Supporting Information

Visible Light-mediated Photochemical Reactions of 2-(2'-Alkenyloxy)cycloalk-2-enones

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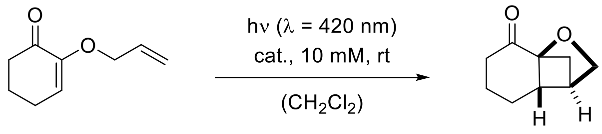
1. Sensitizers and Catalyst Loadings Tested in Initial Studies



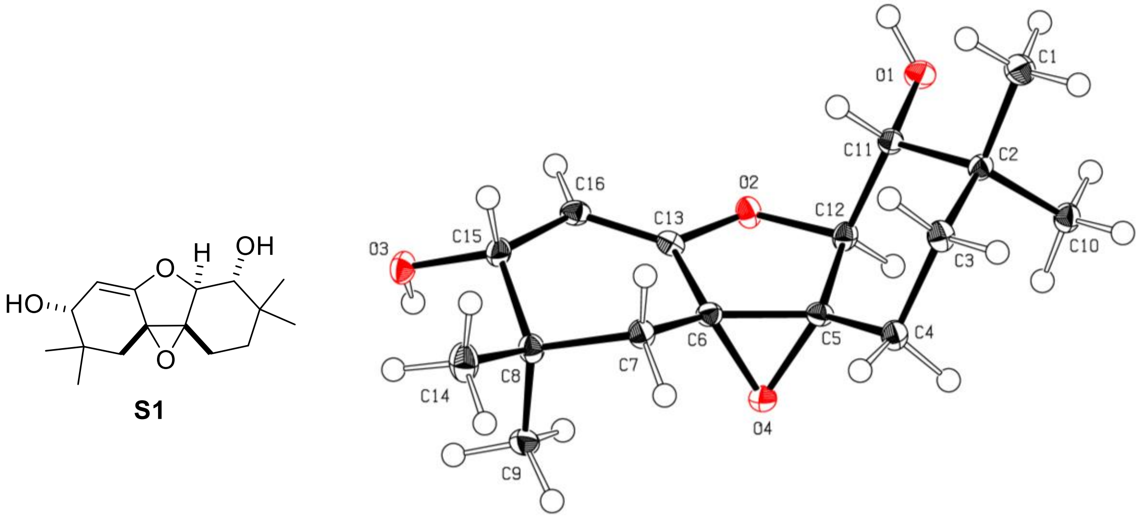
1a **8a**

#	Kat.	[mol%]	E_T [kJ/mol]	T [h]	Y [%]
1	-*	-	-	2.5	35
2	Benzophenone*	10	290[1]	2.0	61
3	Txt	10	268[2]	2.5	73
4	Txt	5.0	268	2.5	72
5	Txt	2.5	268	2.5	70
6	^{F}Irpic	2.5	256[3]	2.5	81
7	Ir(pFppy)$_3$	2.5	245[4]	3.0	69
8	Ir(ppy)$_3$	2.5	231[4]	5.0	85
9	Ru(bpy)$_3$(PF$_6$)$_2$	2.5	193[5]	27	(rsm)

* irradiation performed at 350 nm

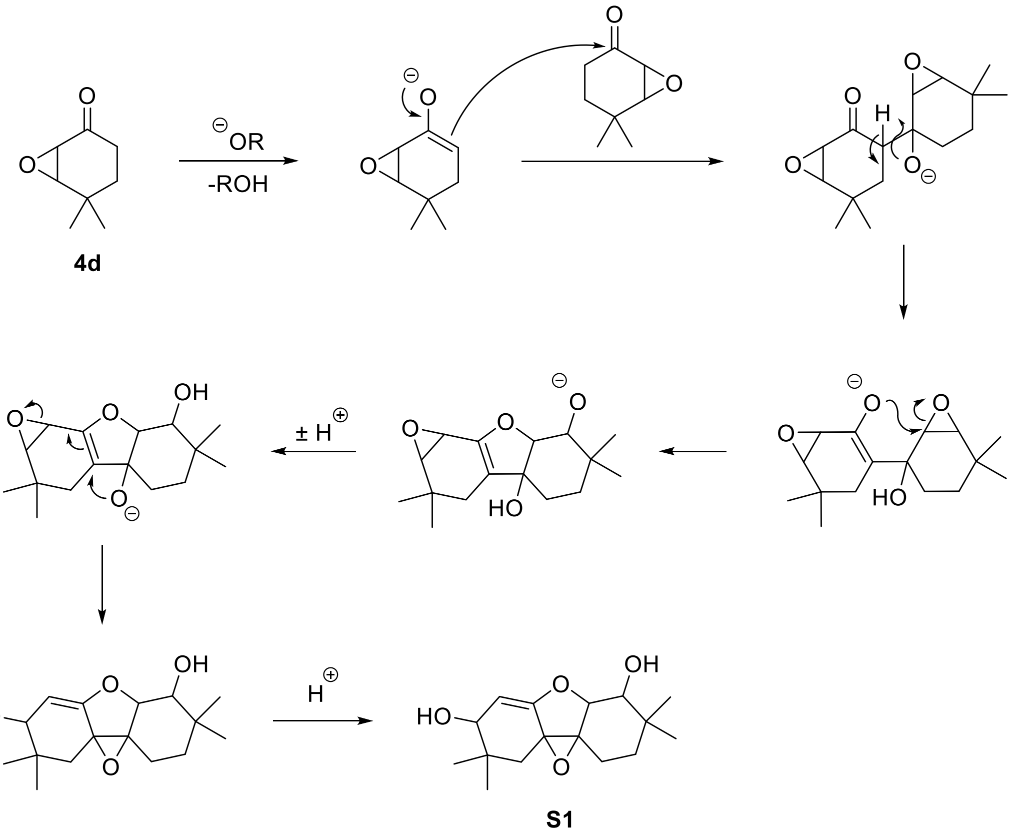
2. Side Product **S1** Formed from **4d**

In analogy to compound **2d**: KOH (85 wt%, 283 mg, 4.28 mmol, 1.0 equiv.) was dissolved in methallylic alcohol (1.5 mL, 1.29 mg, 17.9 mmol, 4.2 equiv.). A solution of epoxide **4d** (603 mg, 4.30 mmol, 1.0 equiv.) in methallylic alcohol (1.0 mL, 0.851 mg, 11.8 mmol, 2.8 equiv.) was then added dropwise and the reaction mixture was stirred at room temperature over night. Subsequently the reaction mixture was diluted with 15 mL CH_2Cl_2 and quenched by the addition of 15 mL H_2O. The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layers were washed with brine (1 × 150 mL), dried over Na_2SO_4, filtrated and the solvent as well as the remaining volatiles were removed under reduced pressure. The irradiation precursor **2d** was dissolved in a minimum of Et_2O and subjected to flash column chromatography. The remaining colorless solid was filtered off. It is insoluble in Et_2O and poorly soluble in CH_2Cl_2. Single crystals were obtained from a saturated solution of **S1** in CH_2Cl_2. The structure and relative configuration of **S1** were determined by data obtained from SC-XRD measurements.



S1

Figure S1. Structure of side product **S1** obtained by dimerization of epoxide **4d**. Ellipsoids in the ORTEP structure are displayed at the 50% probability level.



SCHEME S1. Mechanistic proposal for the formation of **S1**.

