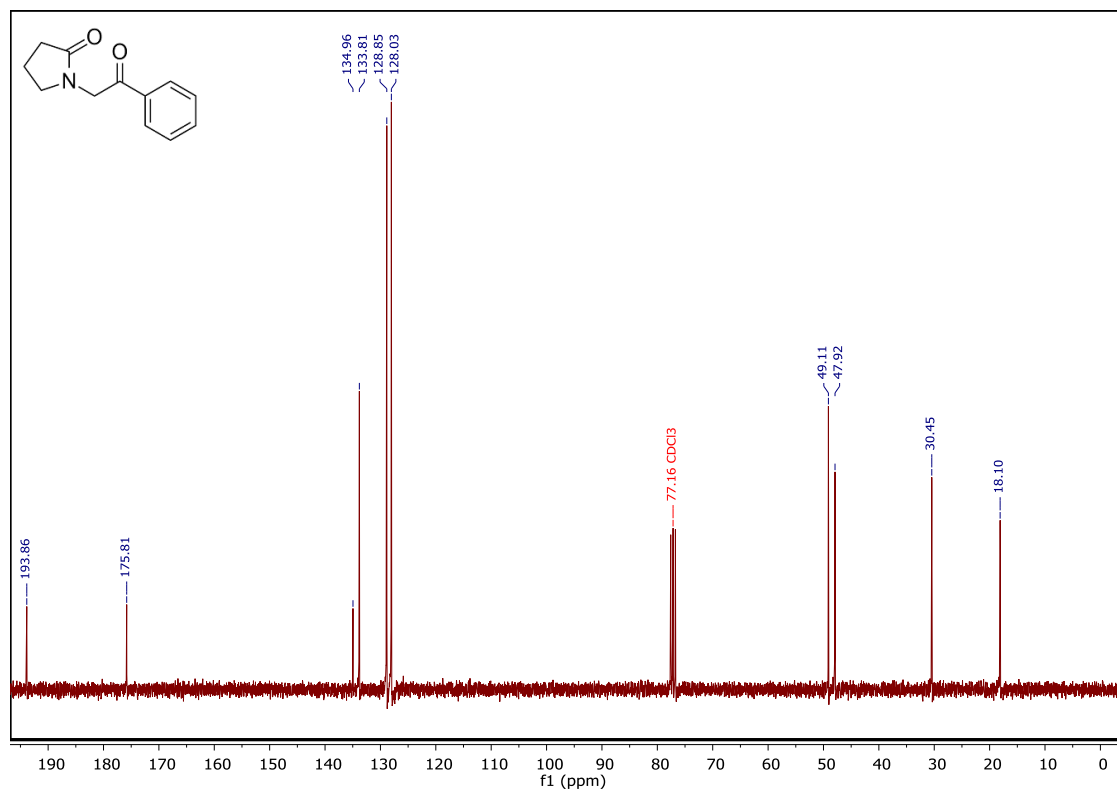
Supplementary Material

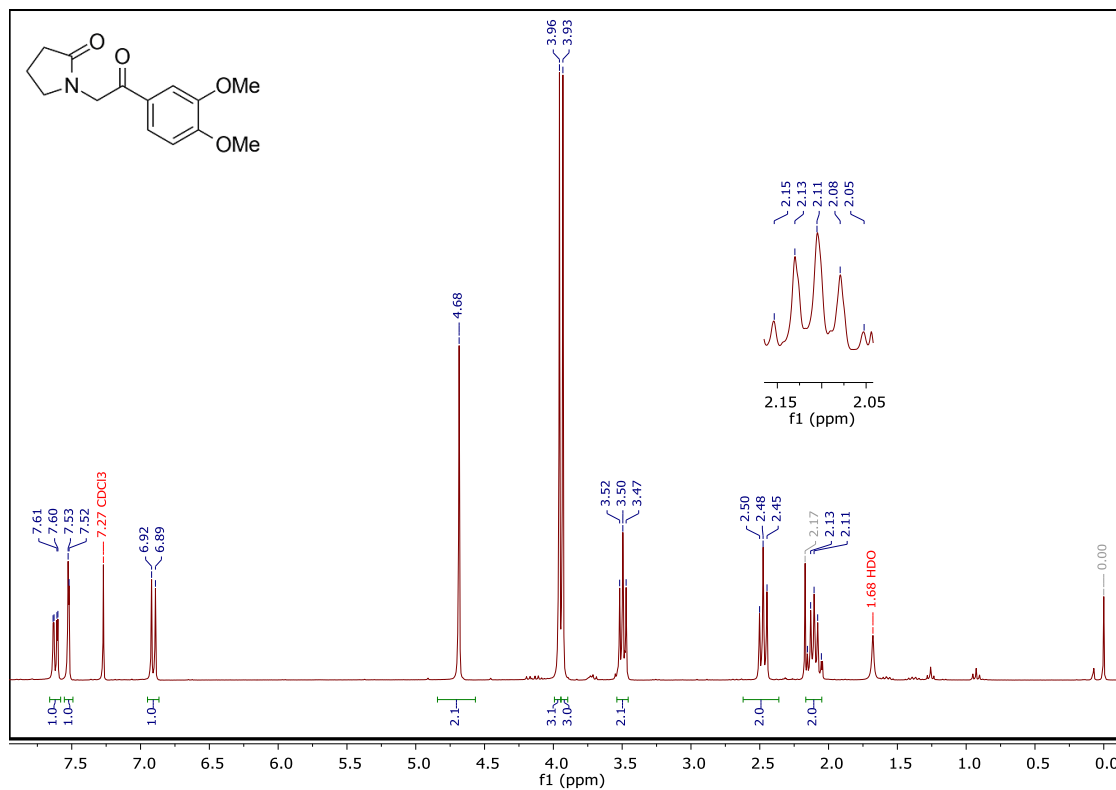
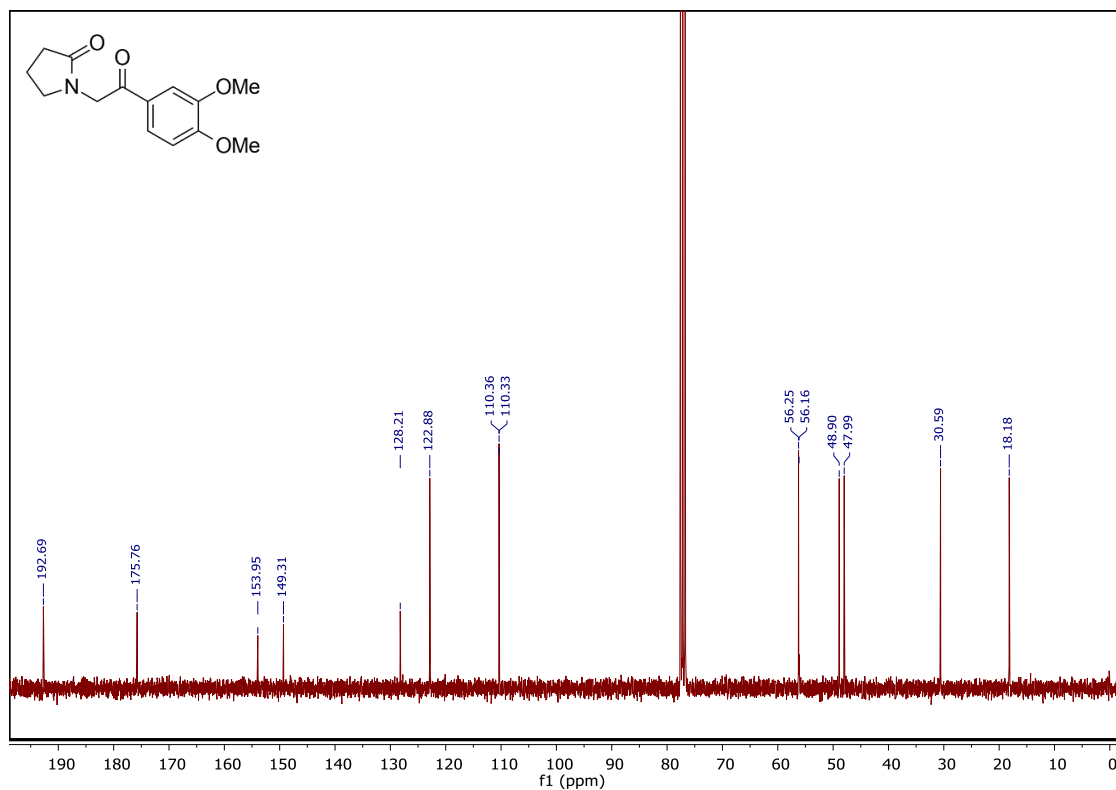
Cyclization of enaminones derived from *N*-phenacylpyrrolidin-2-ones to pyrrolizines under acidic conditionsGarreth L. Morgans,^a Manuel A. Fernandes,^a Willem A. L. van Otterlo,^{a,b} and Joseph P. Michael^{*a}^a Molecular Sciences Institute, School of Chemistry, University of the Witwatersrand, Johannesburg, PO Wits 2050, South Africa^b Department of Chemistry and Polymer Science, Stellenbosch University, Matieland 7602, Stellenbosch, South AfricaEmail: joseph.michael@wits.ac.za

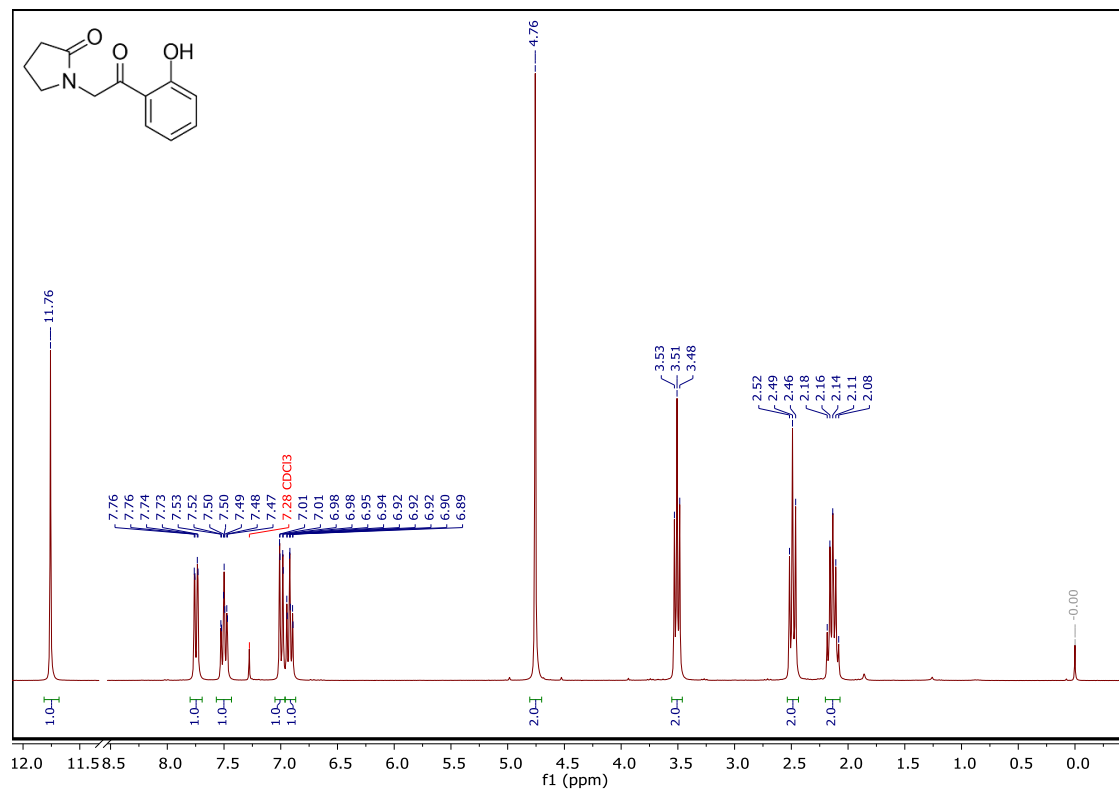
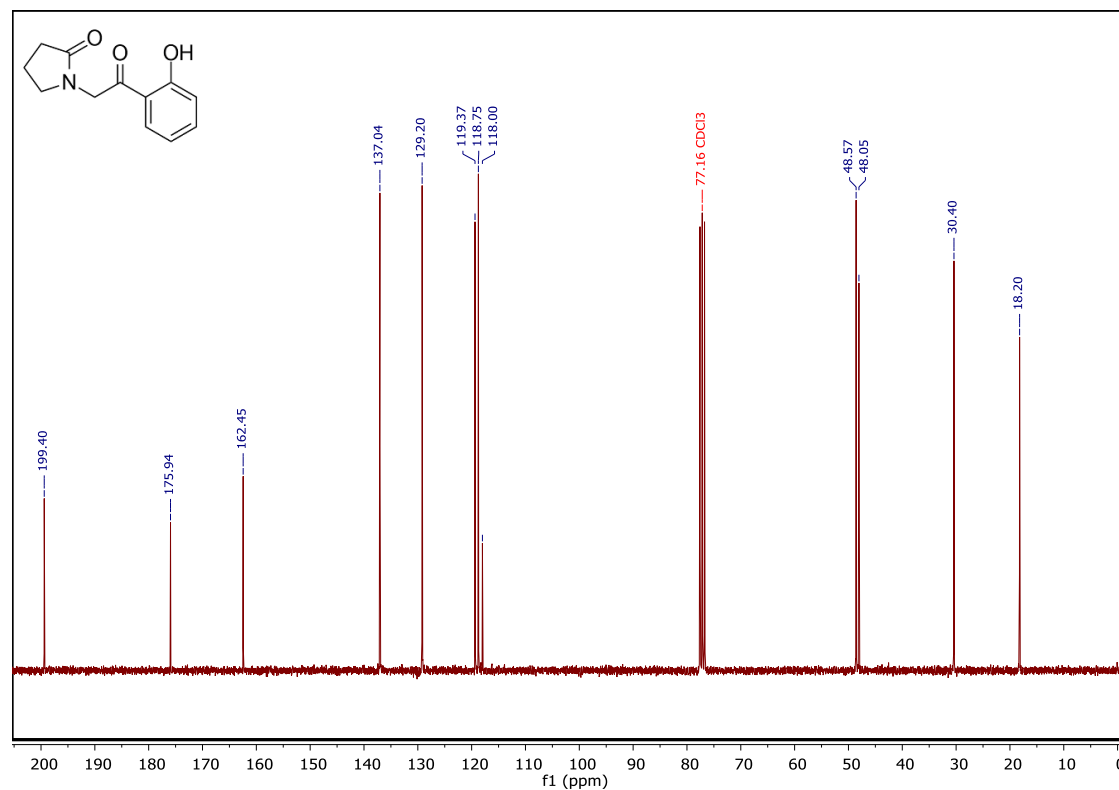
Table of Contents