3. Phosphorescence Spectrum of **1a**

Luminescence Measurements were performed on a *Horiba Scientific* FluoroMax-4P instrument equipped with a continuous Xe source for steady state measurements and a Xe flashlight source for the observation of phosphorescence spectra. Fluorescence spectra were recorded at ambient conditions in 10×10 mm quartz cuvettes (*Hellma, Suprasil*). Luminescence spectra at cryogenic conditions (77 K) were recorded in quartz tubes (inner Ø = 4 mm) in a small quartz Dewar vessel which was filled with liquid nitrogen. All solutions were handled under dry nitrogen and degassed (15 min in an ultrasoundbath) to exclude oxygen as triplet quencher. A longpass filter (*Schott Advanced Optics*,WG300 or *Hebo Spezialglass*, UV280) was introduced in the emissionbeam path to prevent the excitation light to pass the emission monochromator at higher order wavelengths where necessary.

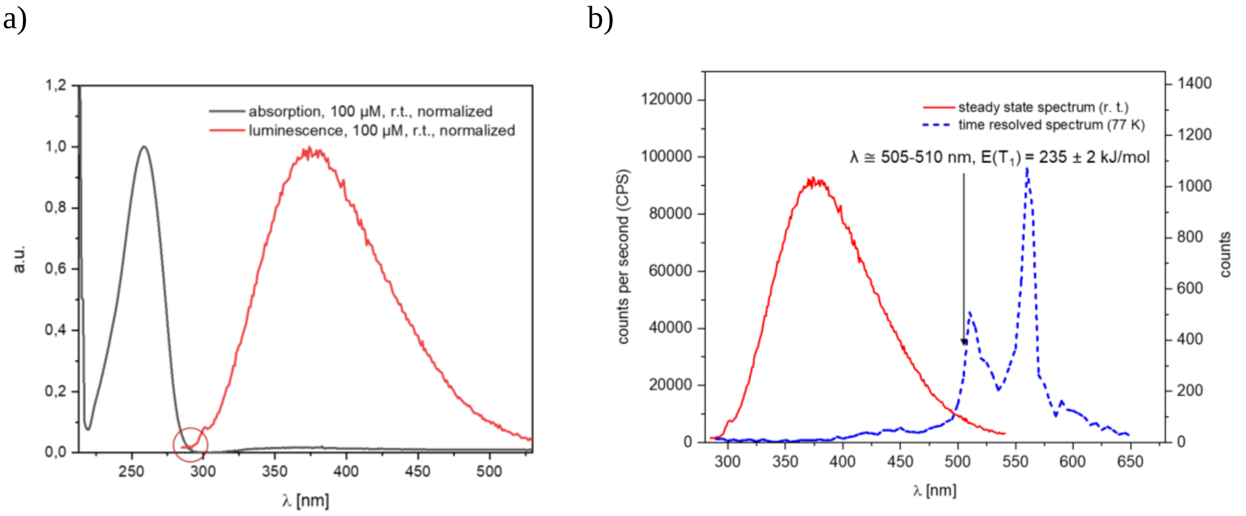
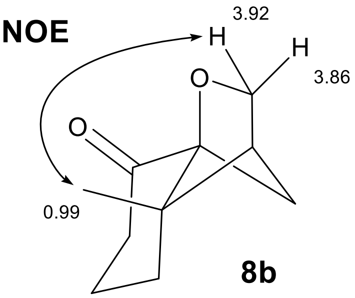
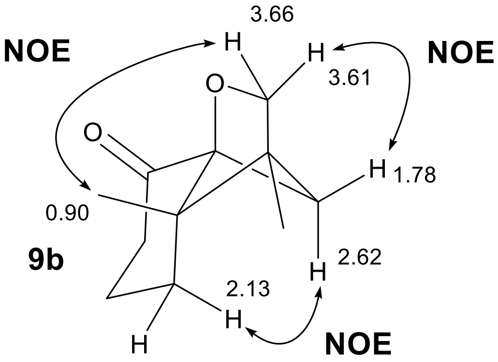


FIGURE S2. (a) Recorded UV/VIS of **1a** in CH_2Cl_2 (c = 100 µM), normalized to $A_{259\ nm}$; recorded luminescence (UV280 filter glass applied) of **1a** in CH_2Cl_2 (c = 100 µM) at ambient conditions, normalized to $I_{374\ nm}$. (b) Steady state spectrum of **1a** in CH_2Cl_2 (c = 100 µM) at ambient conditions (UV280 filter glass applied) given in counts per second (red, solid line); time resolved spectrum (WG300 filter glass applied) of **1a** in CH_2Cl_2 (c = 100 µM) after 50 µs delay at 77 K in counts (blue, dashed line).

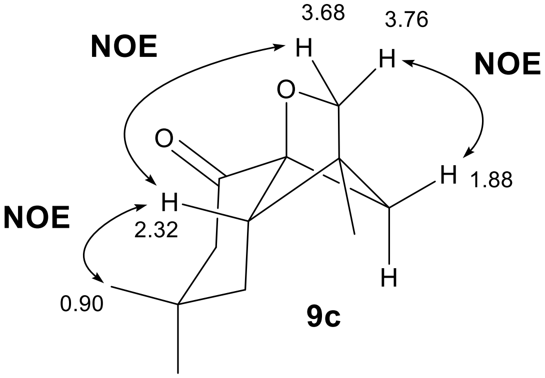
4. Important NOE Contacts Obtained from NOESY Spectra of Compounds **8b**, **9b-e**, **11**, **13** and **16a-c**



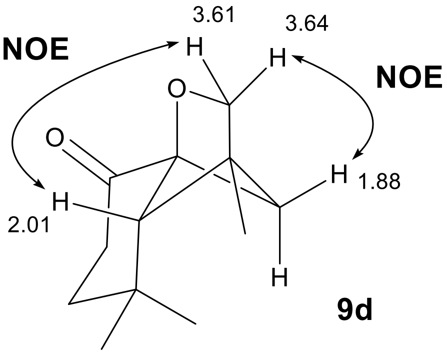
8b



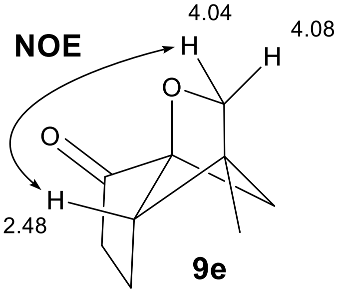
9b



9c



9d



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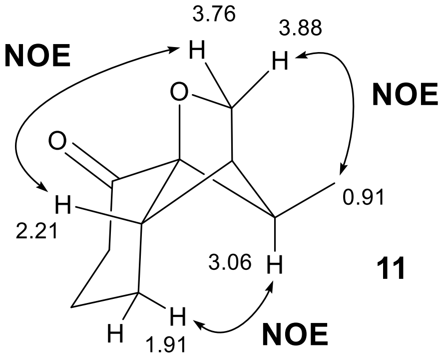

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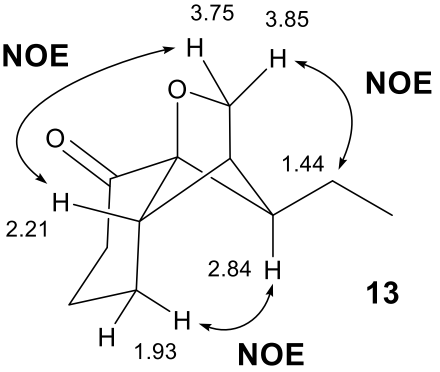

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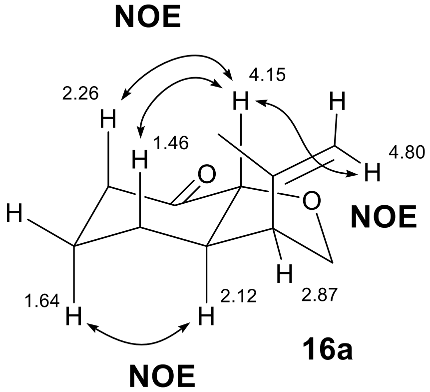
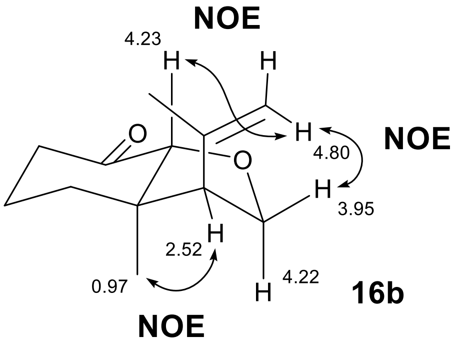
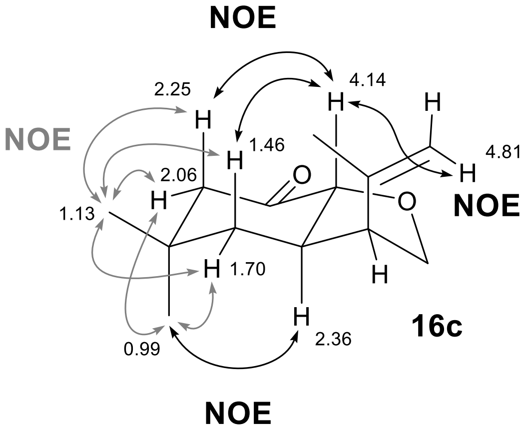