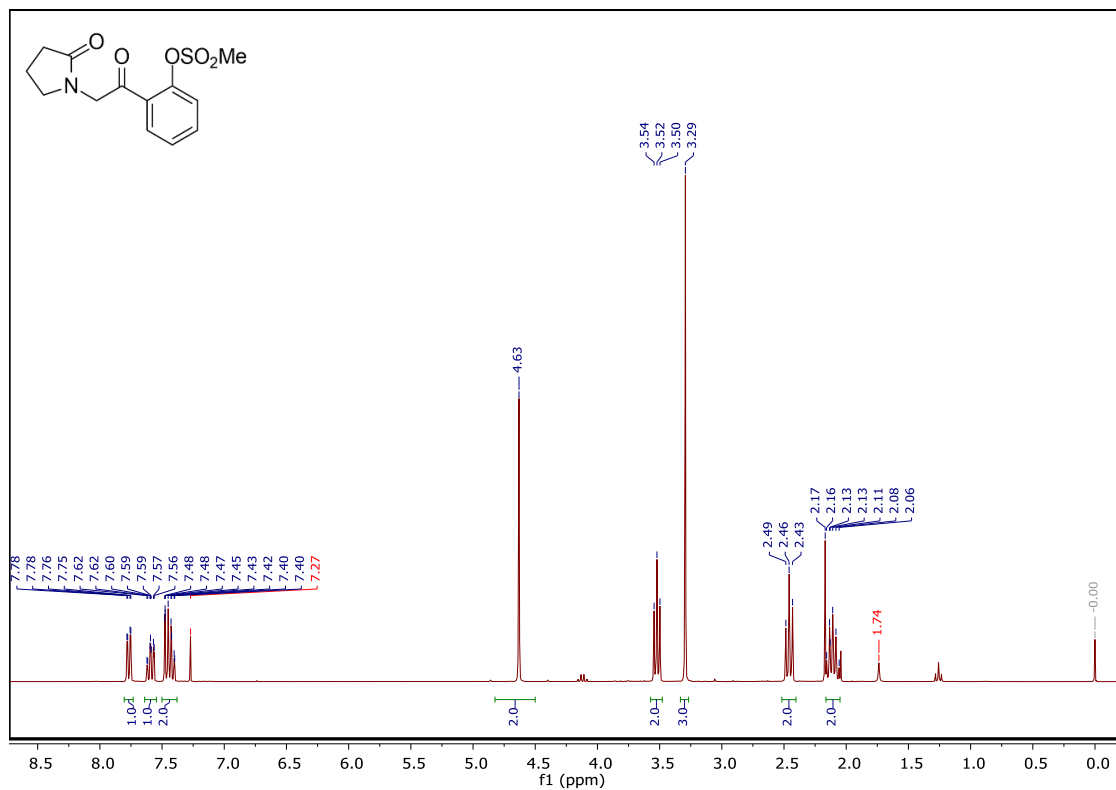
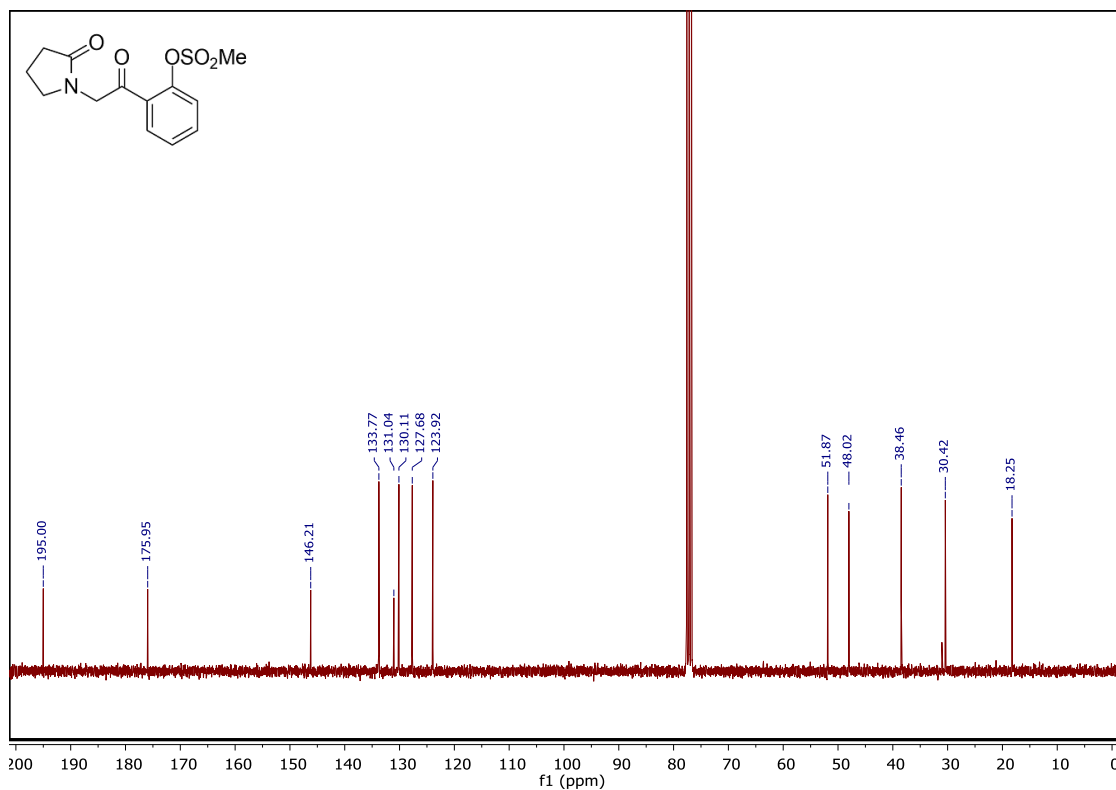
¹ H and ¹³ C NMR spectra of 1-(2-oxo-2-phenylethyl)pyrrolidin-2-one (13a)	S3
¹ H and ¹³ C NMR spectra of 1-[2-(3,4-dimethoxyphenyl)-2-oxoethyl]pyrrolidin-2-one (13b)	S4
¹ H and ¹³ C NMR spectra of 1-[2-(2-hydroxyphenyl)-2-oxoethyl]pyrrolidin-2-one (13c)	S5
¹ H and ¹³ C NMR spectra of 2-[2-(2-oxopyrrolidin-1-yl)acetyl]phenyl methanesulfonate (13d)	S6
¹ H and ¹³ C NMR spectra of [2,3-bis(3,4-dimethoxybenzoyl)cyclopropyl](3,4-dimethoxyphenyl)methanone (14)	S7
¹ H and ¹³ C NMR spectra of 1-phenyl-2-(2-thioxopyrrolidin-1-yl)ethanone (15a)	S8
¹ H and ¹³ C NMR spectra of 1-(3,4-dimethoxyphenyl)-2-(2-thioxopyrrolidin-1-yl)ethanone (15b)	S9
¹ H and ¹³ C NMR spectra of 1-(2-hydroxyphenyl)-2-(2-thioxopyrrolidin-1-yl)ethanone (15c)	S10
¹ H and ¹³ C NMR spectra of 2-[2-(2-thioxopyrrolidin-1-yl)acetyl]phenyl methanesulfonate (15d)	S11
¹ H and ¹³ C NMR spectra of (<i>E</i>)-2-[1-(2-oxo-2-phenylethyl)pyrrolidin-2-ylidene]-1-phenylethanone (16a)	S12
¹ H and ¹³ C NMR spectra of (<i>E</i>)-1-(3,4-dimethoxyphenyl)-2-[1-[2-(3,4-dimethoxyphenyl)-2-oxoethyl]pyrrolidin-2-ylidene]ethanone (16b)	S13
¹ H and ¹³ C NMR spectra of phenyl(6-phenyl-2,3-dihydro-1 <i>H</i> -pyrrolizin-7-yl)methanone (17a)	S14
¹ H and ¹³ C NMR spectra of (3,4-dimethoxyphenyl)[6-(3,4-dimethoxyphenyl)-2,3-dihydro-1 <i>H</i> -pyrrolizin-7-yl]methanone (17b)	S15
¹ H and ¹³ C NMR spectra of 2-(7-benzoyl-2,3-dihydro-1 <i>H</i> -pyrrolizin-6-yl)phenyl methanesulfonate (17d)	S16
¹ H and ¹³ C NMR spectra of [6-(3,4-dimethoxyphenyl)-2,3-dihydro-1 <i>H</i> -pyrrolizin-7-yl](phenyl)methanone (19)	S17
¹ H and ¹³ C NMR spectra of 1-[(<i>E</i>)-2-(2-hydroxyphenyl)-4-oxo-4-phenyl-1-butenyl]pyrrolidin-2-one (20)	S18
¹ H and ¹³ C NMR spectra of 1-(2-phenyl-2-propenyl)pyrrolidin-2-one (27)	S19
¹ H and ¹³ C NMR spectra of 1-(2-phenyl-2-propenyl)pyrrolidine-2-thione (29)	S20
¹ H and ¹³ C NMR spectra of (<i>E</i>)-1-phenyl-2-[1-(2-phenyl-2-propenyl)pyrrolidin-2-ylidene]ethanone (30)	S21
¹ H and ¹³ C NMR spectra of 7-benzoyl-6-phenyl-2,3-dihydro-1 <i>H</i> -pyrrolizine-5-carbaldehyde (32)	S22
¹ H and ¹³ C NMR spectra of (5-bromo-6-phenyl-2,3-dihydro-1 <i>H</i> -pyrrolizin-7-yl)(phenyl)methanone (33a)	S23
¹ H and ¹³ C NMR spectra of [5-bromo-6-(3,4-dimethoxyphenyl)-2,3-dihydro-1 <i>H</i> -pyrrolizin-7-yl](phenyl)methanone (33b)	S24

¹ H and ¹³ C NMR spectra of (5,6-diphenyl-2,3-dihydro-1 <i>H</i> -pyrrolizin-7-yl)(phenyl)methanone (34a)	S25
¹ H and ¹³ C NMR spectra of [5-(3,4-dimethoxyphenyl)-6-phenyl-2,3-dihydro-1 <i>H</i> -pyrrolizin-7-yl](phenyl)methanone (34b)	S26
¹ H and ¹³ C NMR spectra of [5,6-bis(3,4-dimethoxyphenyl)-2,3-dihydro-1 <i>H</i> -pyrrolizin-7-yl](phenyl)methanone (34c)	S27

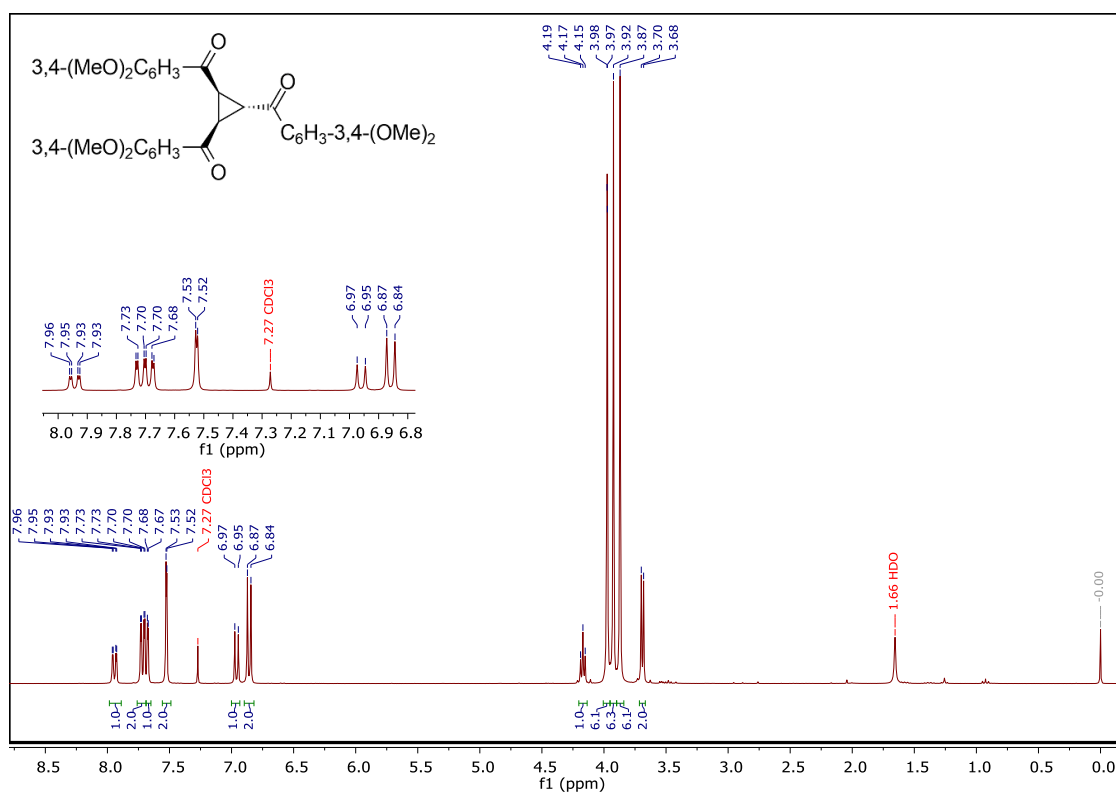
^1H NMR spectrum of 1-(2-oxo-2-phenylethyl)pyrrolidin-2-one (13a) (300 MHz, CDCl_3) **^{13}C NMR spectrum of 1-(2-oxo-2-phenylethyl)pyrrolidin-2-one (13a) (75 MHz, CDCl_3)**

^1H NMR spectrum of 1-[2-(3,4-dimethoxyphenyl)-2-oxoethyl]pyrrolidin-2-one (13b) (300 MHz, CDCl_3) **^{13}C NMR spectrum of 1-[2-(3,4-dimethoxyphenyl)-2-oxoethyl]pyrrolidin-2-one (13b) (75 MHz, CDCl_3)**

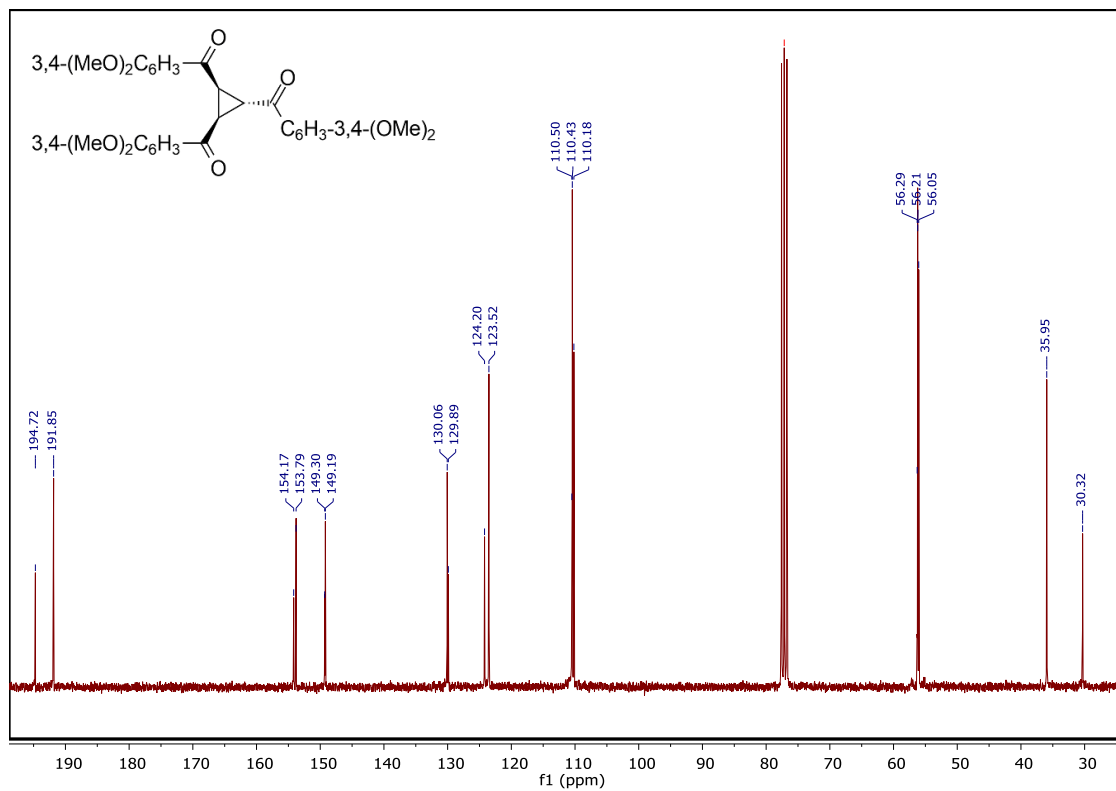
^1H NMR spectrum of 1-[2-(2-hydroxyphenyl)-2-oxoethyl]-pyrrolidin-2-one (13c) (300 MHz, CDCl_3) **^{13}C NMR spectrum of 1-[2-(2-hydroxyphenyl)-2-oxoethyl]pyrrolidin-2-one (13c) (750 MHz, CDCl_3)**

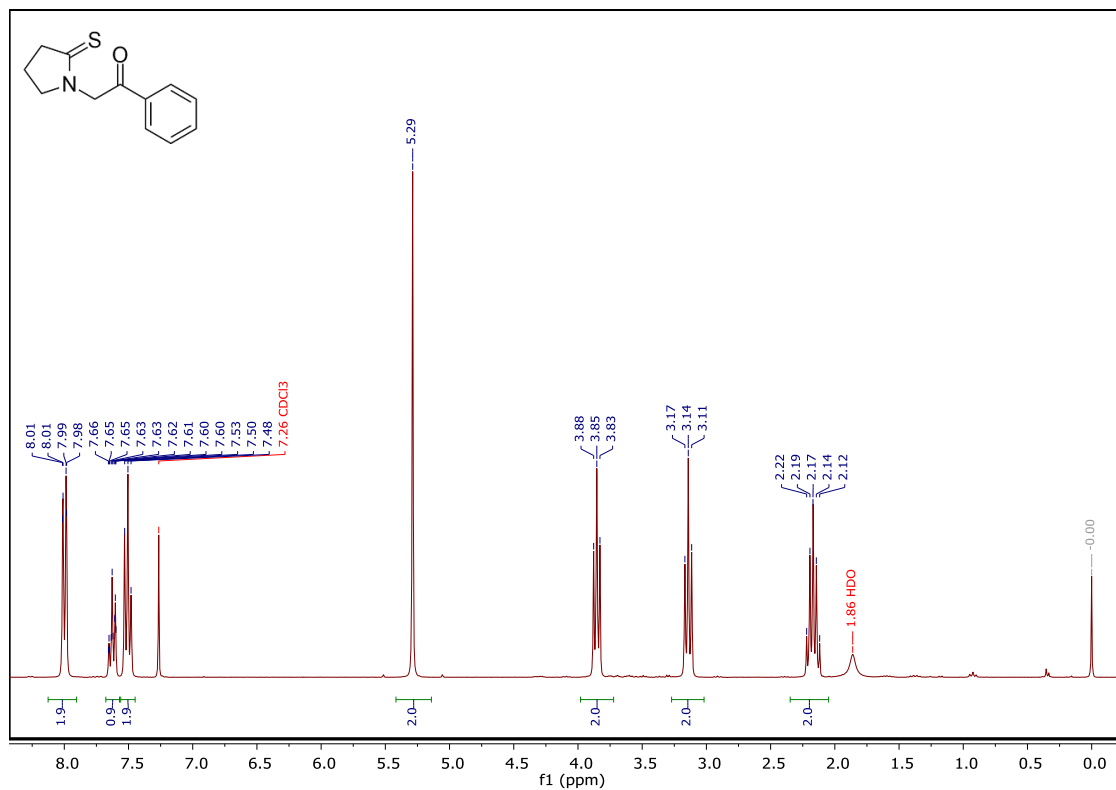
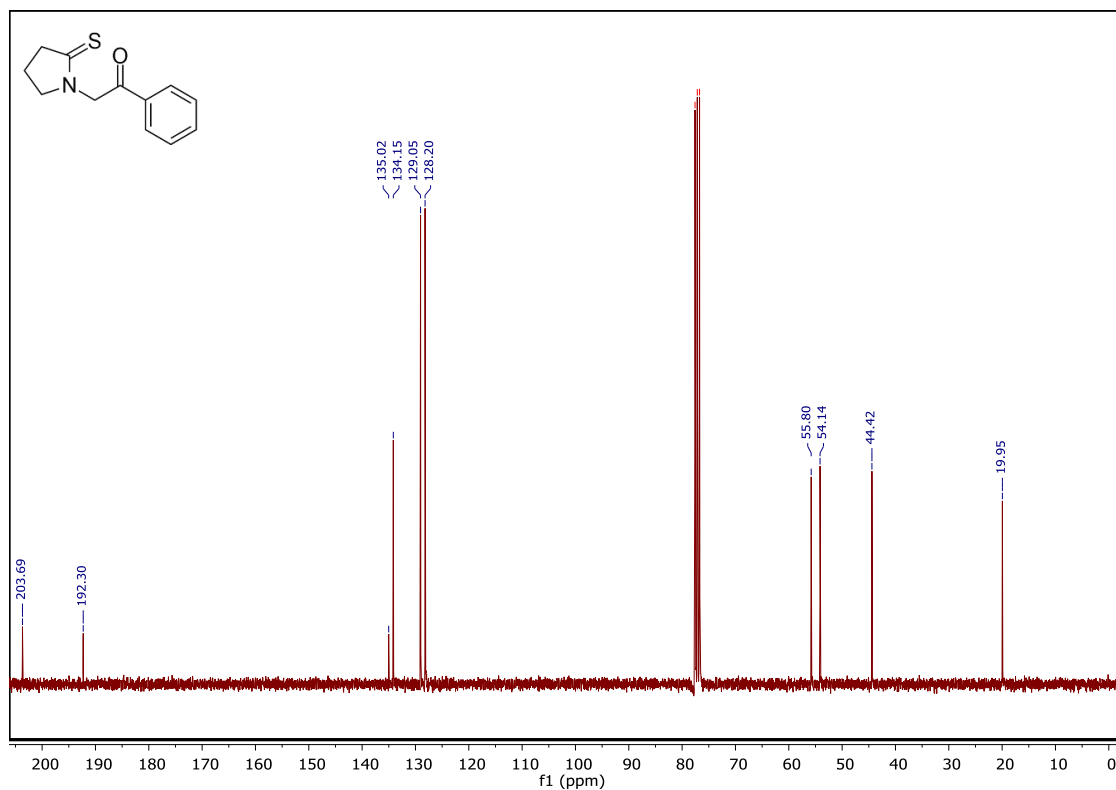
^1H NMR spectrum of 2-[2-(2-oxopyrrolidin-1-yl)acetyl]phenyl methanesulfonate (13d) (300 MHz, CDCl_3) **^{13}C NMR spectrum of 2-[2-(2-oxopyrrolidin-1-yl)acetyl]phenyl methanesulfonate (13d) (75 MHz, CDCl_3)**