16a

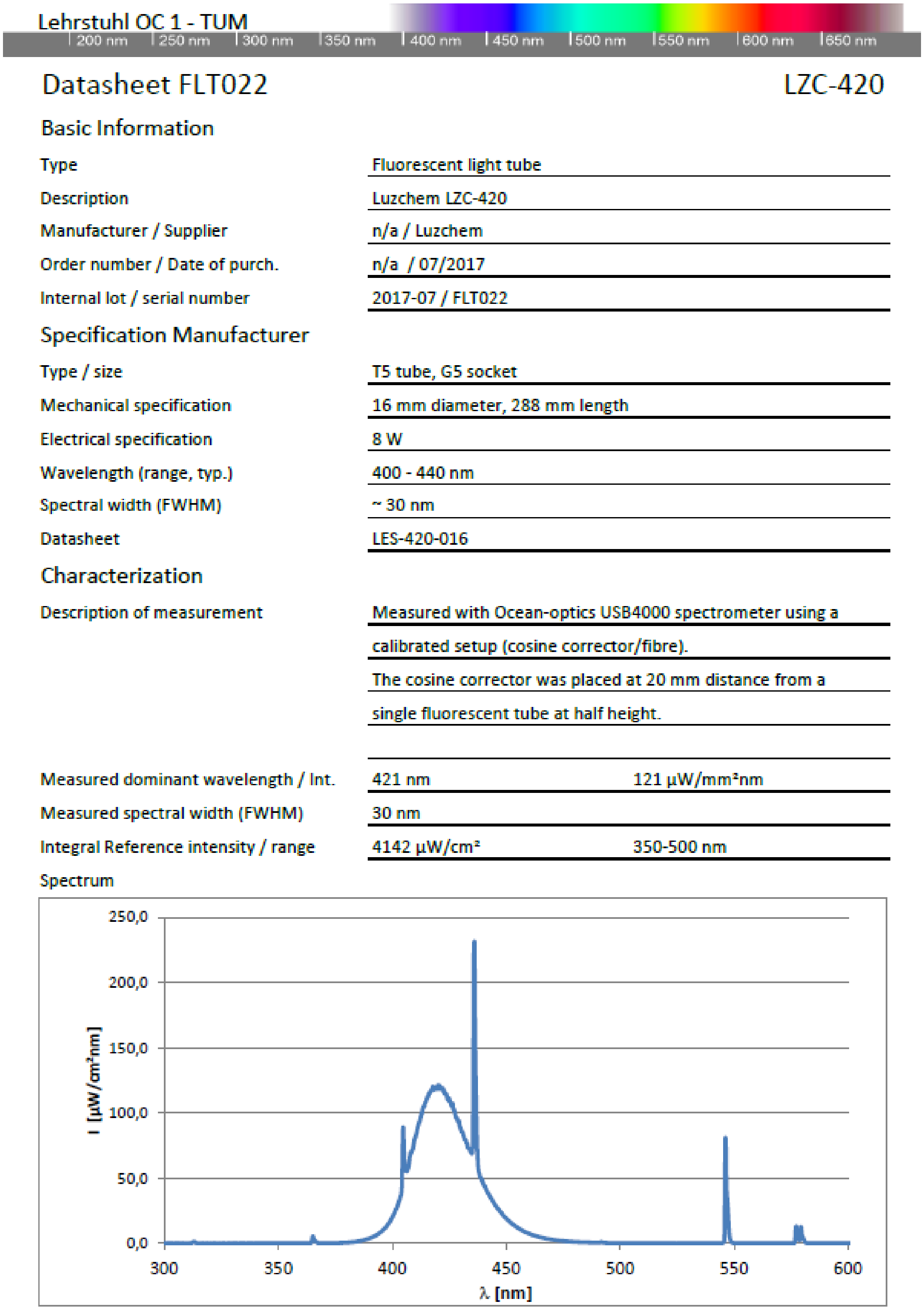


16b



16c

5. 420 nm Fluorescent Light Tube Emission Spectrum



Lehrstuhl OC 1 - TUM

200 nm 250 nm 300 nm 350 nm 400 nm 450 nm 500 nm 550 nm 600 nm 650 nm

Datasheet FLT022 LZC-420

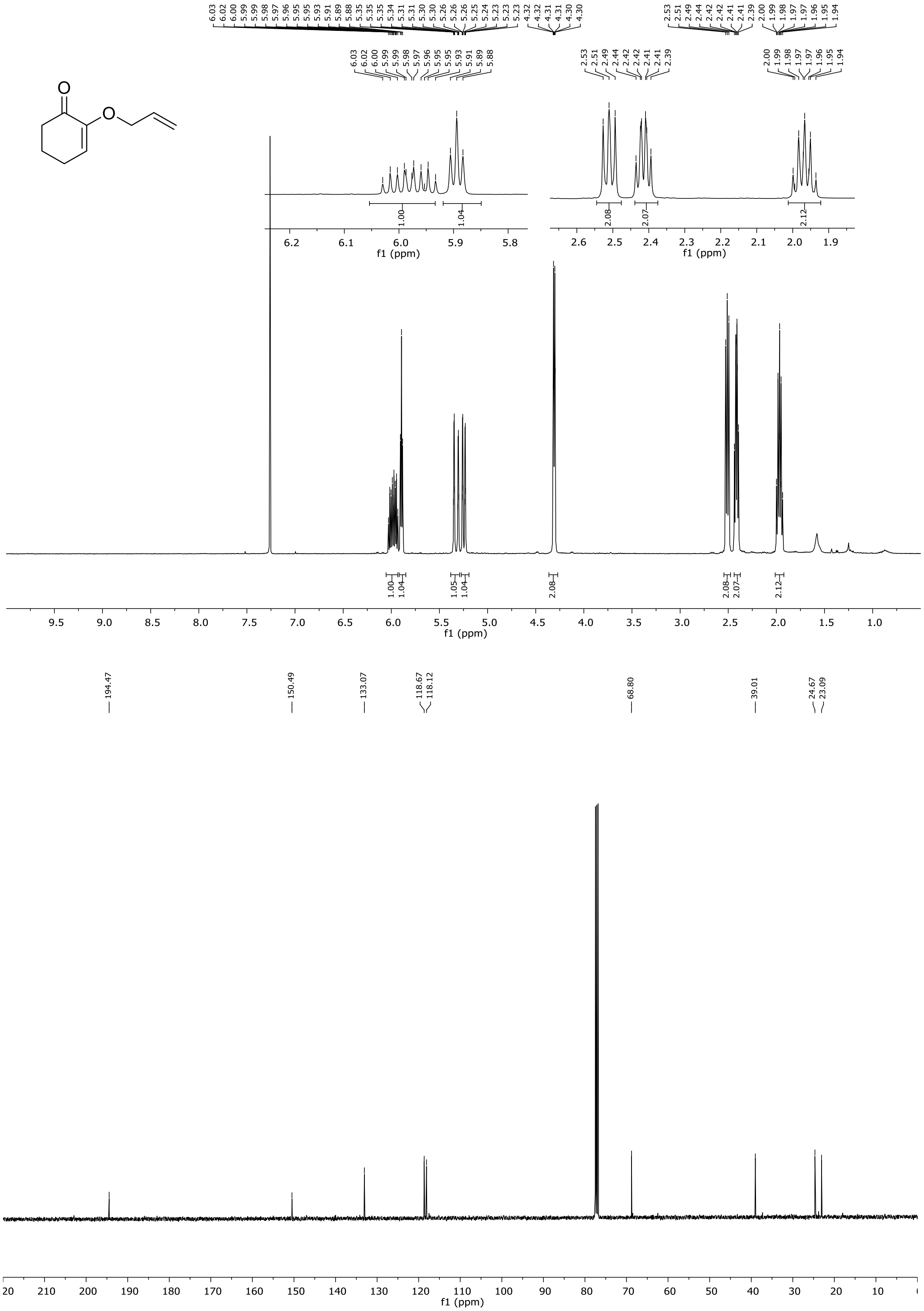
Basic Information

Type	Fluorescent light tube	
Description	Luzchem LZC-420	
Manufacturer / Supplier	n/a / Luzchem	
Order number / Date of purch.	n/a / 07/2017	
Internal lot / serial number	2017-07 / FLT022	
Specification Manufacturer		
Type / size	T5 tube, G5 socket	
Mechanical specification	16 mm diameter, 288 mm length	
Electrical specification	8 W	
Wavelength (range, typ.)	400 - 440 nm	
Spectral width (FWHM)	~ 30 nm	
Datasheet	LES-420-016	
Characterization		
Description of measurement	Measured with Ocean-optics USB4000 spectrometer using a calibrated setup (cosine corrector/fibre). The cosine corrector was placed at 20 mm distance from a single fluorescent tube at half height.	
Measured dominant wavelength / Int.	421 nm	121 µW/mm²nm
Measured spectral width (FWHM)	30 nm	
Integral Reference intensity / range	4142 µW/cm²	350-500 nm