^1H NMR spectrum of [2,3-bis(3,4-dimethoxybenzoyl)cyclopropyl](3,4-dimethoxyphenyl)methanone (14)
(300 MHz, CDCl_3)

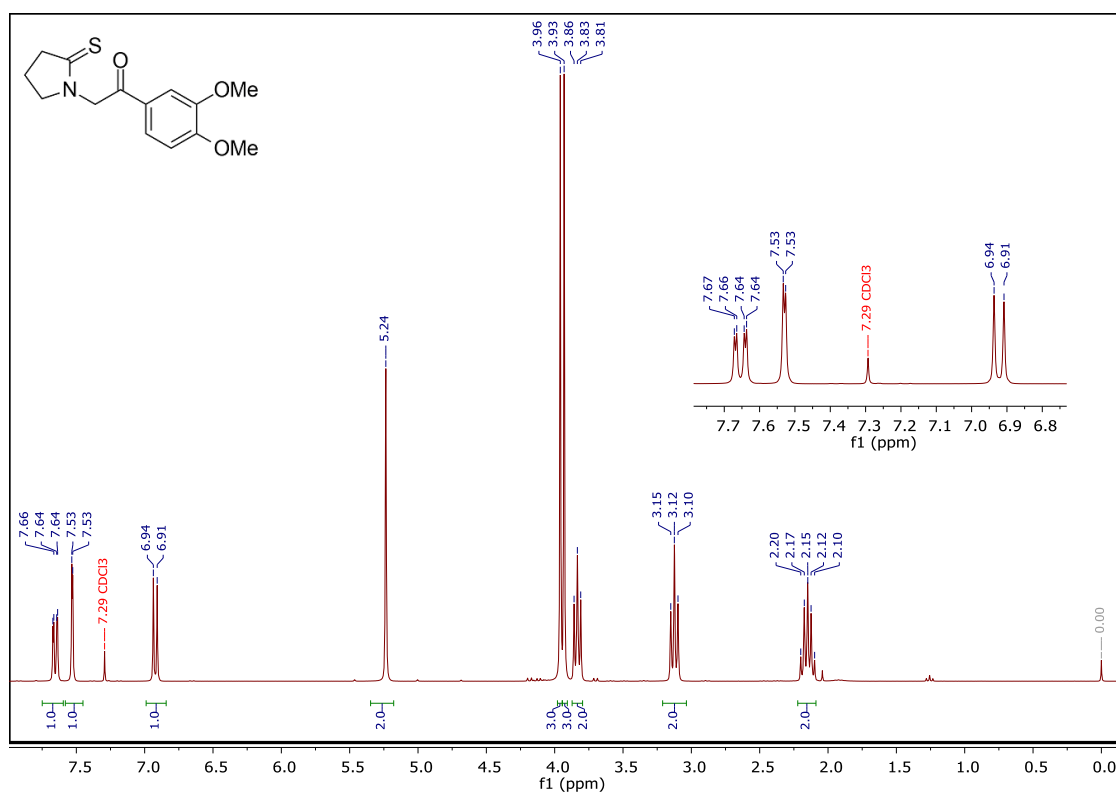


^{13}C NMR spectrum of [2,3-bis(3,4-dimethoxybenzoyl)cyclopropyl](3,4-dimethoxyphenyl)methanone (14)
(75 MHz, CDCl_3)

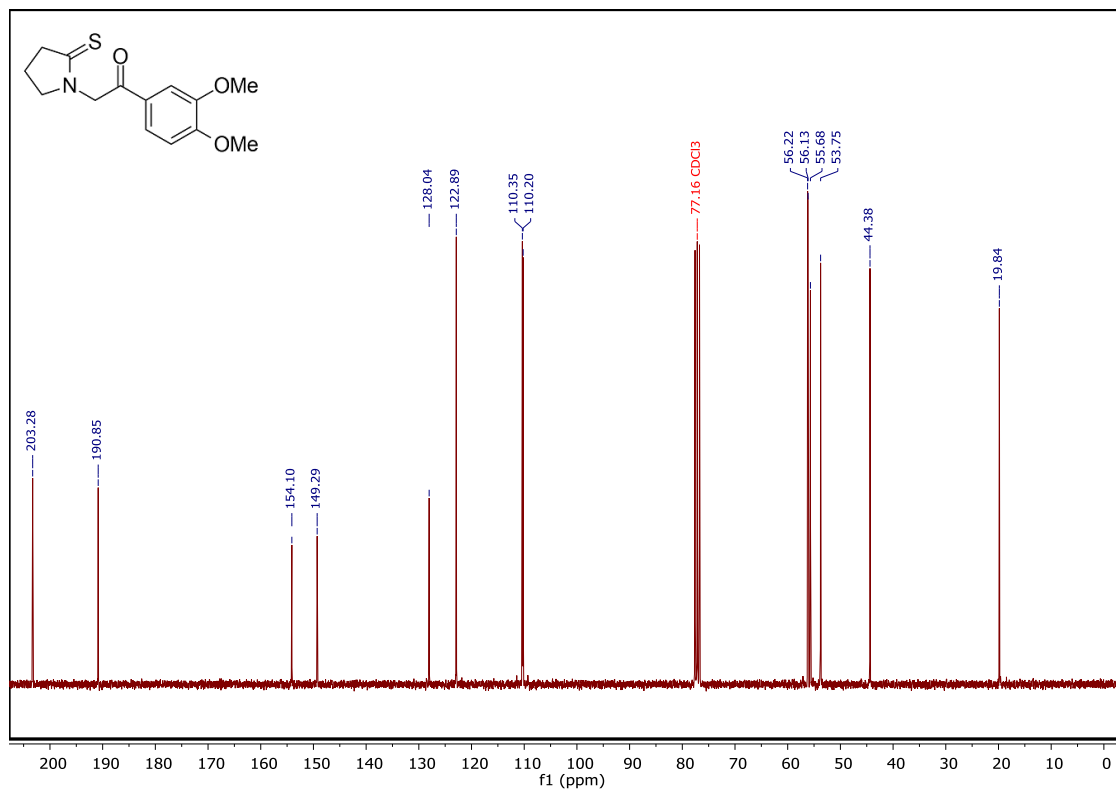


^1H NMR spectrum of 1-phenyl-2-(2-thioxopyrrolidin-1-yl)ethanone (15a) (300 MHz, CDCl_3) **^{13}C NMR spectrum of 1-phenyl-2-(2-thioxopyrrolidin-1-yl)ethanone (15a) (75 MHz, CDCl_3)**

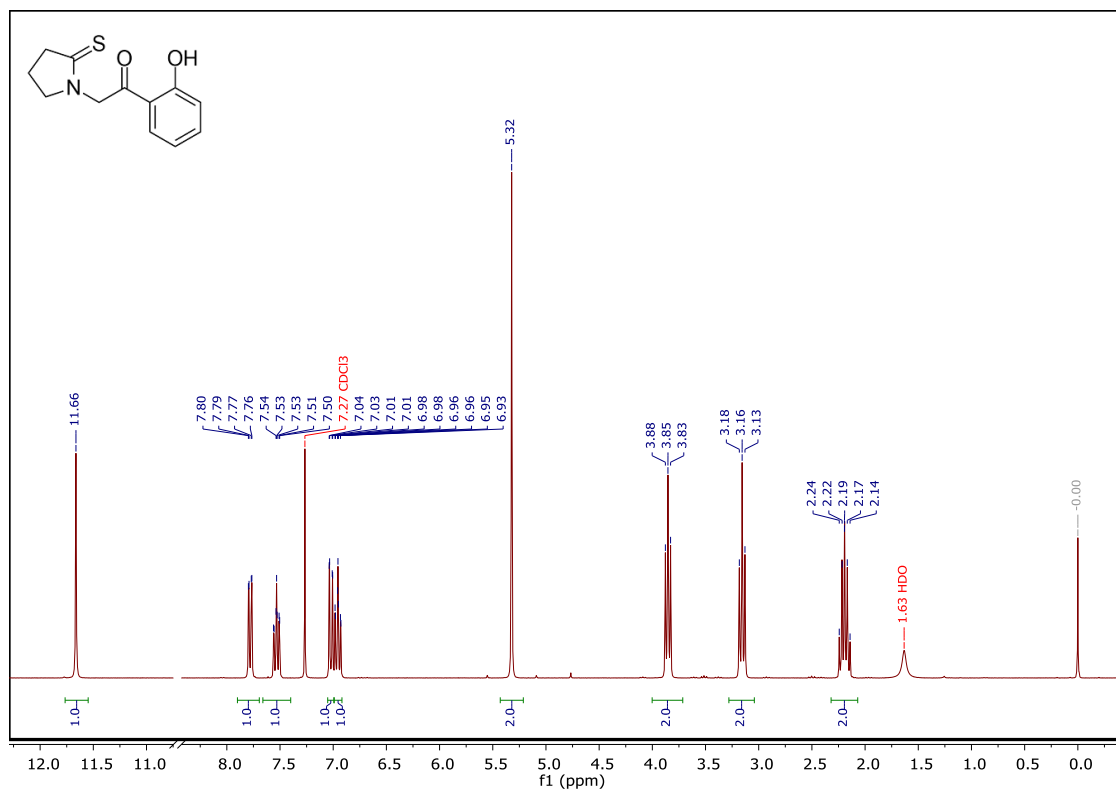
¹H NMR spectrum of 1-(3,4-dimethoxyphenyl)-2-(2-thioxopyrrolidin-1-yl)ethanone (15b)
(300 MHz, CDCl₃)



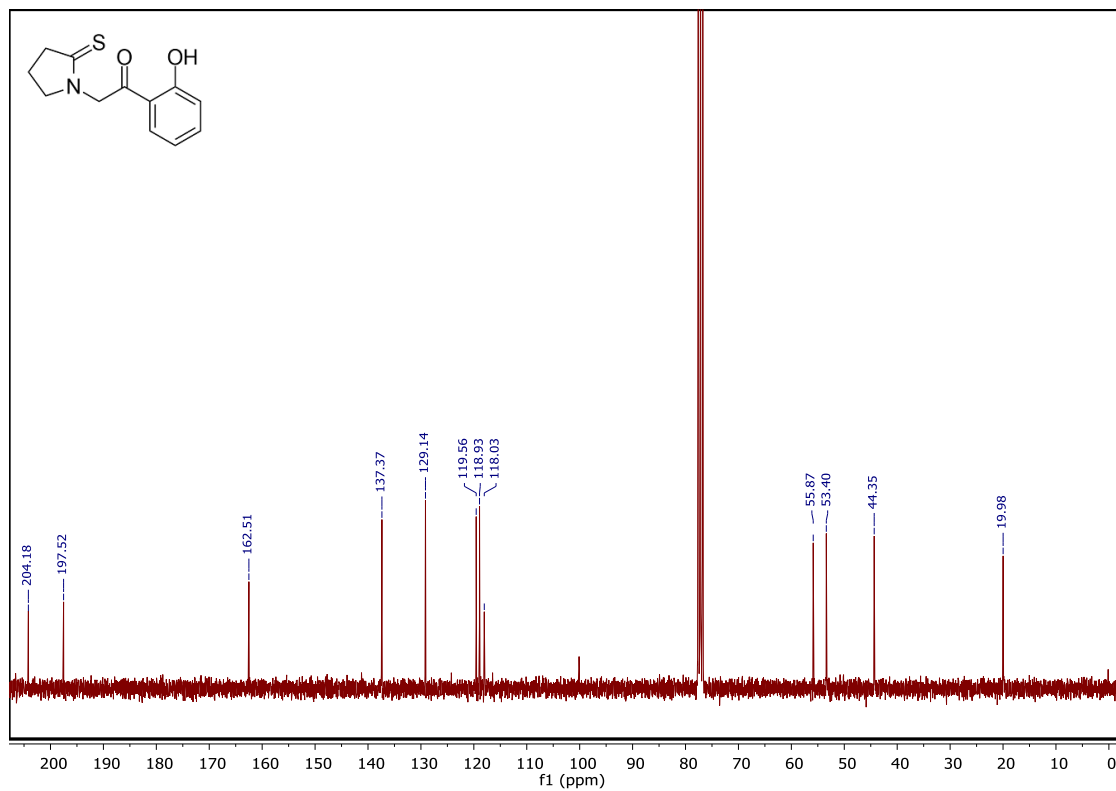
¹³C NMR spectrum of 1-(3,4-dimethoxyphenyl)-2-(2-thioxopyrrolidin-1-yl)ethanone (15b)
(75 MHz, CDCl₃)



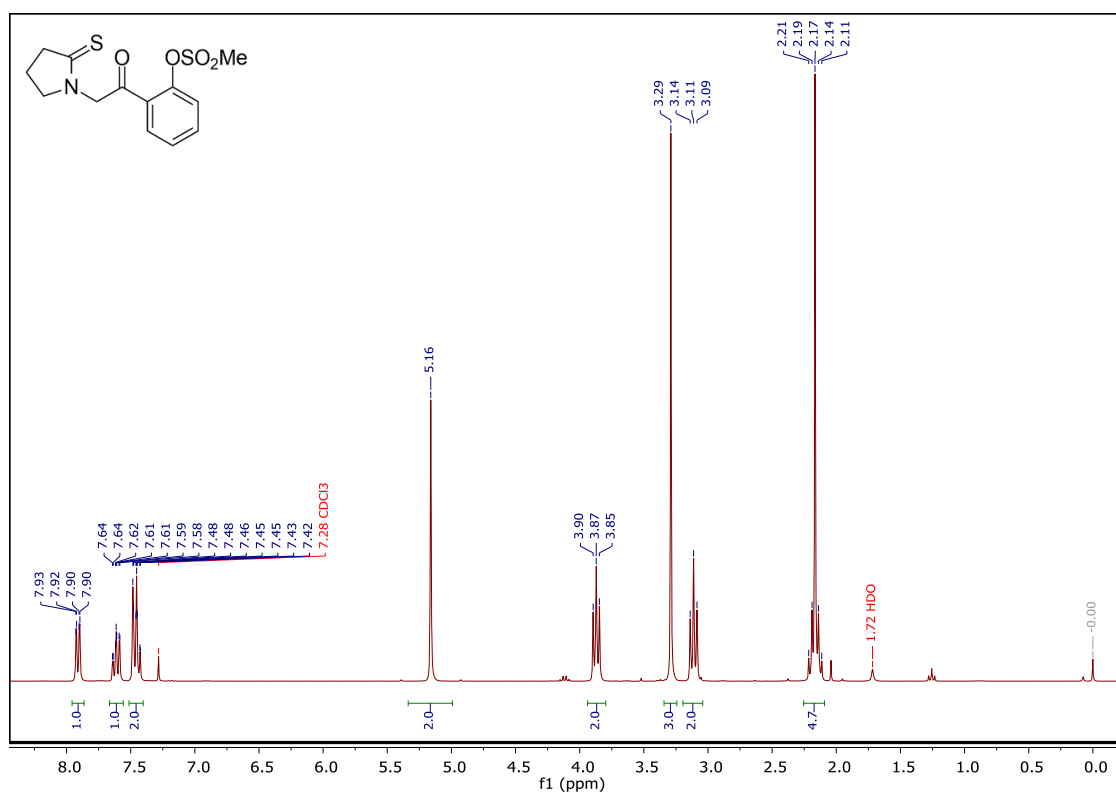
¹H NMR spectrum of 1-(2-hydroxyphenyl)-2-(2-thioxopyrrolidin-1-yl)ethanone (15c)
(300 MHz, CDCl₃)



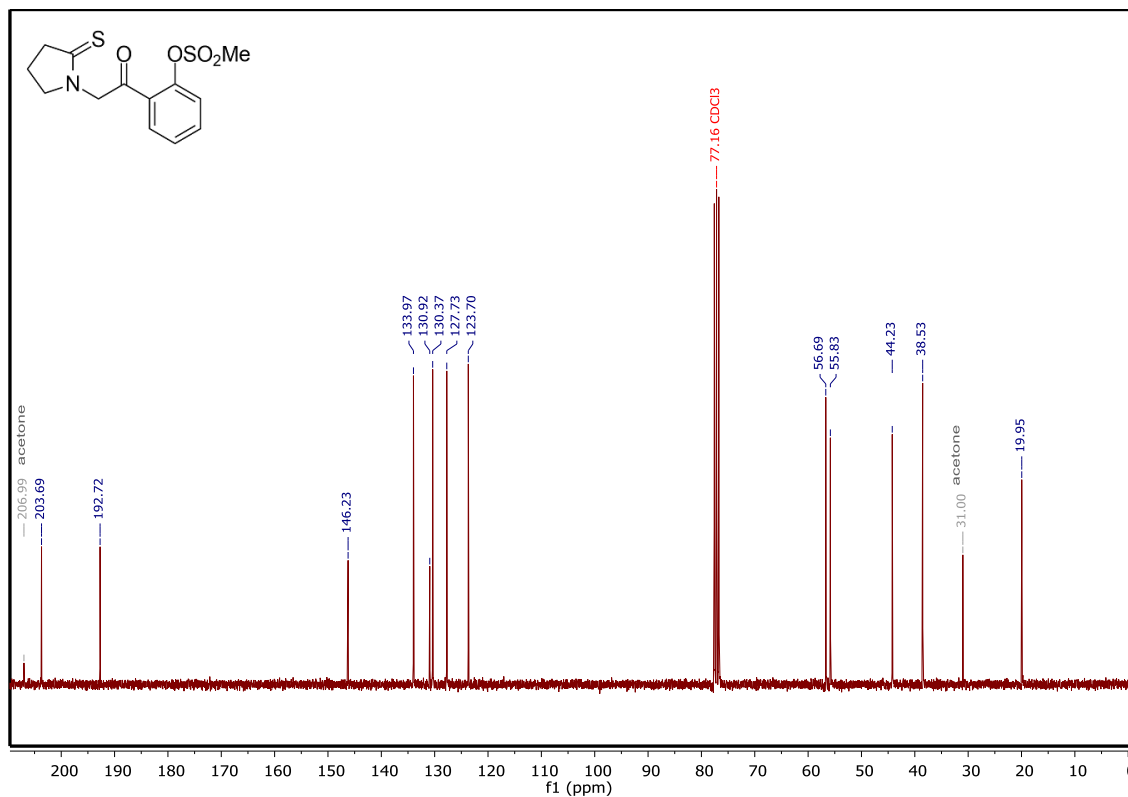
¹³C NMR spectrum of 1-(2-hydroxyphenyl)-2-(2-thioxopyrrolidin-1-yl)ethanone (15c)
(75 MHz, CDCl₃)



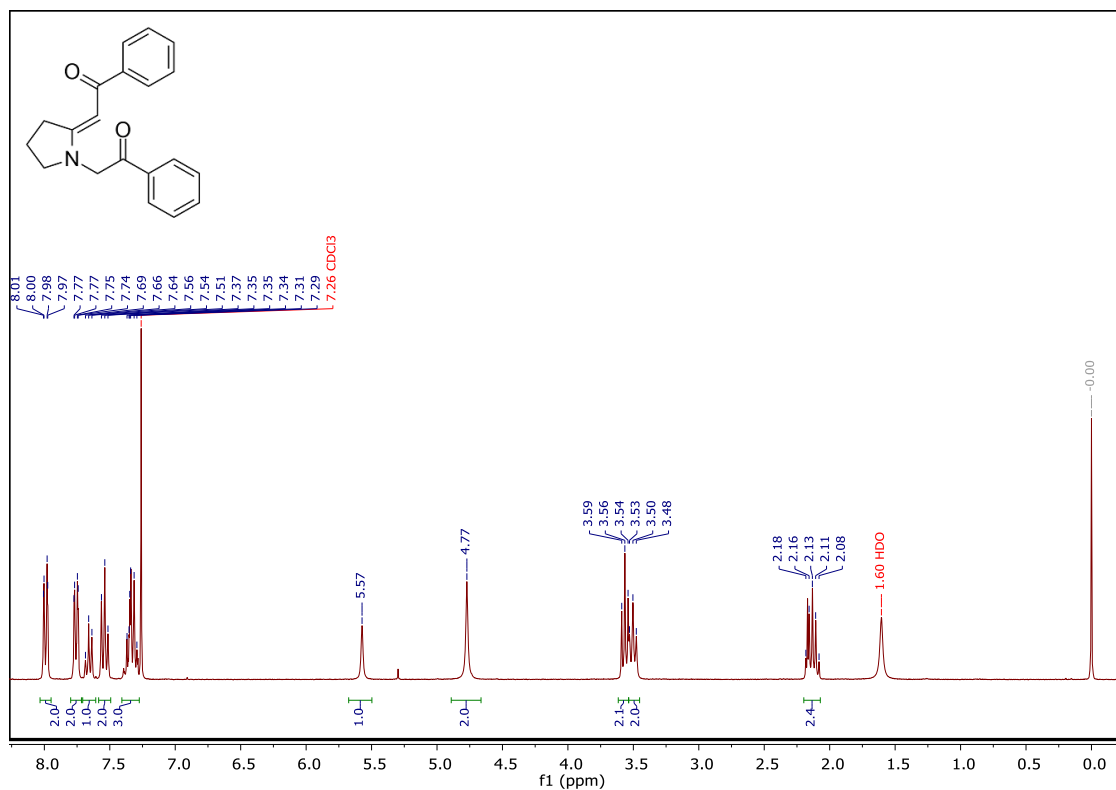
¹H NMR spectrum of 2-[2-(2-thioxopyrrolidin-1-yl)acetyl]phenyl methanesulfonate (15d)
(300 MHz, CDCl₃)



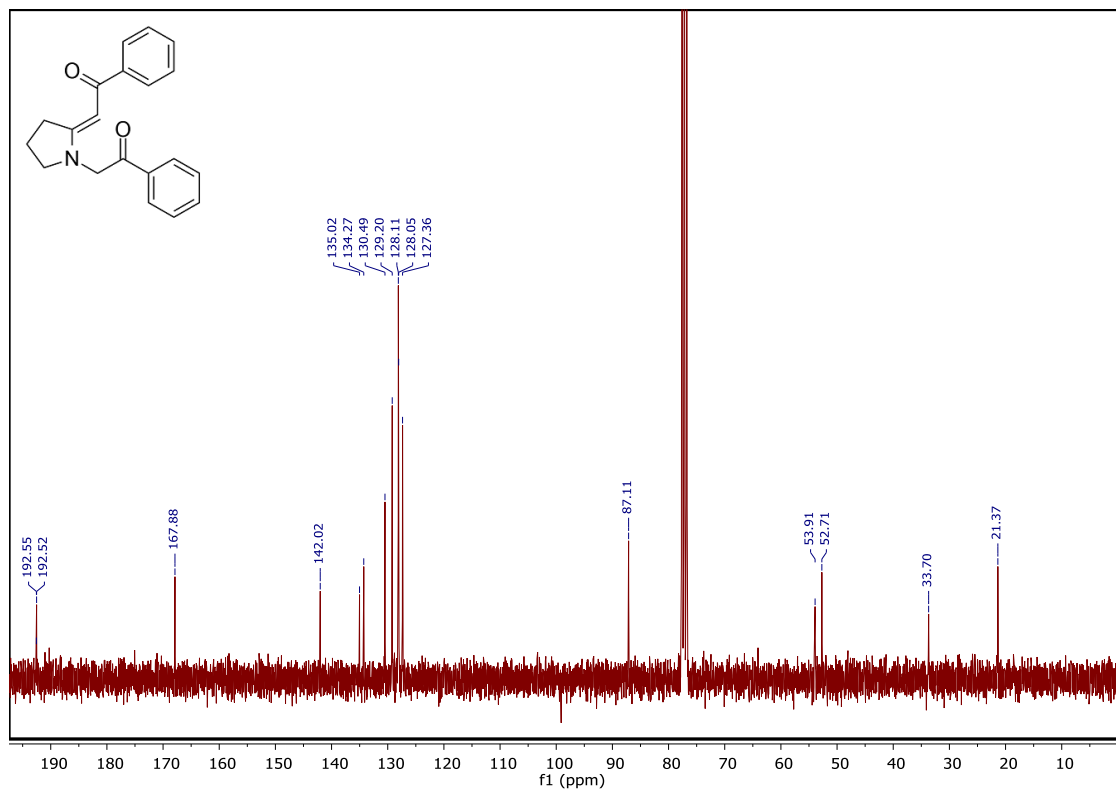
¹³C NMR spectrum of 2-[2-(2-thioxopyrrolidin-1-yl)acetyl]phenyl methanesulfonate (15d)
(75 MHz, CDCl₃)



¹H NMR spectrum of (*E*)-2-[1-(2-oxo-2-phenylethyl)pyrrolidin-2-ylidene]-1-phenylethanone (16a)
(300 MHz, CDCl₃)



¹³C NMR spectrum of (*E*)-2-[1-(2-oxo-2-phenylethyl)pyrrolidin-2-ylidene]-1-phenylethanone (16a)
(75 MHz, CDCl₃)



Chemical Structure of 10: COc1ccc(cc1)C(=O)CN2CCCC2C(=O)C3=CC(OC)=CC(OC)=C3

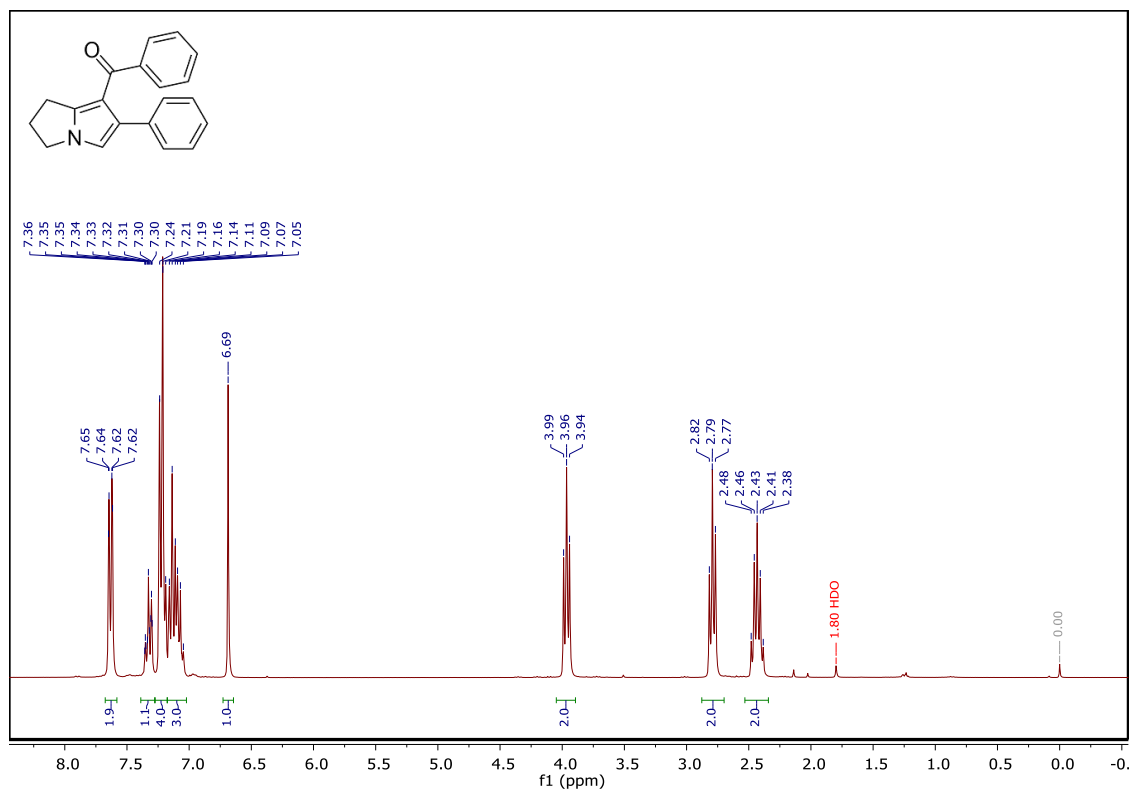
¹H NMR Spectrum (CDCl₃):

Chemical Shift (ppm)	Integration
7.63, 7.61, 7.60, 7.55, 7.54, 7.49, 7.48, 7.34, 7.33, 7.31, 7.30	1.11, 1.04, 1.07, 1.01, 1.01, 1.01, 1.01, 1.01, 1.01, 1.01
6.95, 6.93, 6.77, 6.74	1.01, 1.01, 1.01, 1.01
5.60	1.00
4.72	1.90
3.98, 3.94, 3.87, 3.86	3.00, 3.14, 3.14, 3.40
3.58, 3.56, 3.53, 3.51, 3.49, 3.46	2.01, 2.01, 2.01, 2.01, 2.01, 2.01
2.17, 2.14, 2.12, 2.09, 2.07	2.11, 2.11, 2.11, 2.11, 2.11
1.69 (H ₂ O)	-
0.00 (TMS)	-

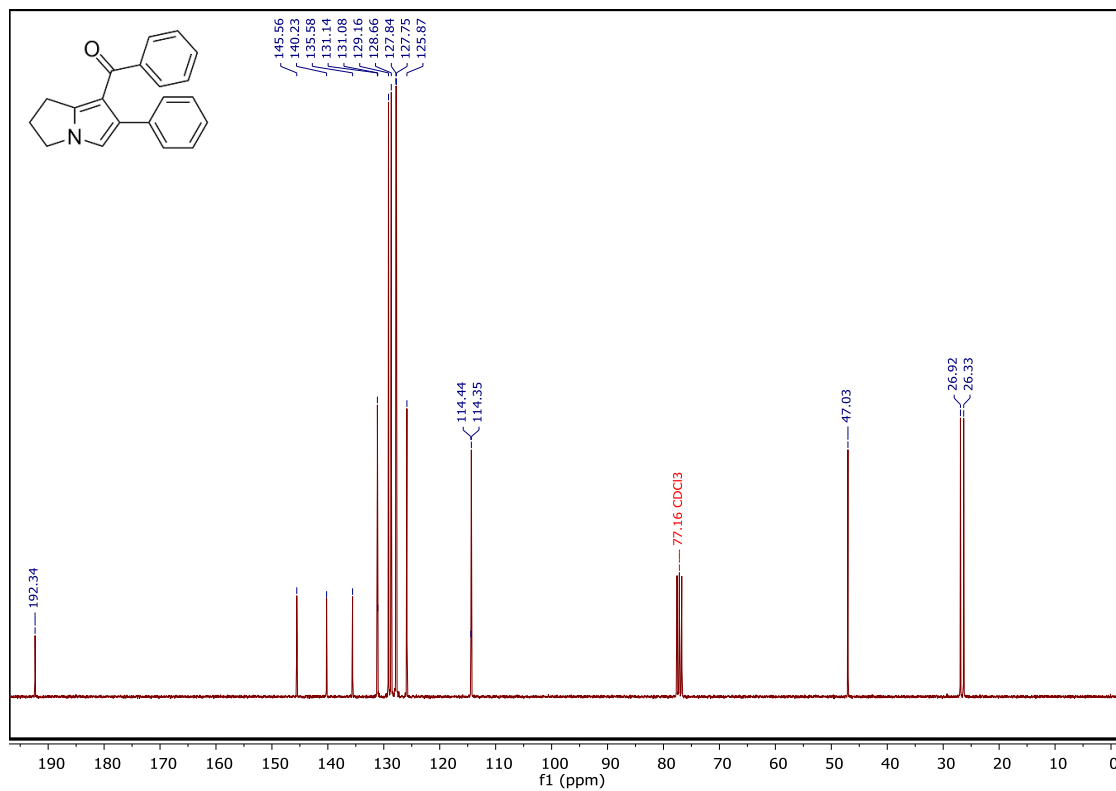
Chemical structure of compound 10a is shown above the spectrum. The spectrum displays peaks corresponding to the ^{13}C NMR data, with the following chemical shifts (ppm) labeled:

- 191.31
- 187.01
- 167.45
- 154.29
- 151.22
- 148.92
- 148.76
- 134.91
- 128.25
- 122.54
- 120.52
- 110.60
- 110.39
- 110.21
- 109.92
- 86.59
- 56.34
- 56.20
- 56.04
- 55.94
- 53.87
- 52.34
- 33.61
- 21.41

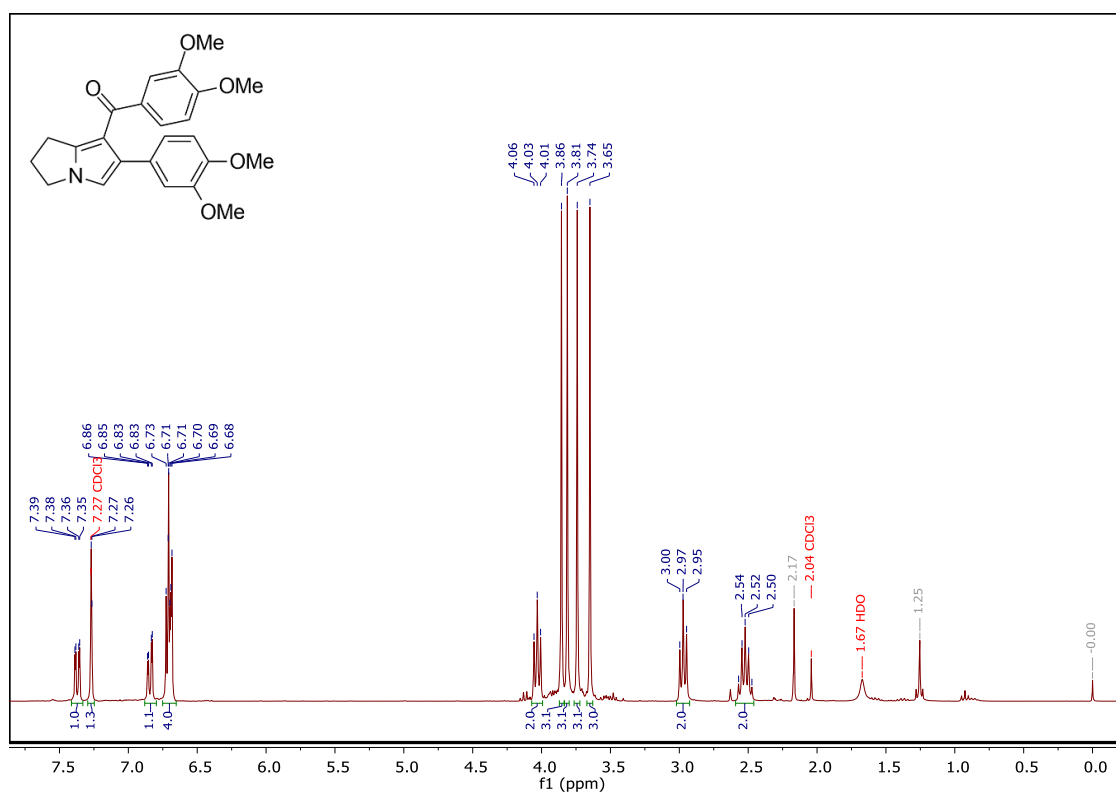
¹H NMR spectrum of phenyl(6-phenyl-2,3-dihydro-1H-pyrrolizin-7-yl)methanone (17a)
(300 MHz, CDCl₃)