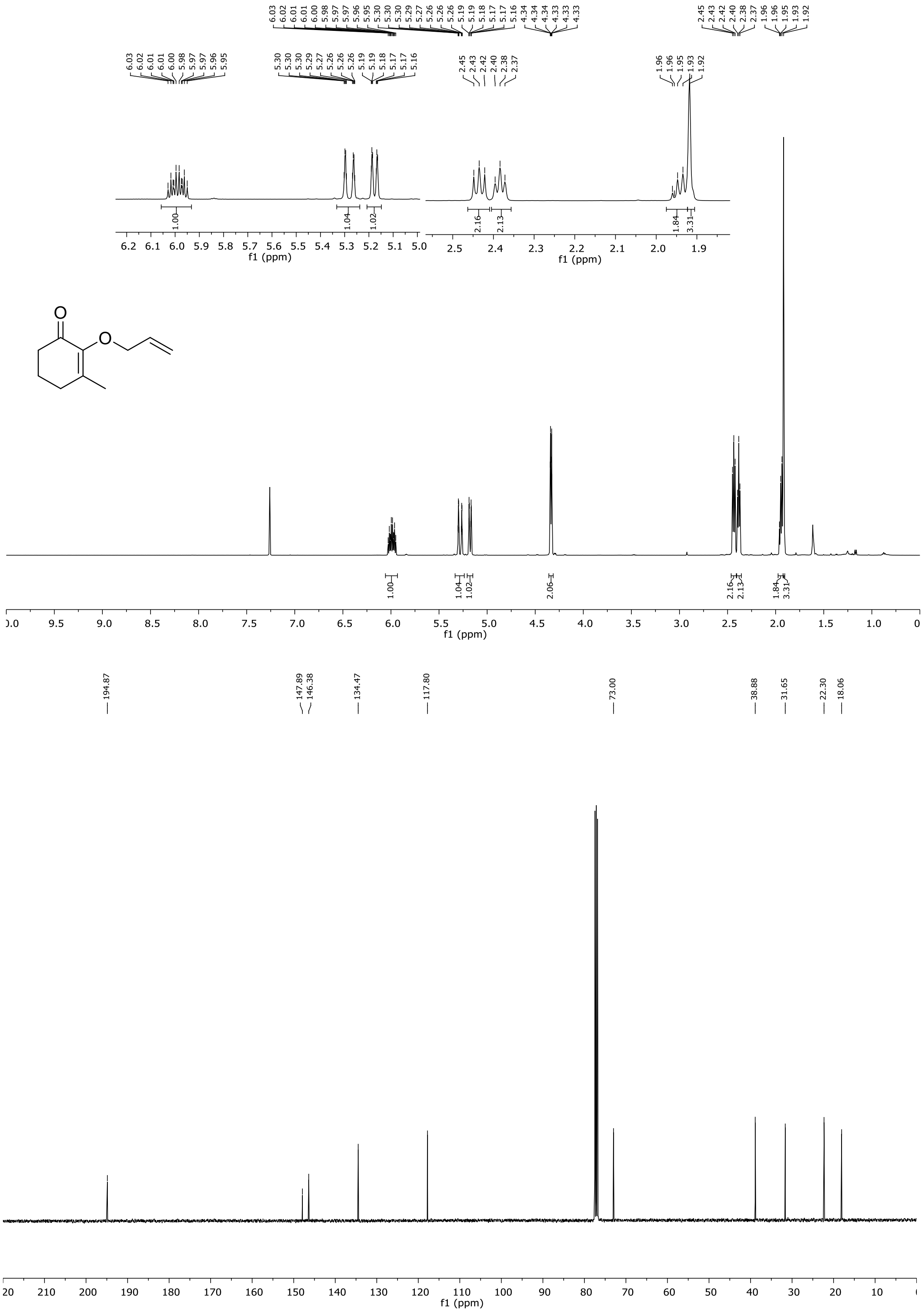
Spectrum

6. NMR Spectra of All Compounds

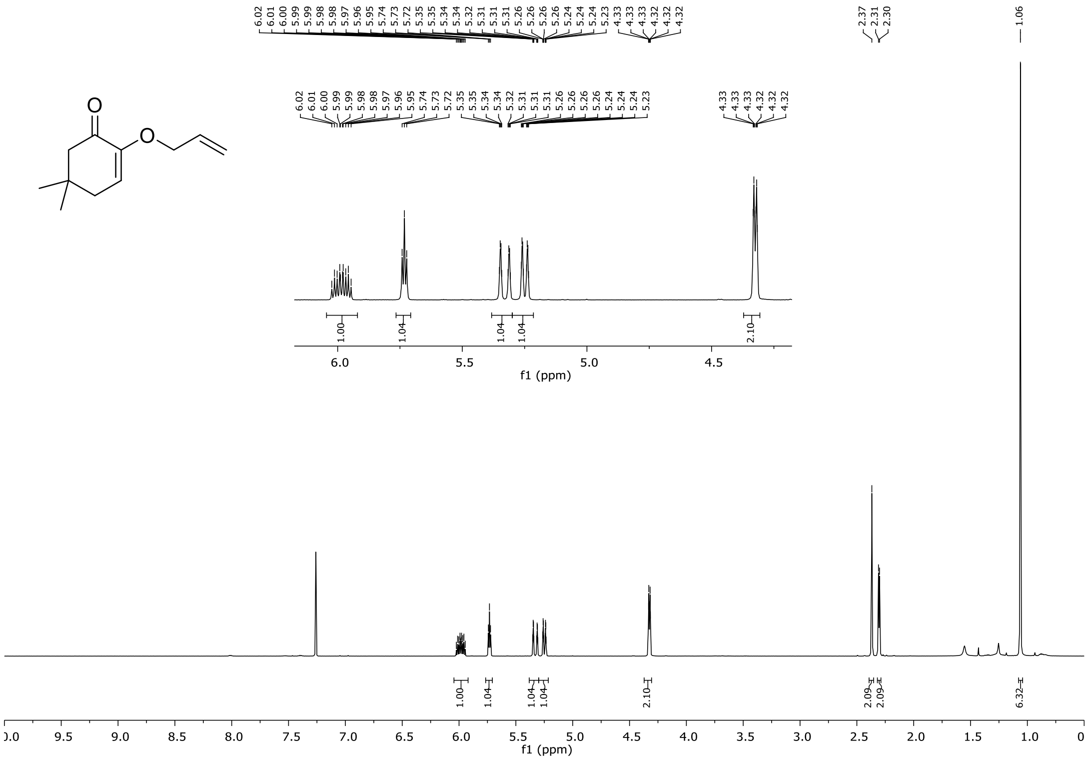
^{1}H (400 MHz) and $^{13}C\{^{1}H\}$ NMR (101 MHz) spectra of compound **1a** ($CDCl_3$)

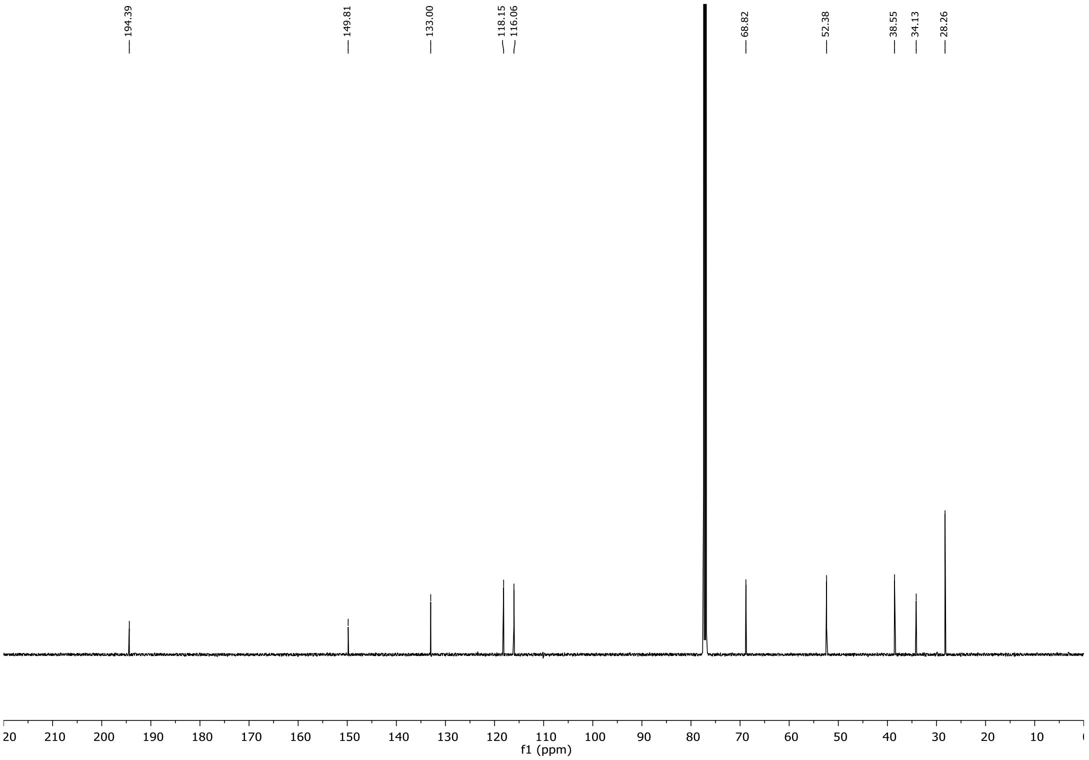


^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ NMR (101 MHz) spectra of compound **1b** ($CDCl_3$)