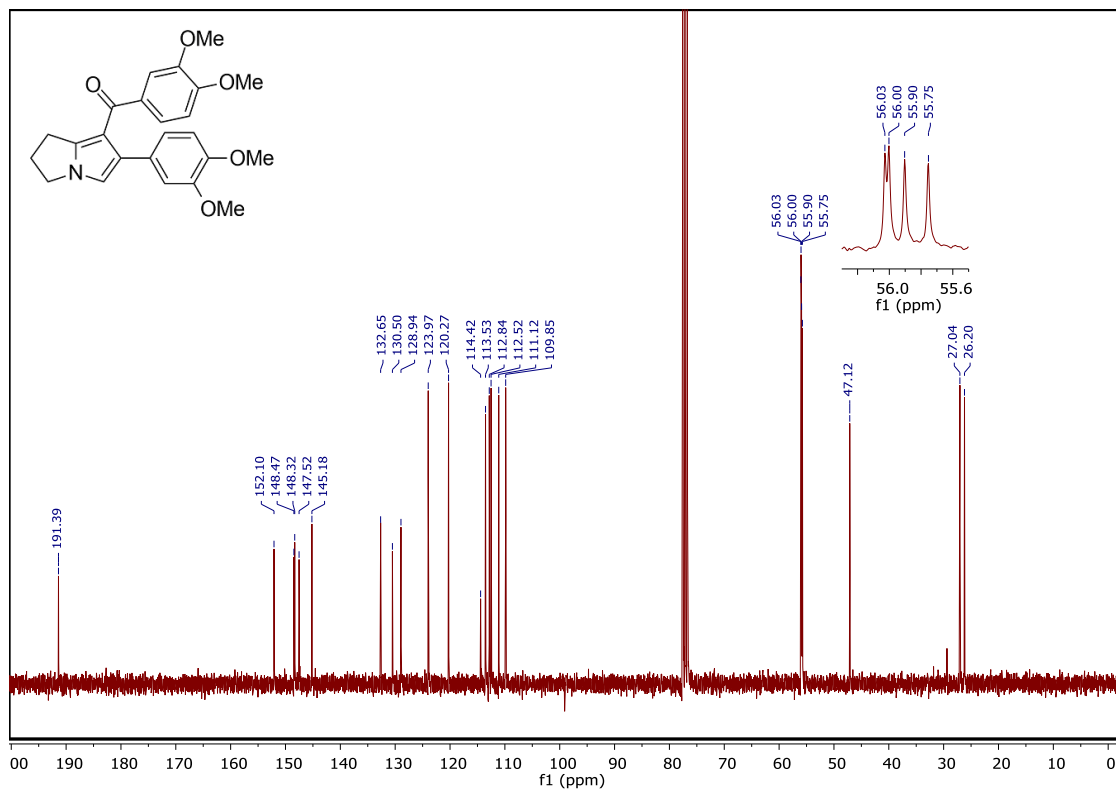
¹³C NMR spectrum of phenyl(6-phenyl-2,3-dihydro-1H-pyrrolizin-7-yl)methanone (17a)
(75 MHz, CDCl₃)



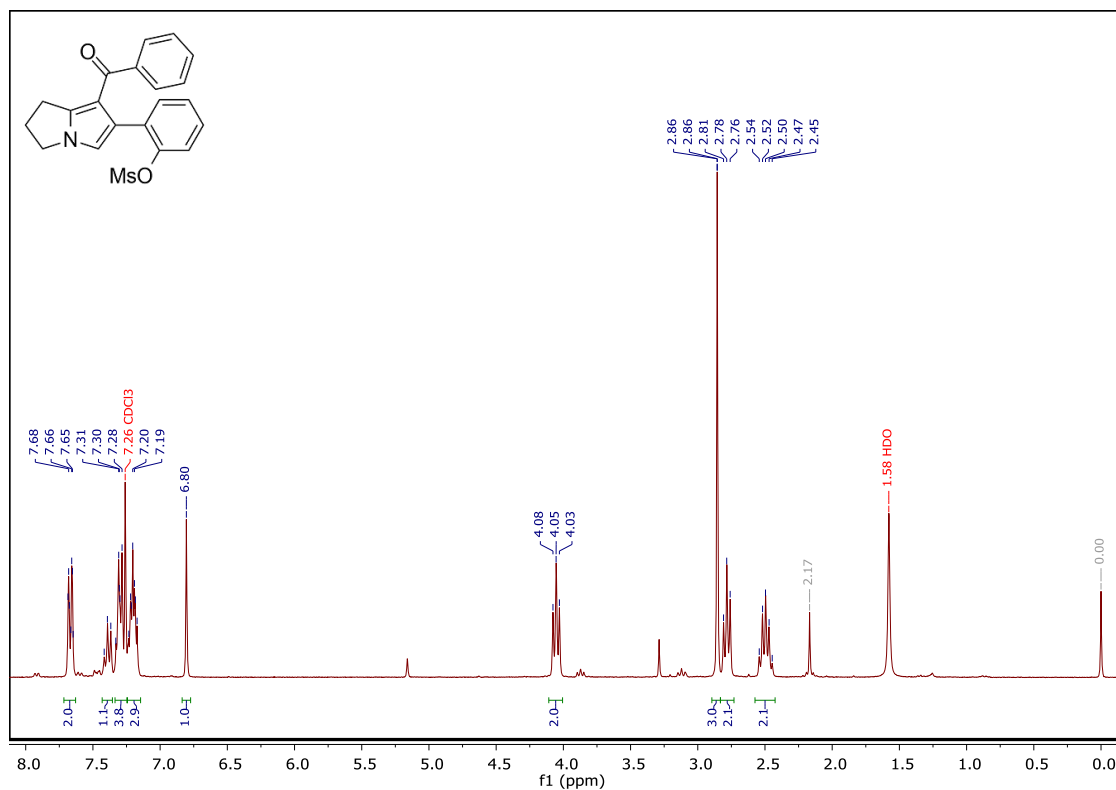
¹H NMR spectrum of (3,4-dimethoxyphenyl)[6-(3,4-dimethoxyphenyl)-2,3-dihydro-1H-pyrrolizin-7-yl]methanone (17b) (300 MHz, CDCl₃)



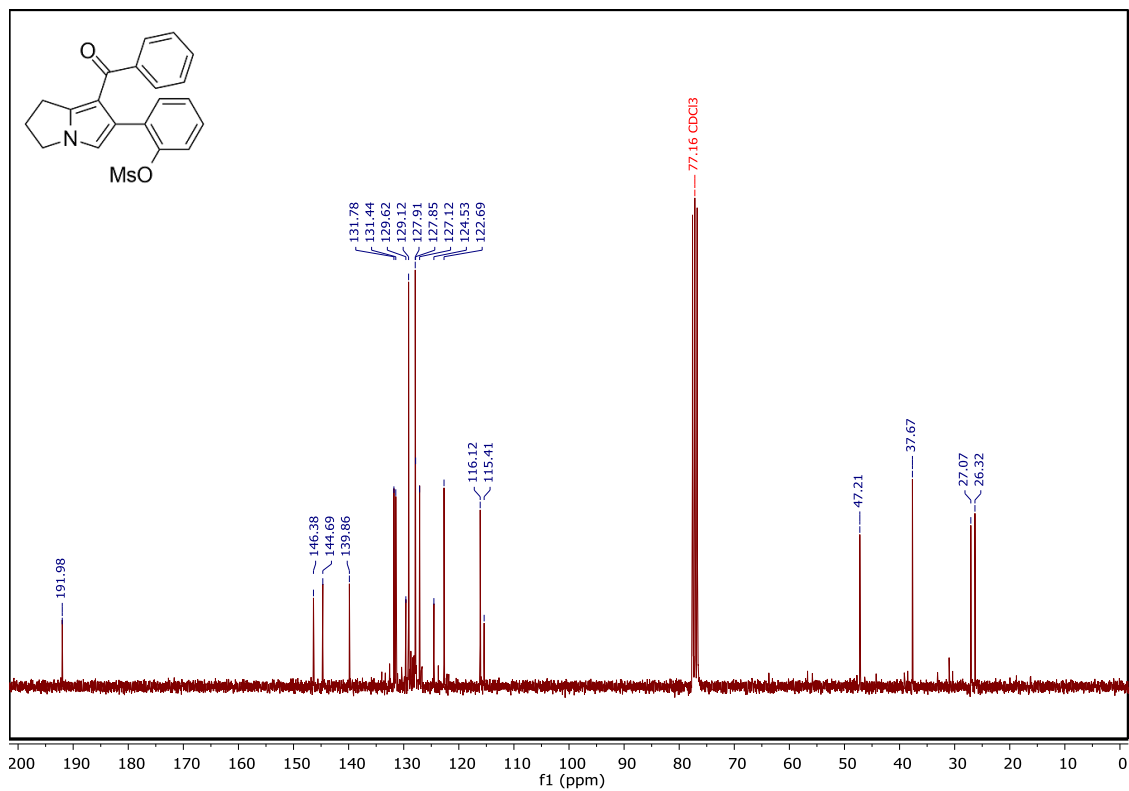
¹³C NMR spectrum of (3,4-dimethoxyphenyl)[6-(3,4-dimethoxyphenyl)-2,3-dihydro-1H-pyrrolizin-7-yl]methanone (17b) (75 MHz, CDCl₃)



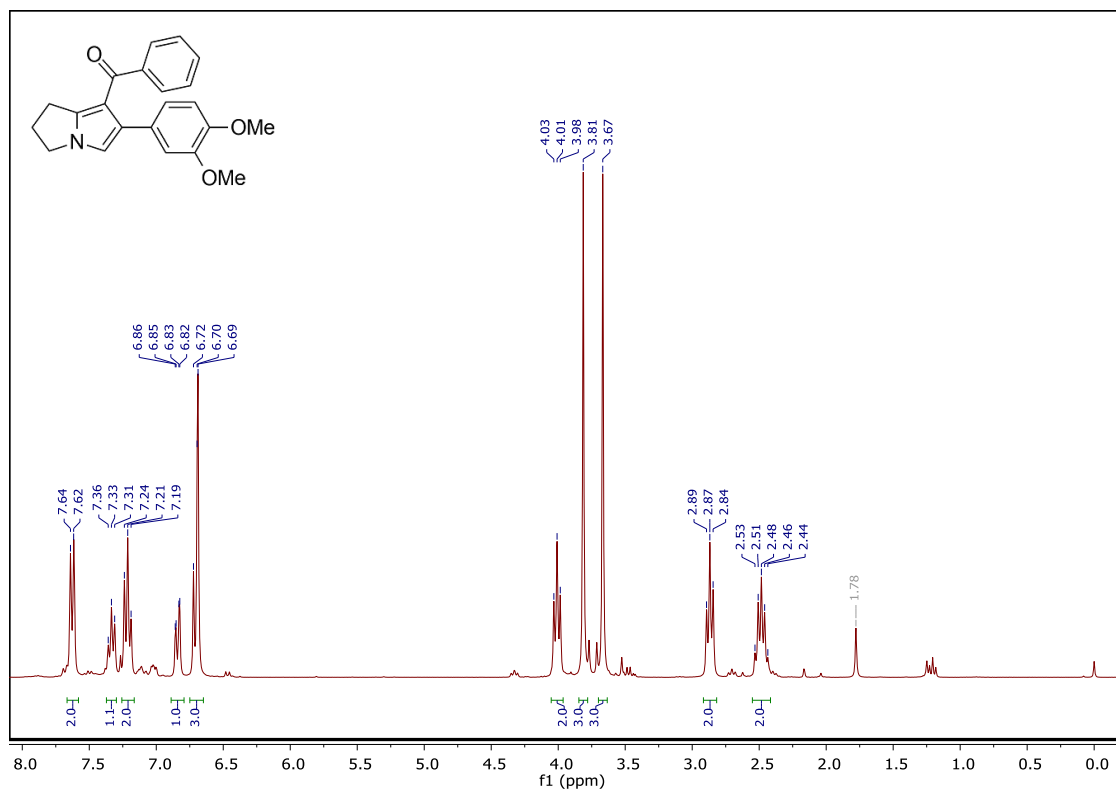
¹H NMR spectrum of 2-(7-benzoyl-2,3-dihydro-1H-pyrrolizin-6-yl)phenyl methanesulfonate (17d)
(300 MHz, CDCl₃)



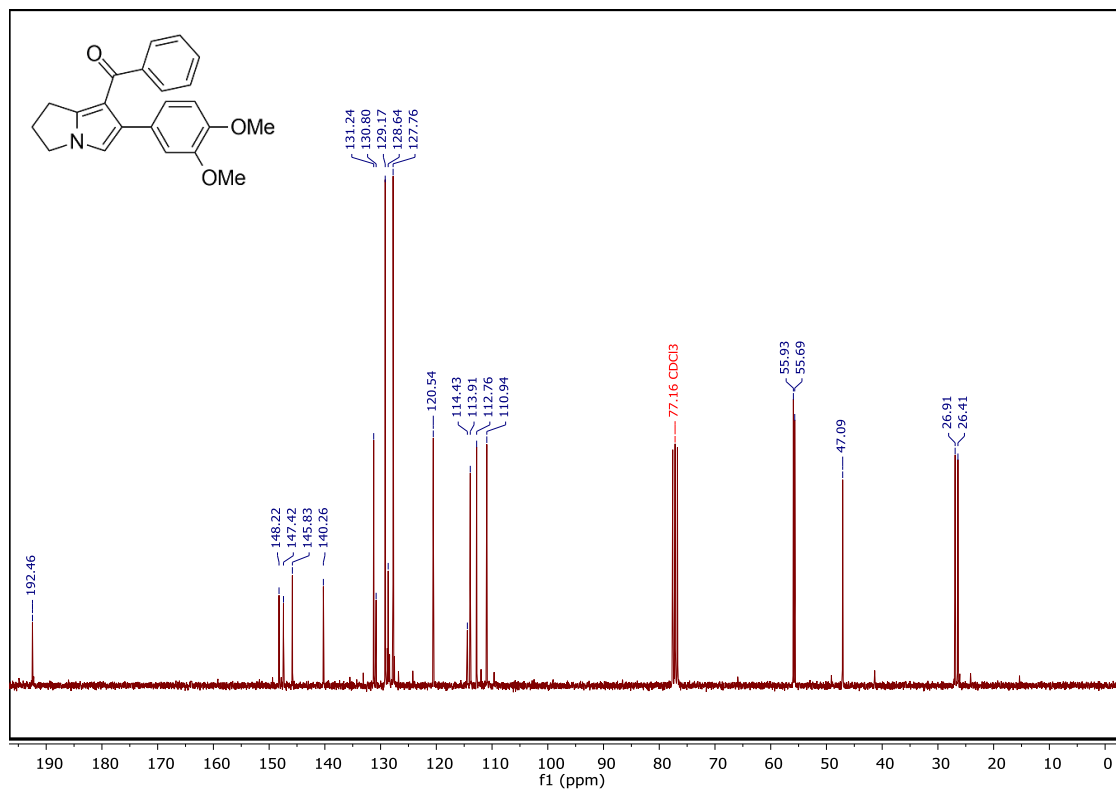
¹³C NMR spectrum of 2-(7-benzoyl-2,3-dihydro-1H-pyrrolizin-6-yl)phenyl methanesulfonate (17d)
(75 MHz, CDCl₃)



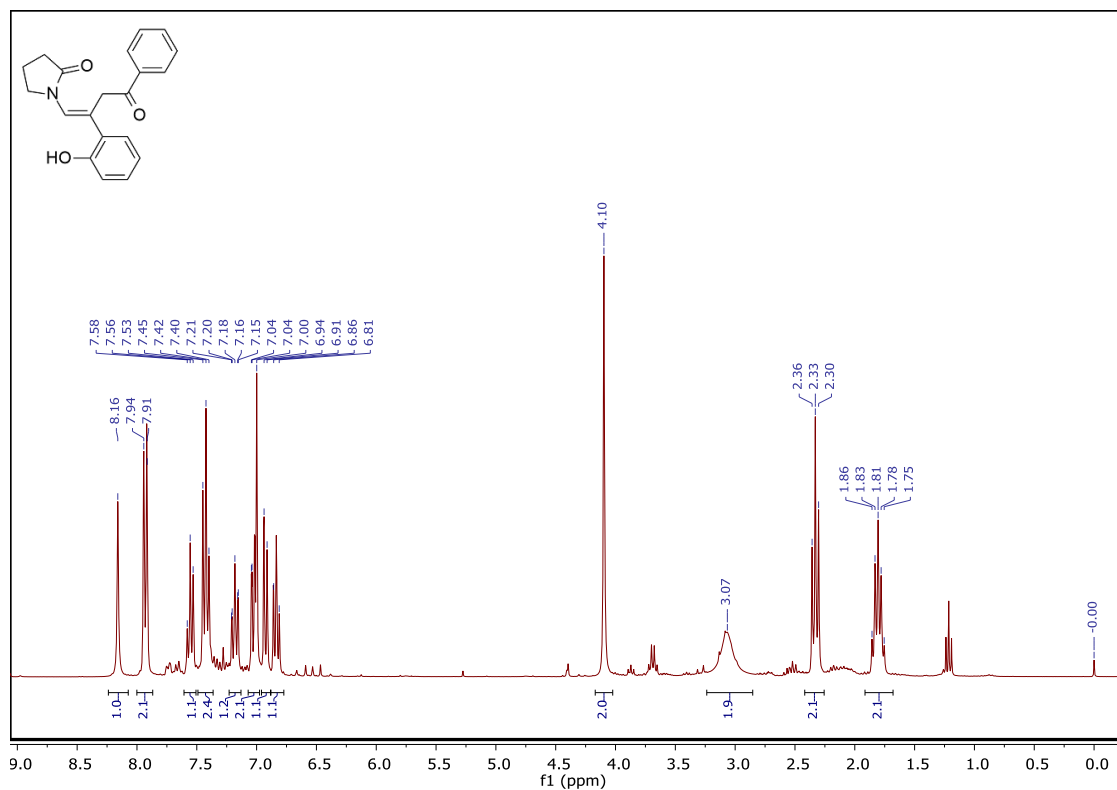
^1H NMR spectrum of [6-(3,4-dimethoxyphenyl)-2,3-dihydro-1H-pyrrolizin-7-yl](phenyl)methanone (19)
(300 MHz, CDCl_3)