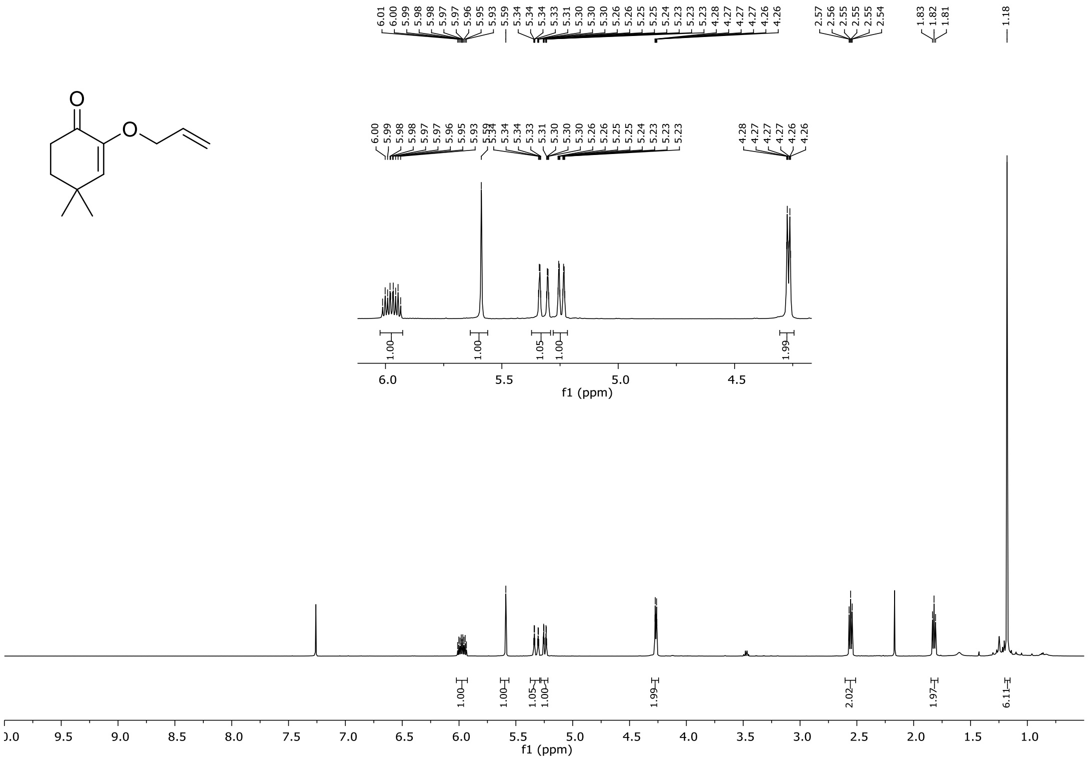


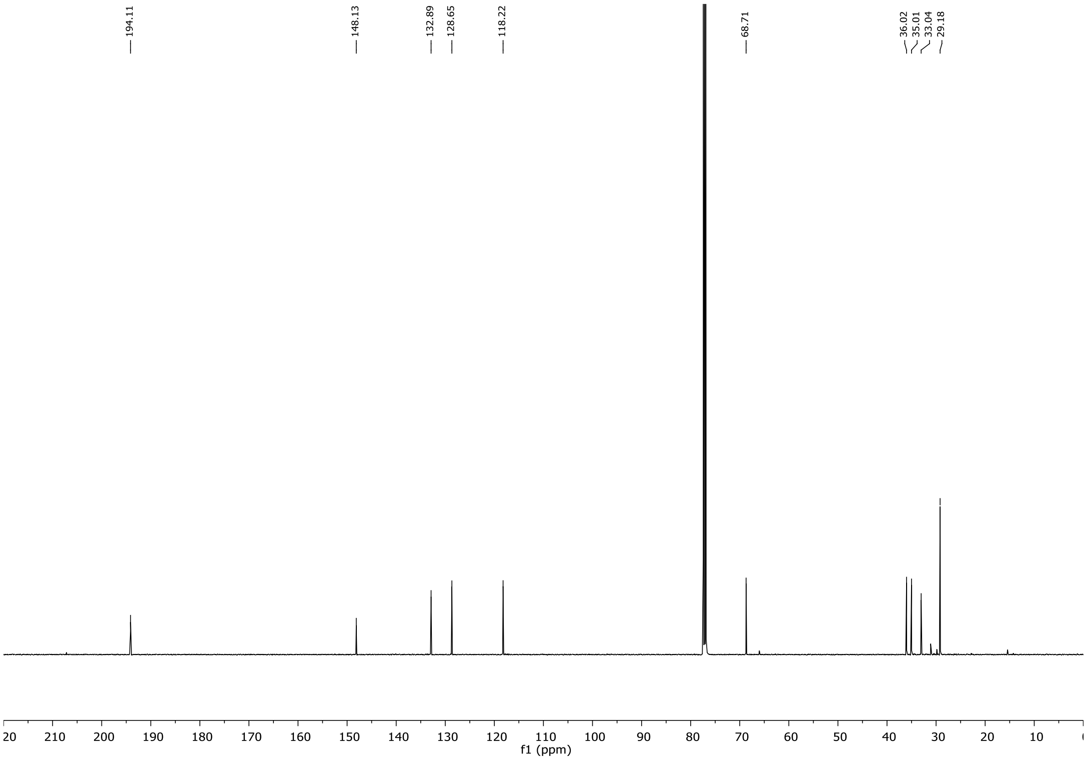
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **1c** (CDCl$_3$)



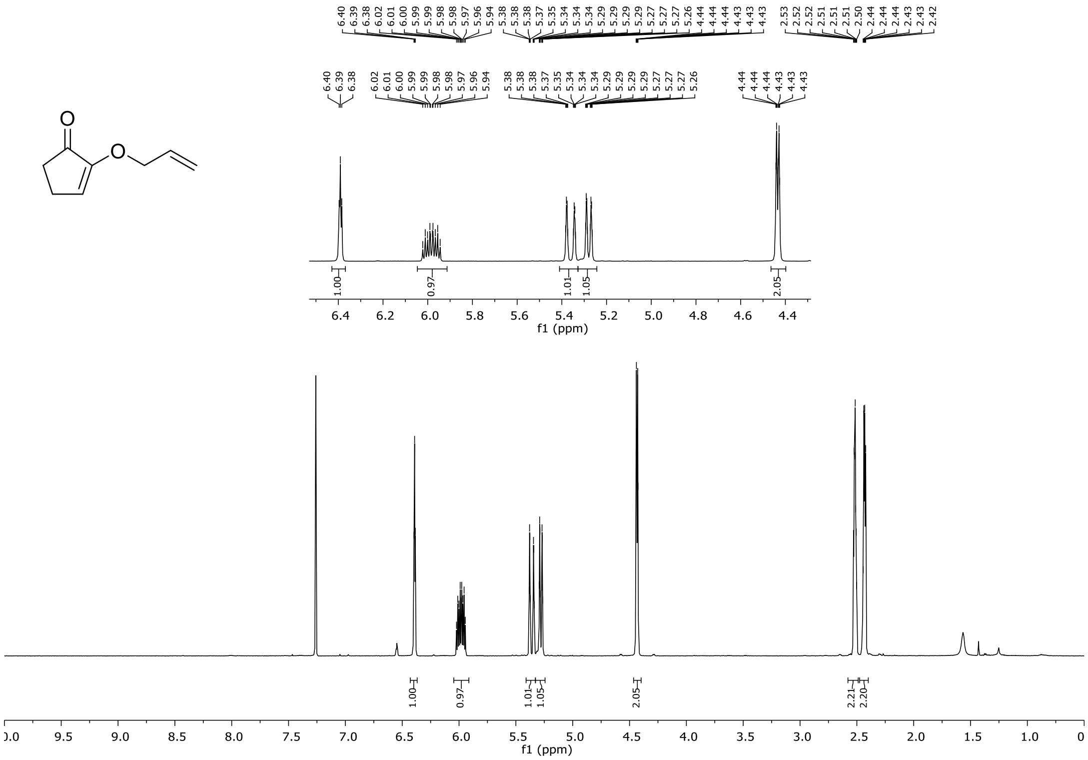


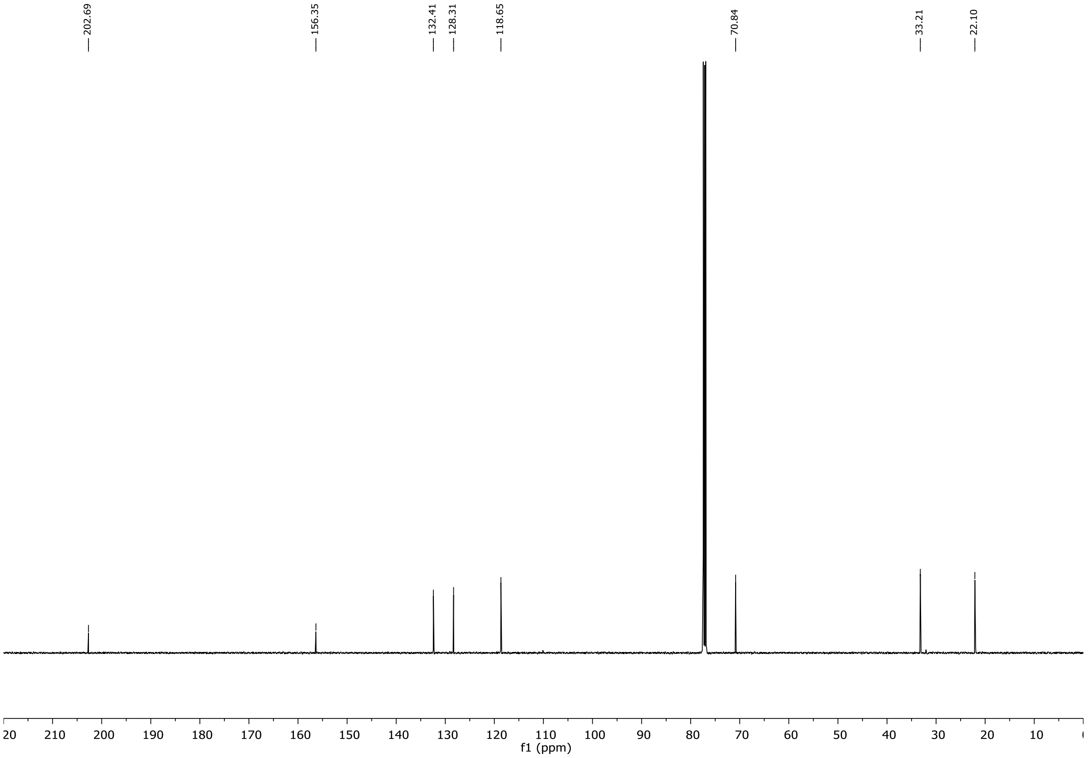
^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ NMR (126 MHz) spectra of compound **1d** ($CDCl_3$)



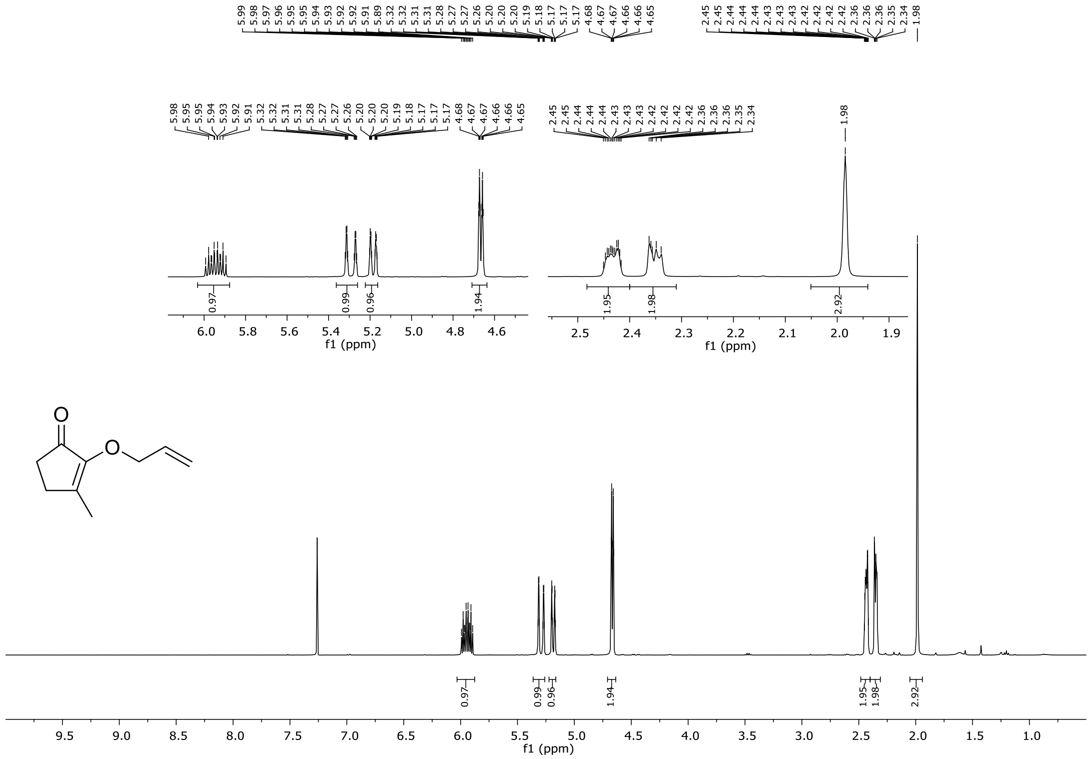


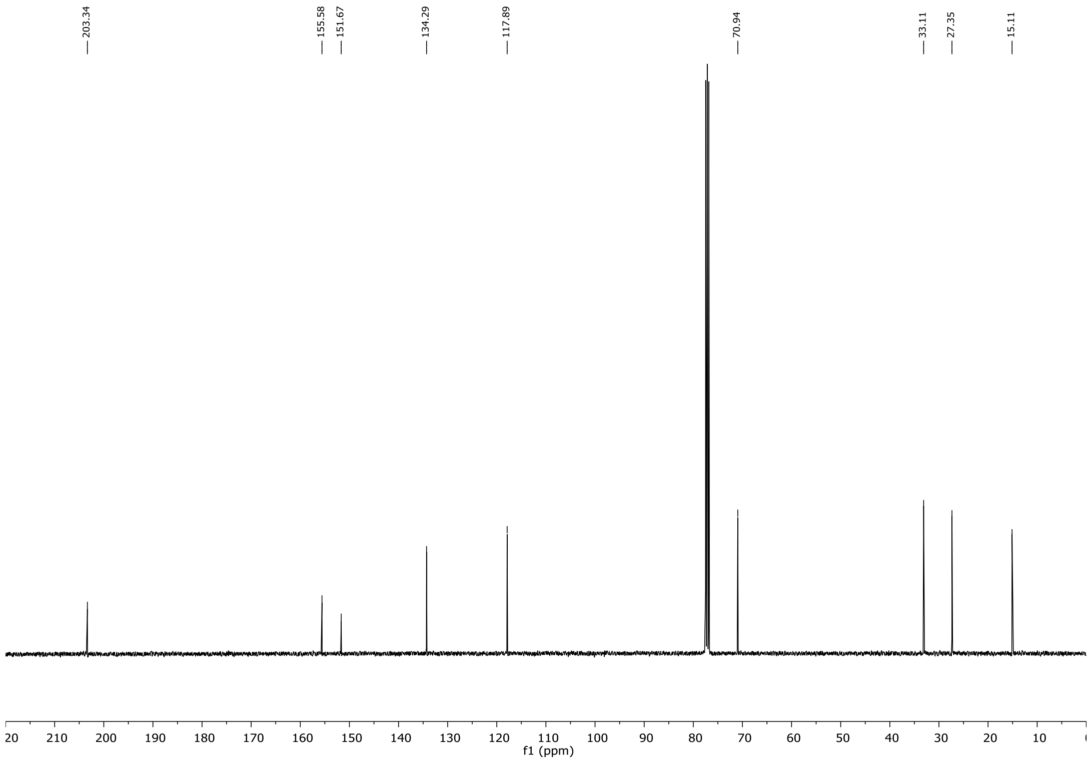
^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ NMR (126 MHz) spectra of compound **1e** ($CDCl_3$)