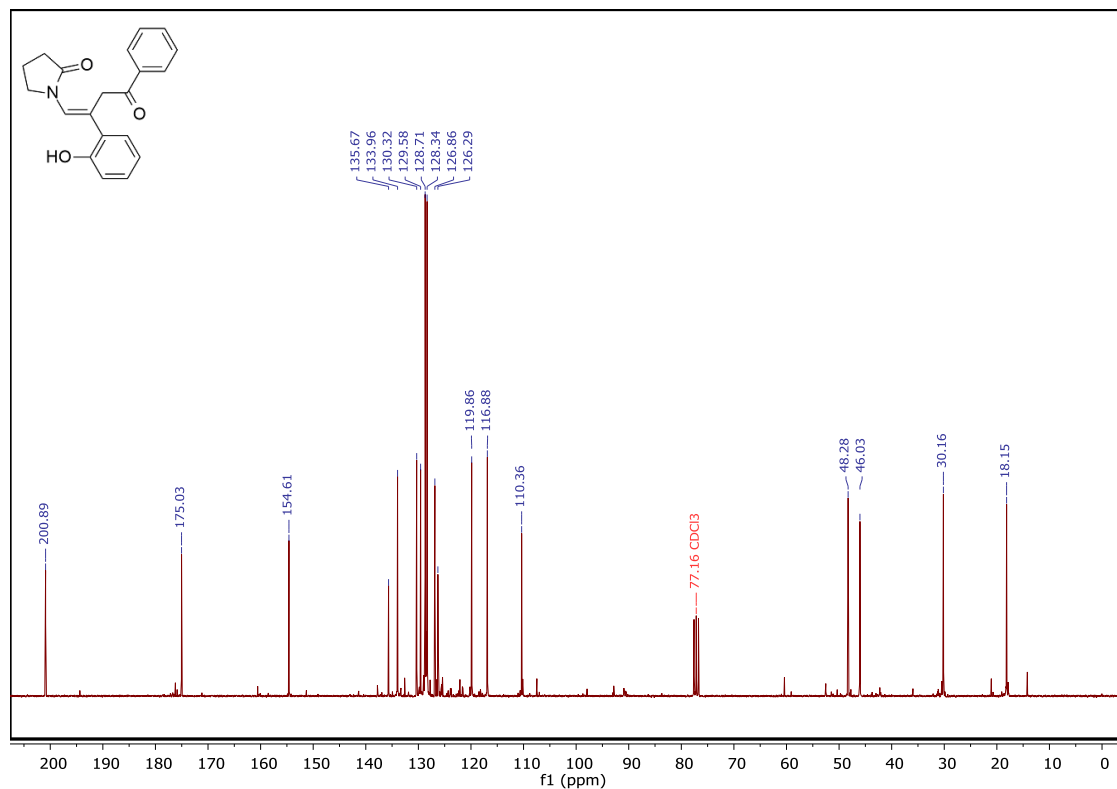
^{13}C NMR spectrum of [6-(3,4-dimethoxyphenyl)-2,3-dihydro-1H-pyrrolizin-7-yl](phenyl)methanone (19)
(75 MHz, CDCl_3)

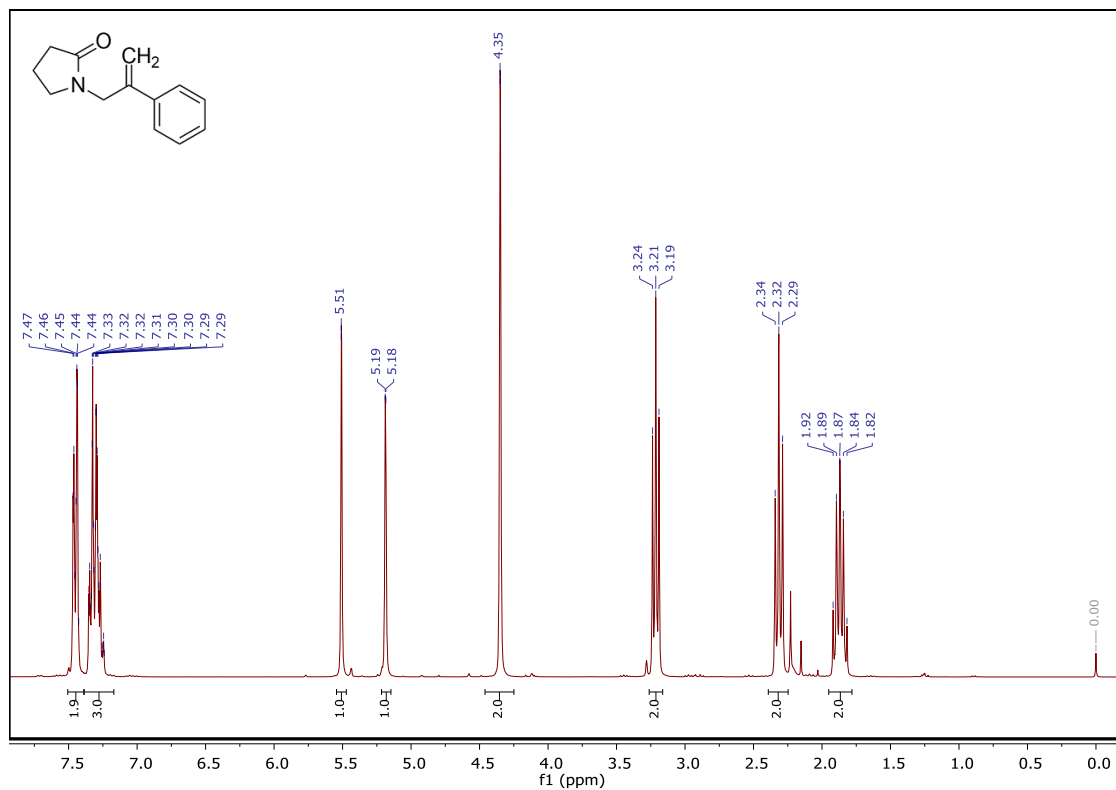
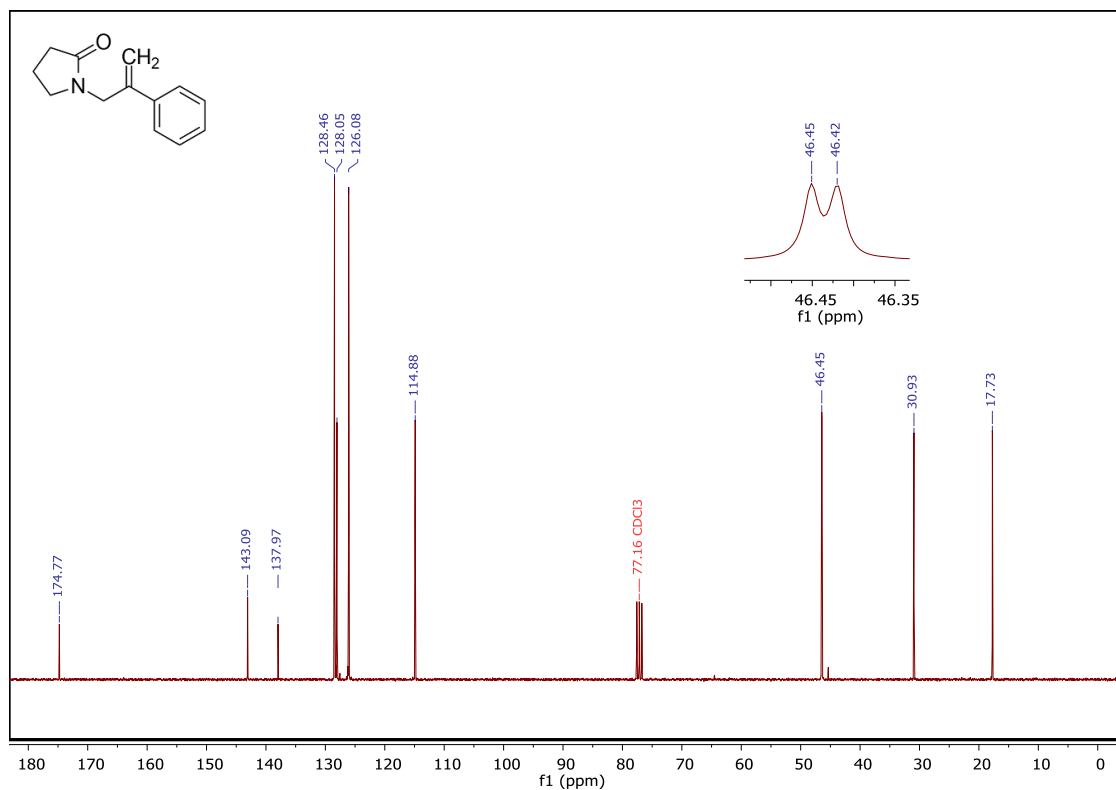


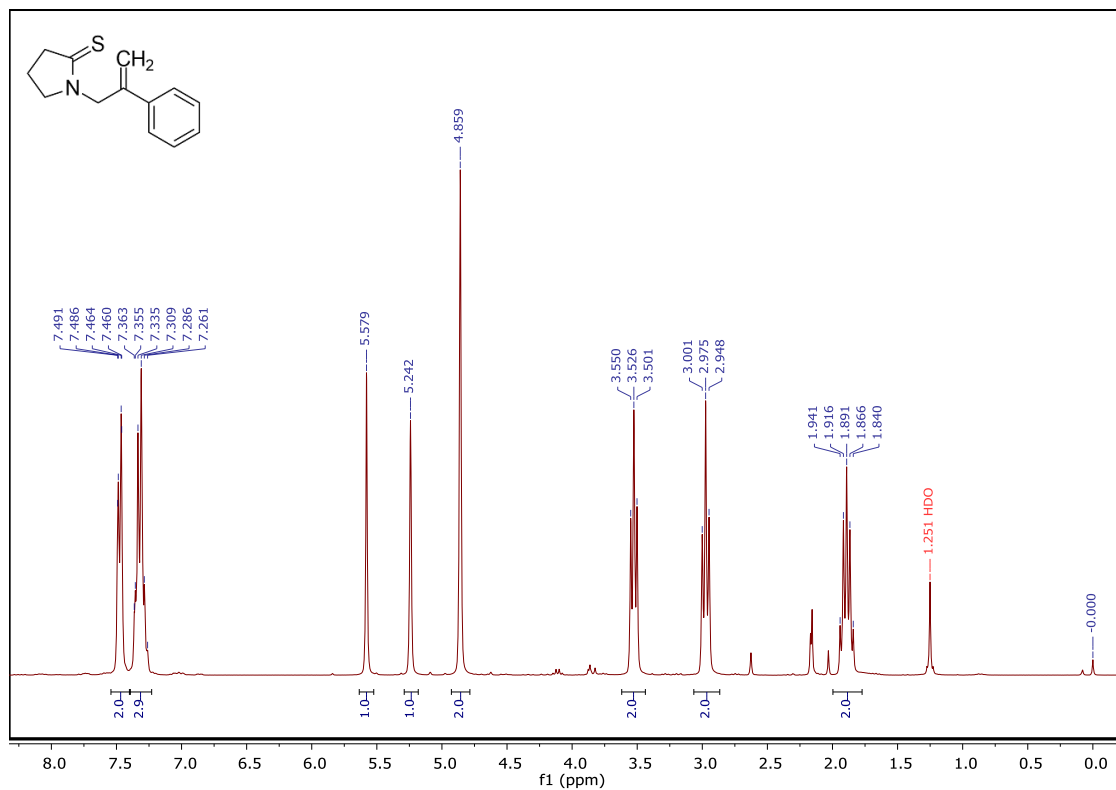
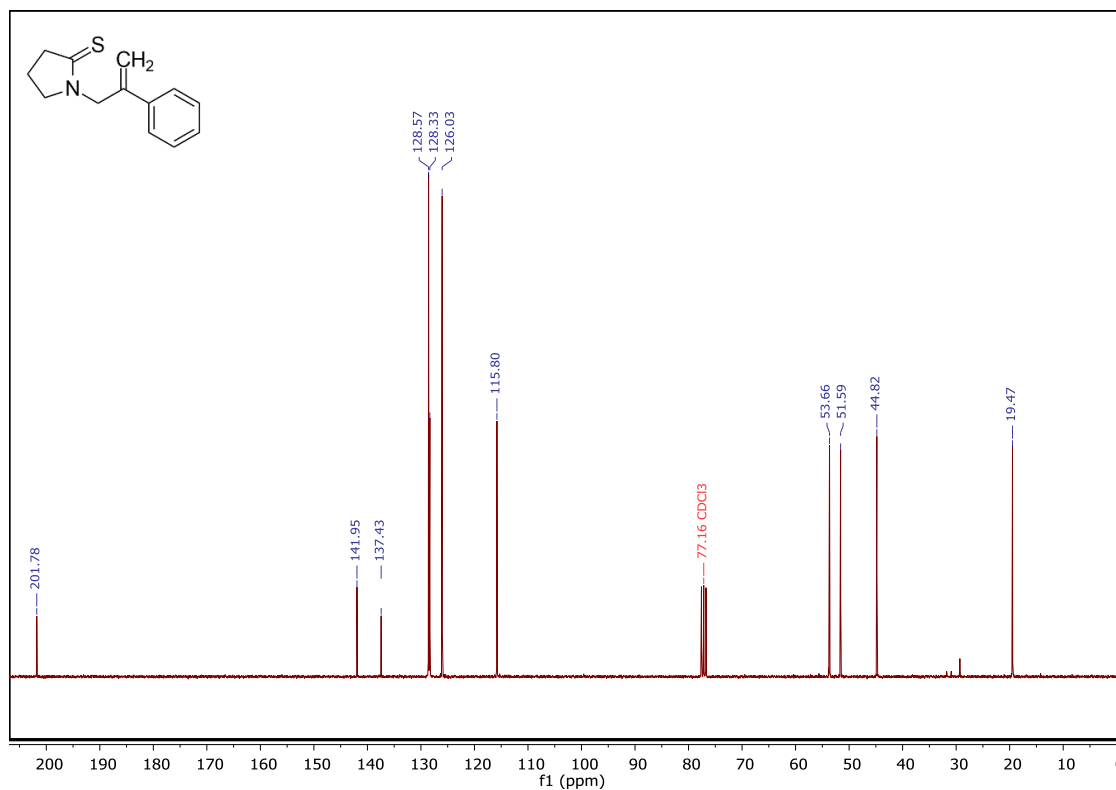
^1H NMR spectrum of 1-[(*E*)-2-(2-hydroxyphenyl)-4-oxo-4-phenyl-1-butenyl]pyrrolidin-2-one (20) (300 MHz, CDCl_3)



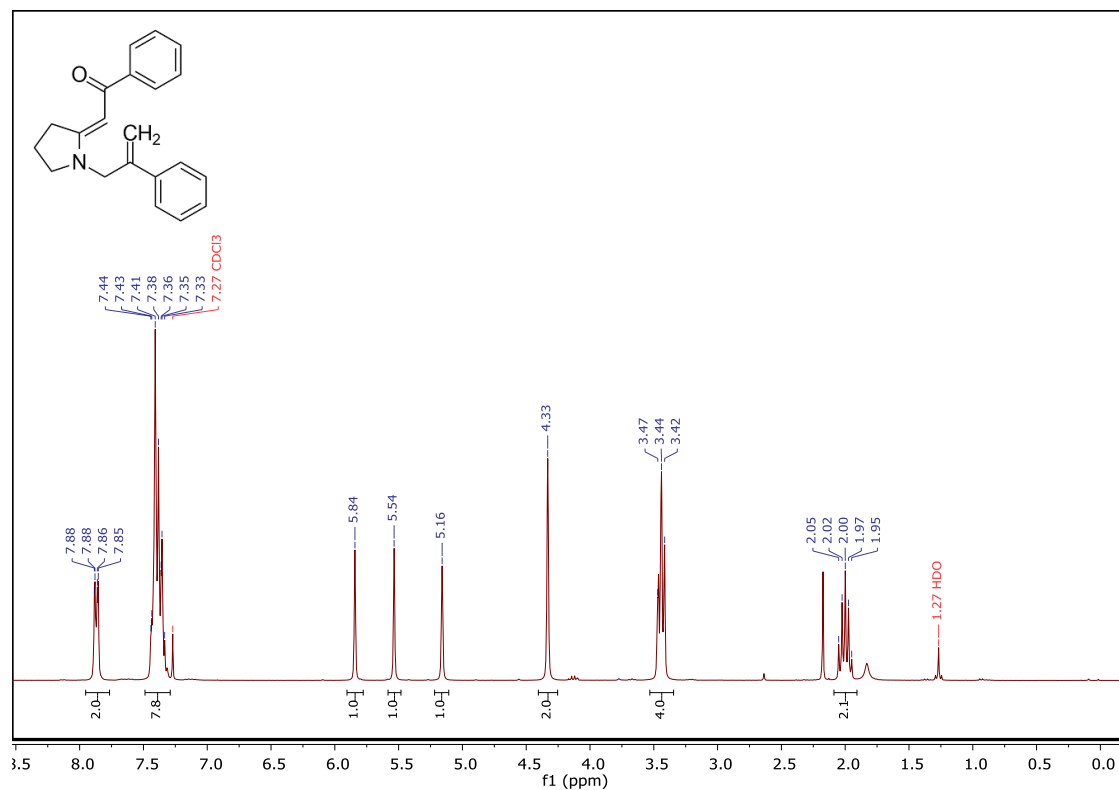
^{13}C NMR spectrum of 1-[(*E*)-2-(2-hydroxyphenyl)-4-oxo-4-phenyl-1-butenyl]pyrrolidin-2-one (20) (75 MHz, CDCl_3)



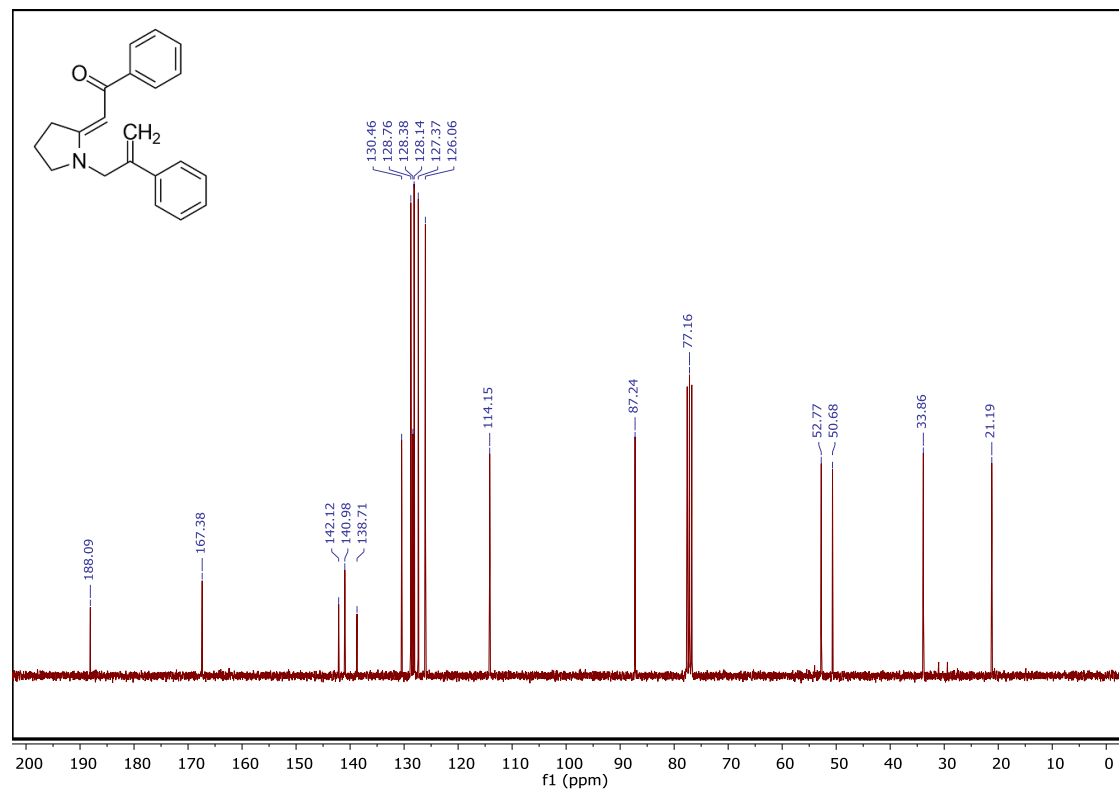
¹H NMR spectrum of 1-(2-phenyl-2-propenyl)pyrrolidin-2-one (27) (300 MHz, CDCl₃)**¹³C NMR spectrum of 1-(2-phenyl-2-propenyl)pyrrolidin-2-one (27) (75 MHz, CDCl₃)**

^1H NMR spectrum of 1-(2-phenyl-2-propenyl)pyrrolidine-2-thione (29) (300 MHz, CDCl_3) **^{13}C NMR spectrum of 1-(2-phenyl-2-propenyl)pyrrolidine-2-thione (29) (75 MHz, CDCl_3)**

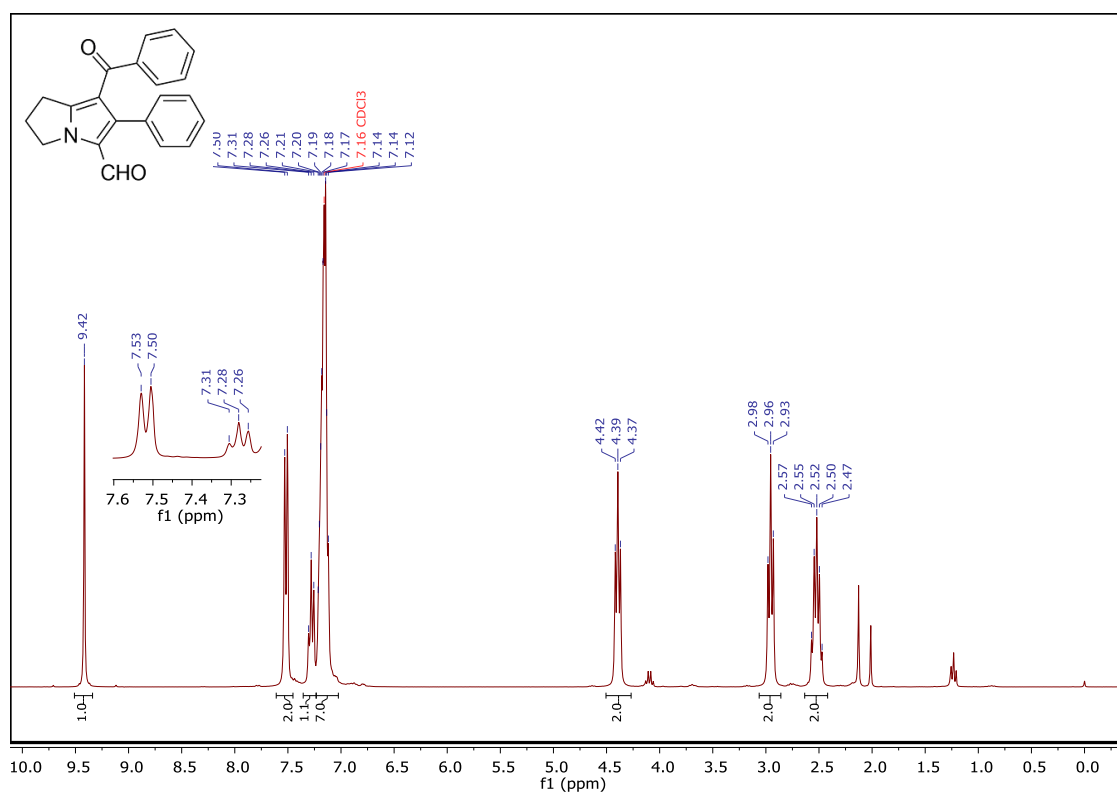
¹H NMR spectrum of (*E*)-1-phenyl-2-[1-(2-phenyl-2-propenyl)pyrrolidin-2-ylidene]ethanone (30)
(300 MHz, CDCl₃)



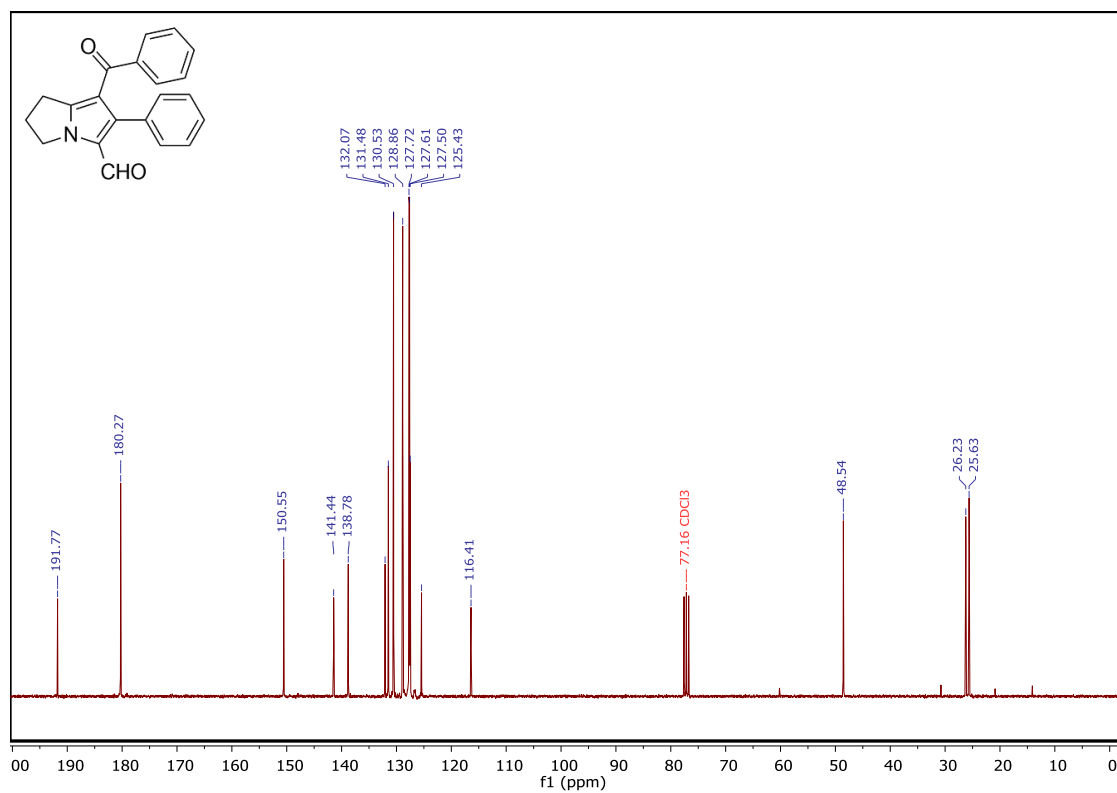
¹³C NMR spectrum of (*E*)-1-phenyl-2-[1-(2-phenyl-2-propenyl)pyrrolidin-2-ylidene]ethanone (30)
(75 MHz, CDCl₃)



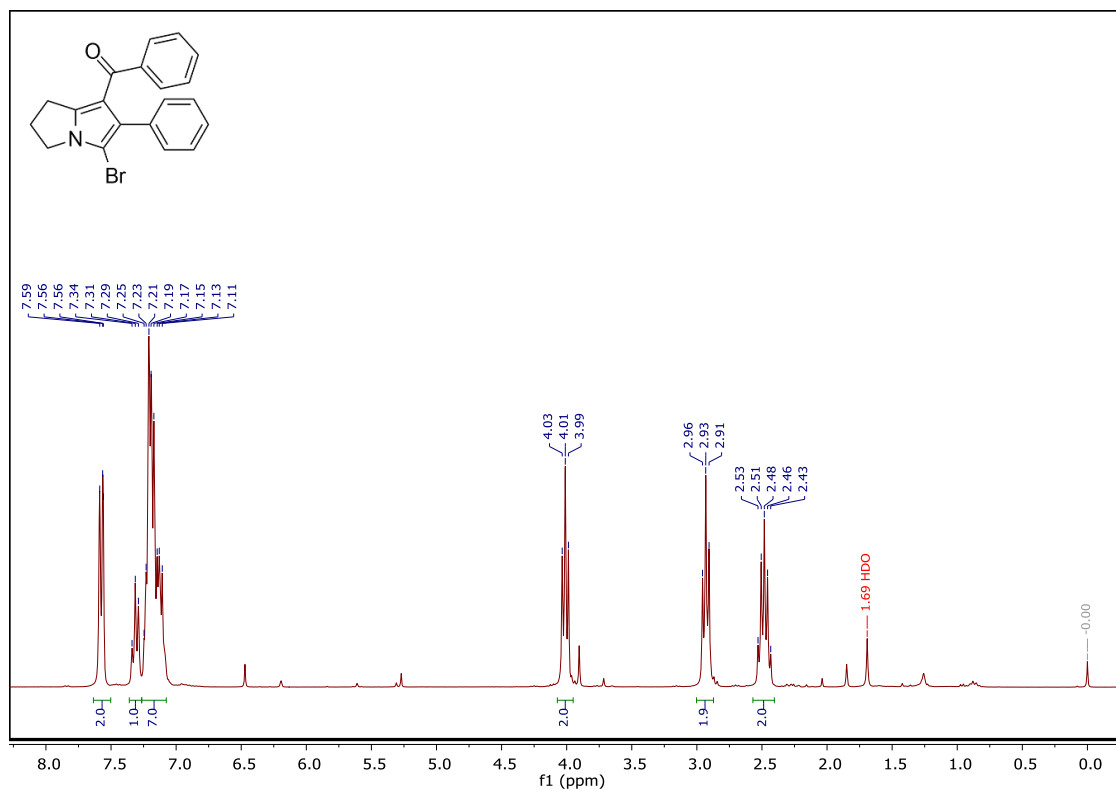
¹H NMR spectrum of 7-benzoyl-6-phenyl-2,3-dihydro-1H-pyrrolizine-5-carbaldehyde (32)
(300 MHz, CDCl₃)



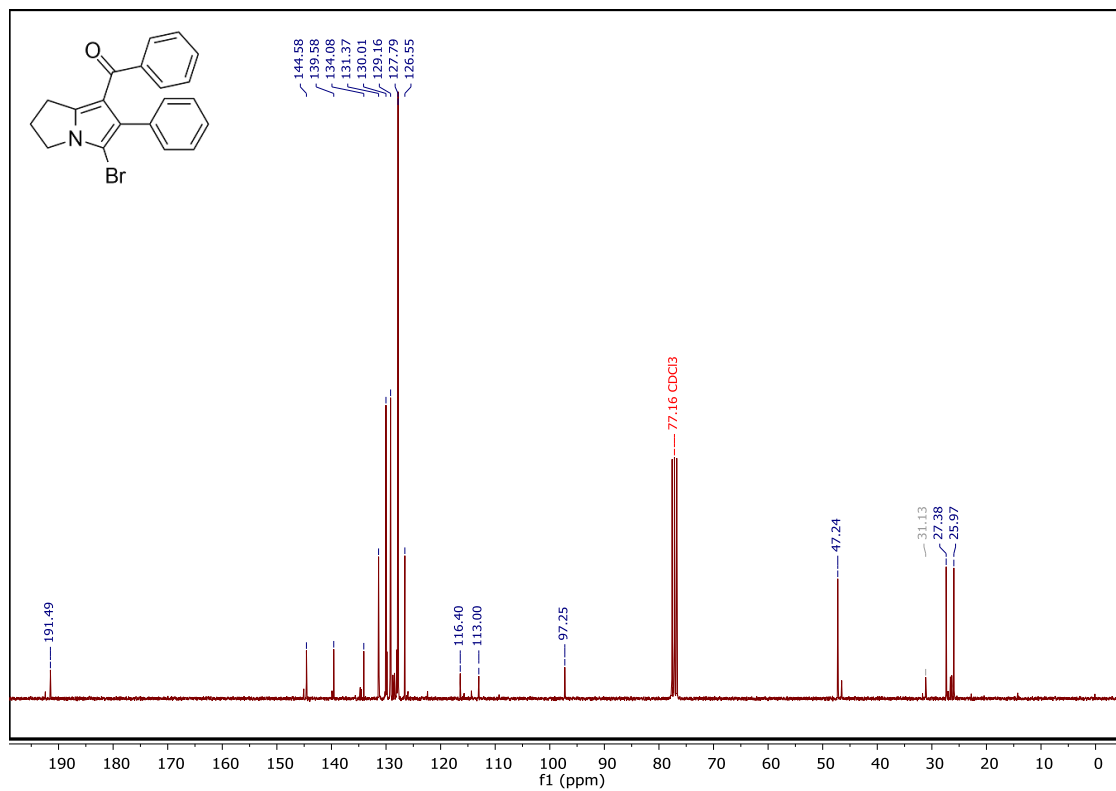
¹³C NMR spectrum of 7-benzoyl-6-phenyl-2,3-dihydro-1H-pyrrolizine-5-carbaldehyde (32)
(75 MHz, CDCl₃)



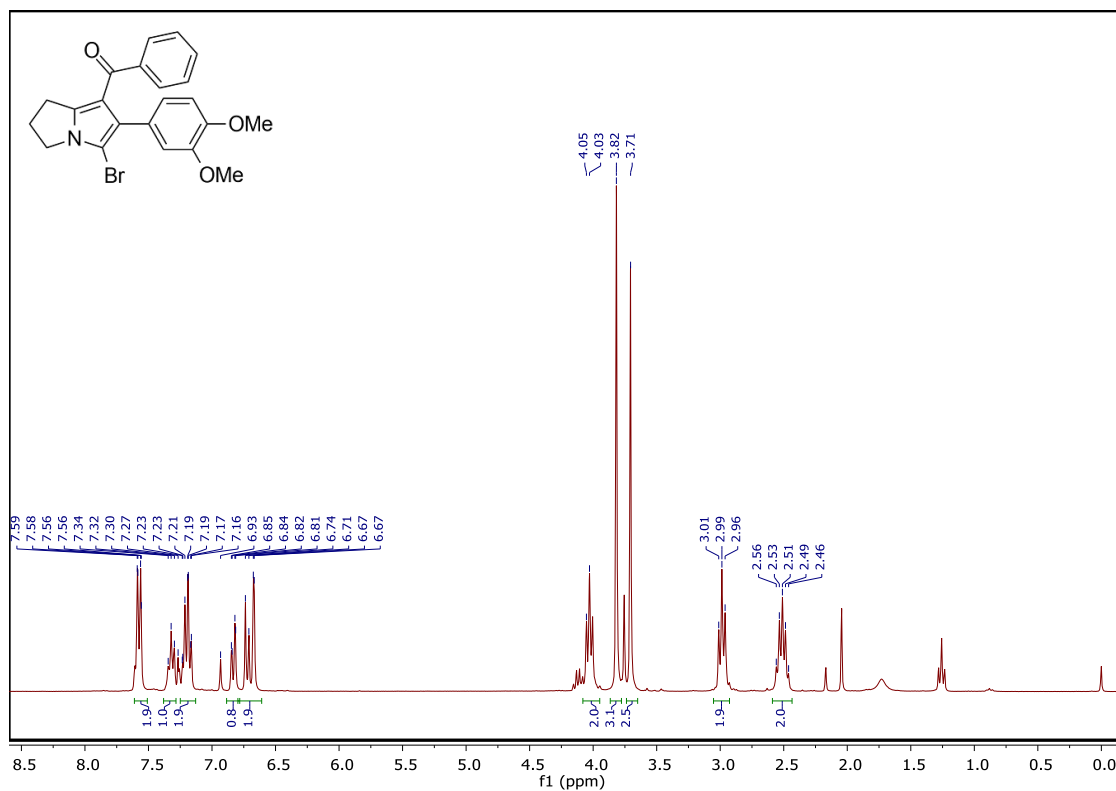
^1H NMR spectrum of (5-bromo-6-phenyl-2,3-dihydro-1*H*-pyrrolizin-7-yl)(phenyl)methanone (33a)
(300 MHz, CDCl_3)



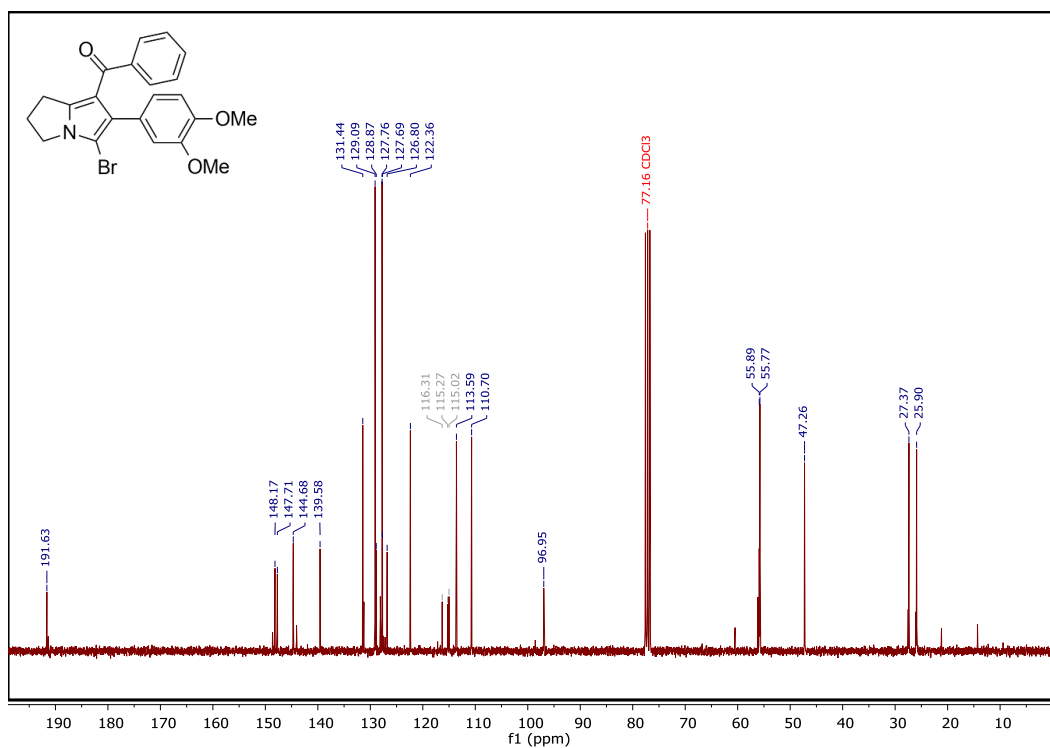
^{13}C NMR spectrum of (5-bromo-6-phenyl-2,3-dihydro-1*H*-pyrrolizin-7-yl)(phenyl)methanone (33a)
(75 MHz, CDCl_3)



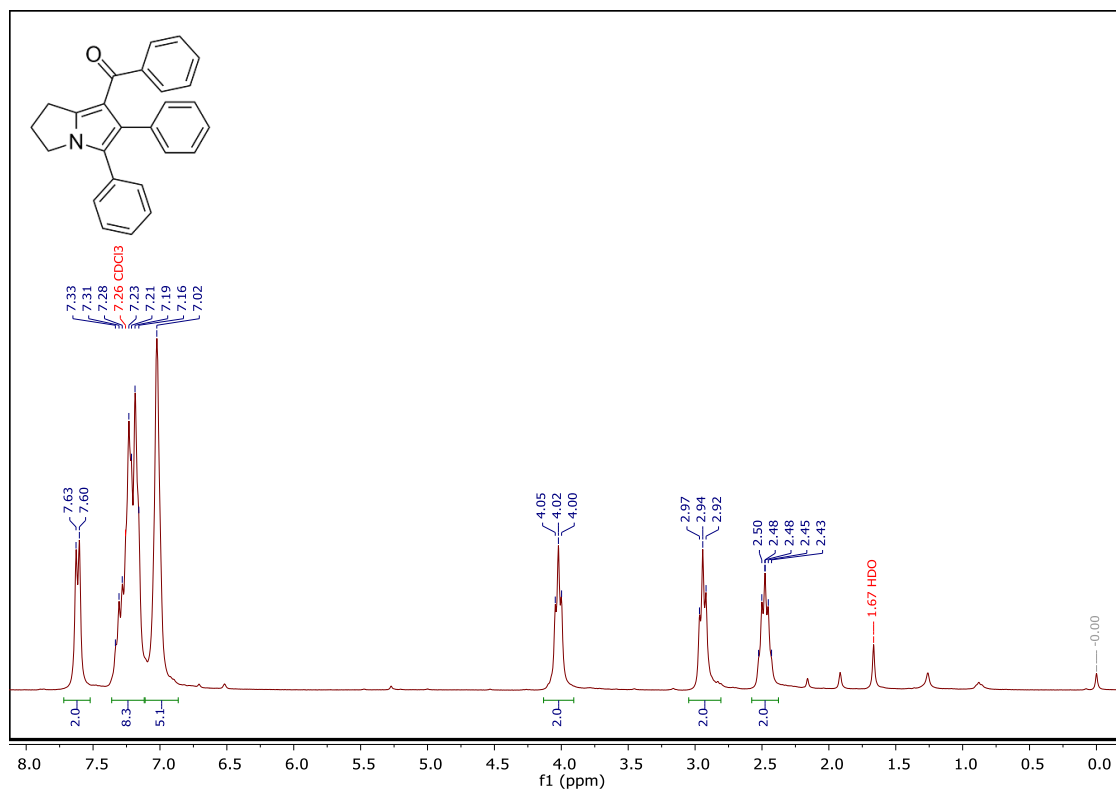
^1H NMR spectrum of [5-bromo-6-(3,4-dimethoxyphenyl)-2,3-dihydro-1H-pyrrolizin-7-yl](phenyl)methanone (33b)
(300 MHz, CDCl_3)



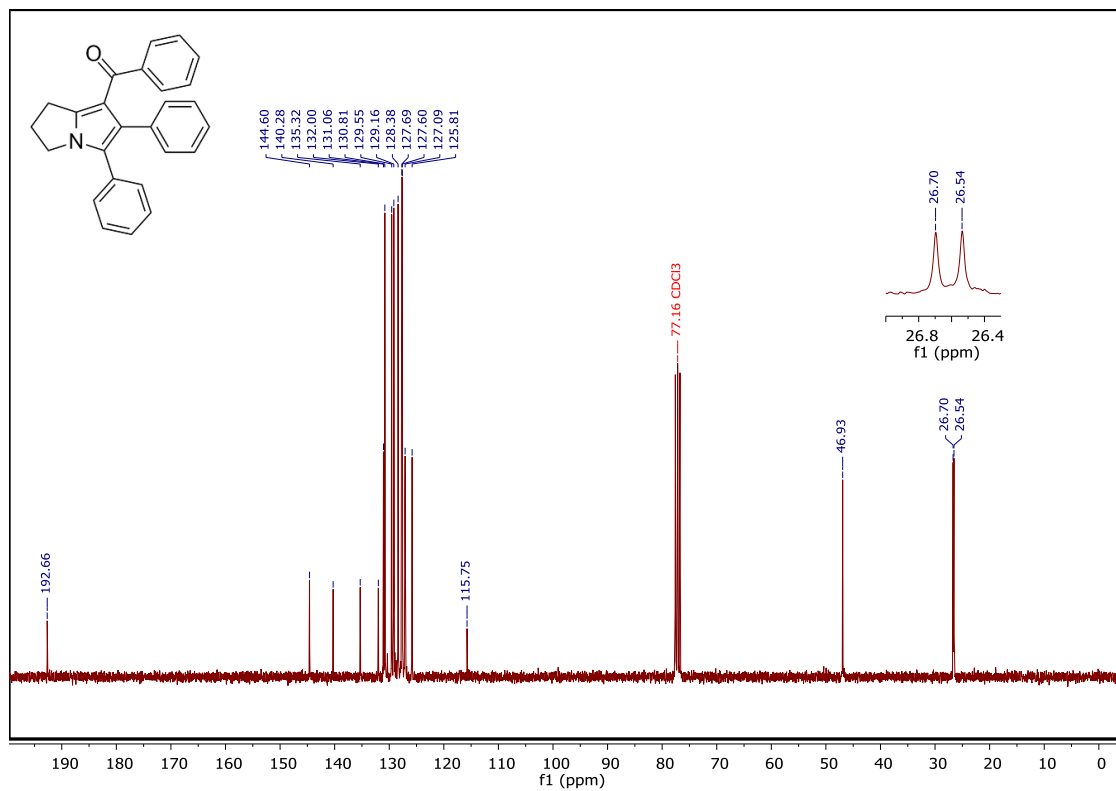
^{13}C NMR spectrum of [5-bromo-6-(3,4-dimethoxyphenyl)-2,3-dihydro-1H-pyrrolizin-7-yl](phenyl)methanone (33b)
(75 MHz, CDCl_3)



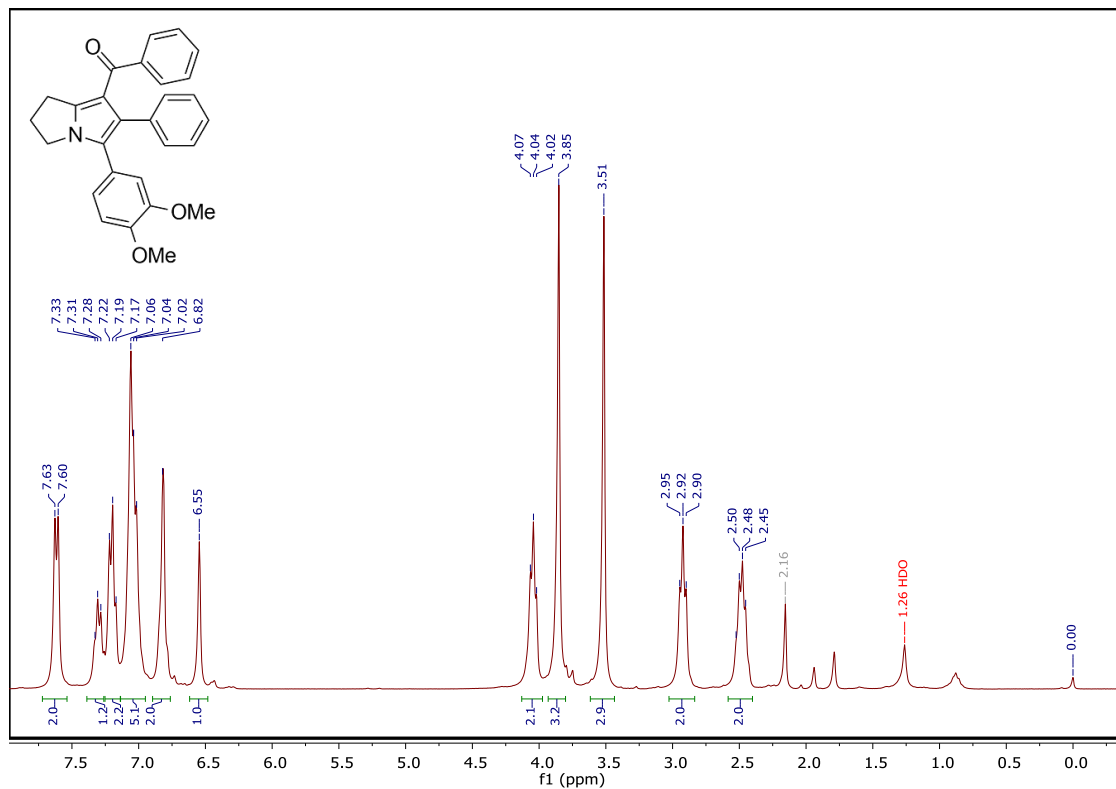
¹H NMR spectrum of (5,6-diphenyl-2,3-dihydro-1H-pyrrolizin-7-yl)(phenyl)methanone (34a)
(300 MHz, CDCl₃)



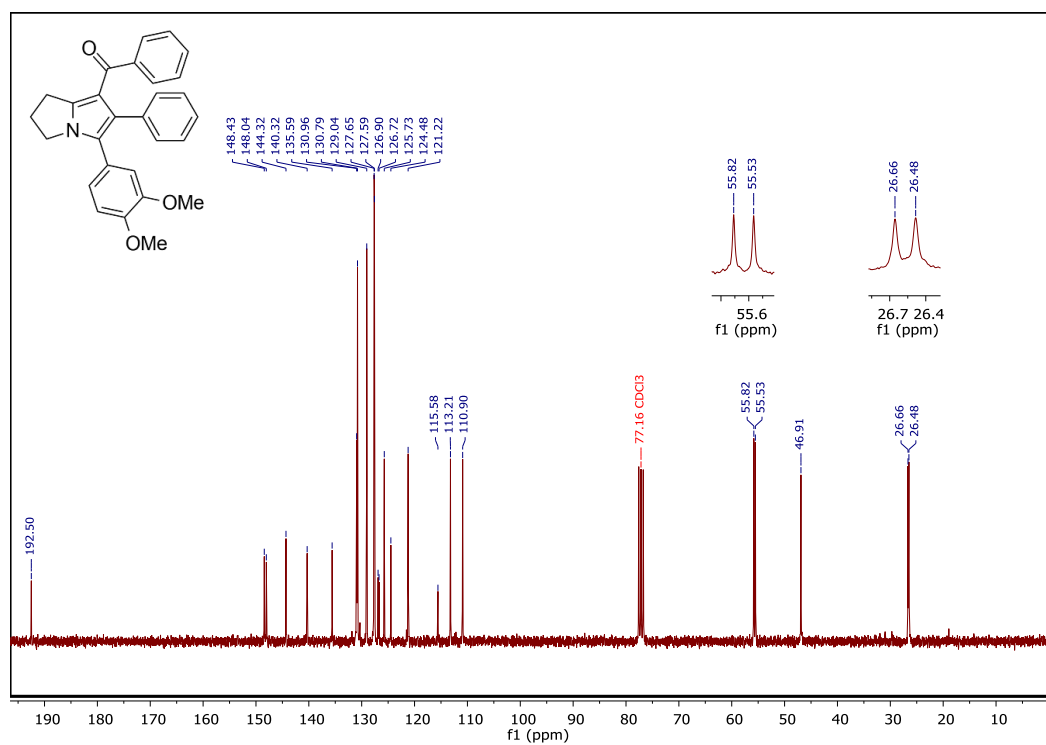
¹³C NMR spectrum of (5,6-diphenyl-2,3-dihydro-1H-pyrrolizin-7-yl)(phenyl)methanone (34a)
(75 MHz, CDCl₃)



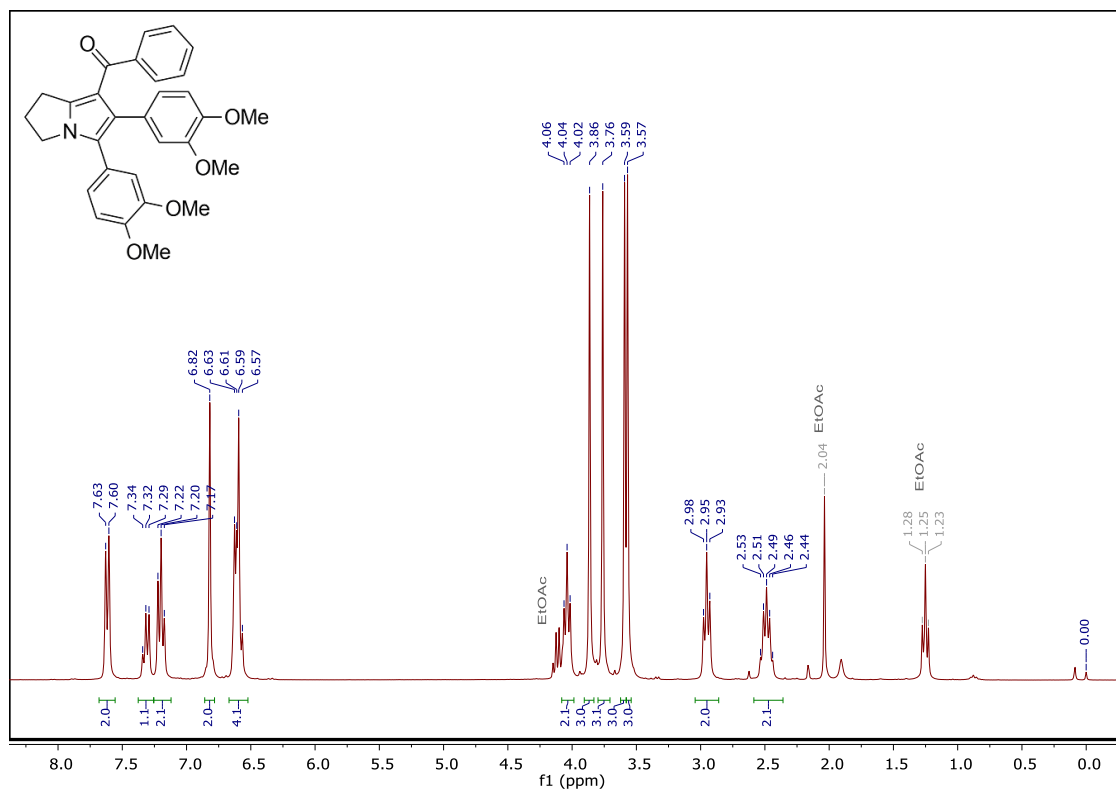
¹H NMR spectrum of [5-(3,4-dimethoxyphenyl)-6-phenyl-2,3-dihydro-1H-pyrrolizin-7-yl](phenyl)methanone (34b)
(300 MHz, CDCl₃)



¹³C NMR spectrum of [5-(3,4-dimethoxyphenyl)-6-phenyl-2,3-dihydro-1H-pyrrolizin-7-yl](phenyl)methanone (34b) (75 MHz, CDCl₃)



^1H NMR spectrum of [5,6-bis(3,4-dimethoxyphenyl)-2,3-dihydro-1H-pyrrolizin-7-yl](phenyl)methanone (34c)
(300 MHz, CDCl_3)



^{13}C NMR spectrum of [5,6-bis(3,4-dimethoxyphenyl)-2,3-dihydro-1H-pyrrolizin-7-yl](phenyl)methanone (34c) (75 MHz, CDCl_3)