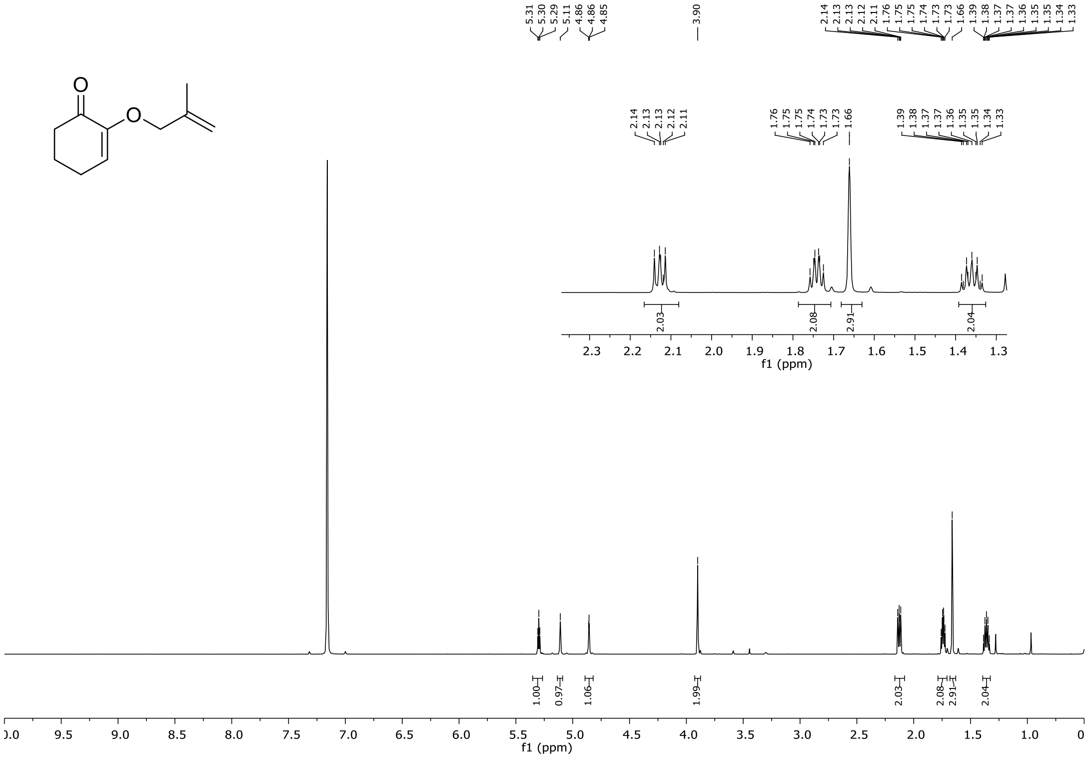


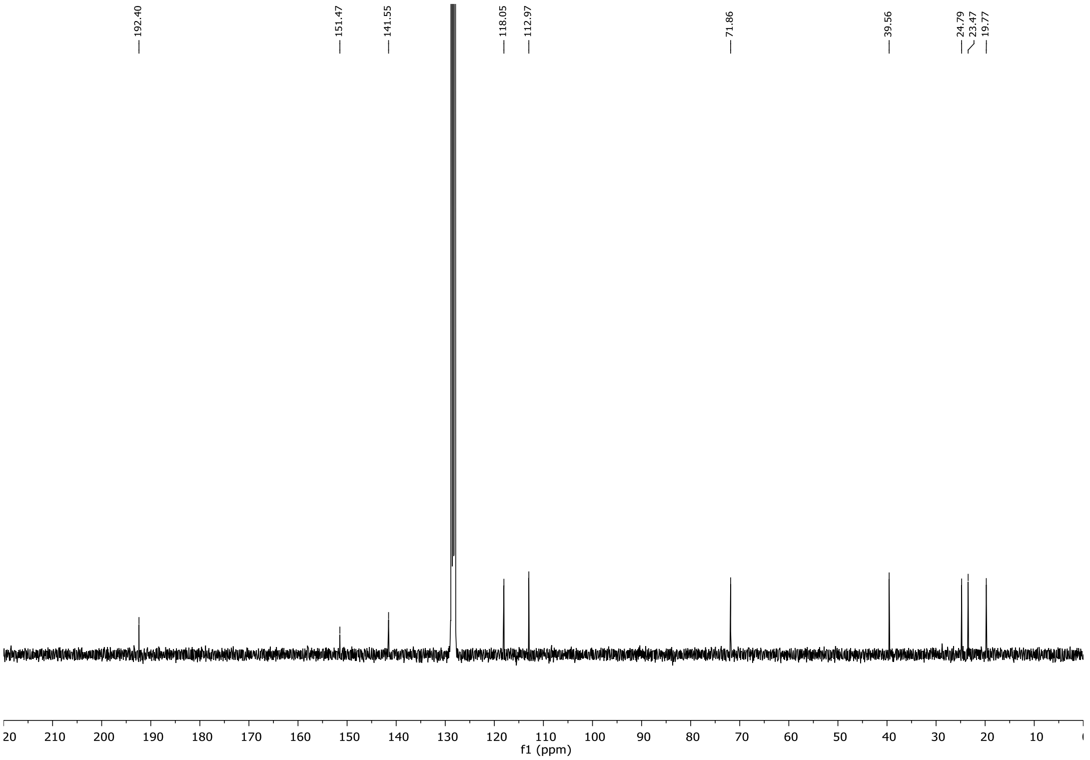
^{1}H (400 MHz) and ^{13}C{^{1}H} NMR (101 MHz) spectra of compound **1f** (CDCl$_3$)



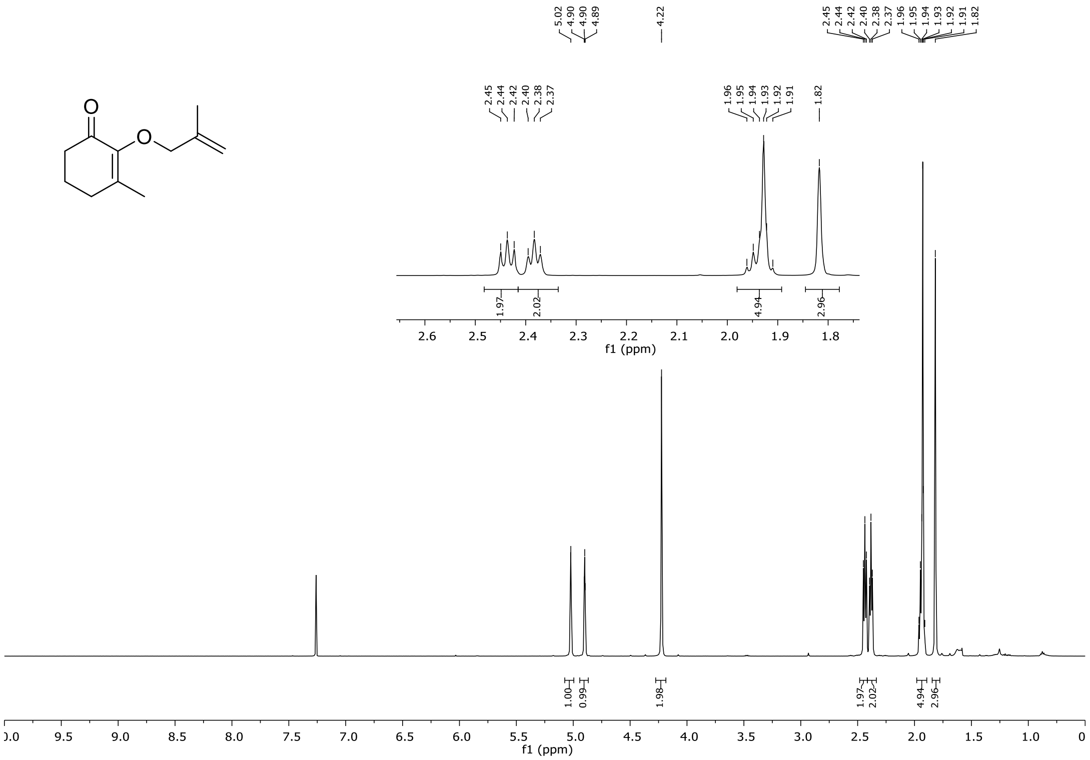


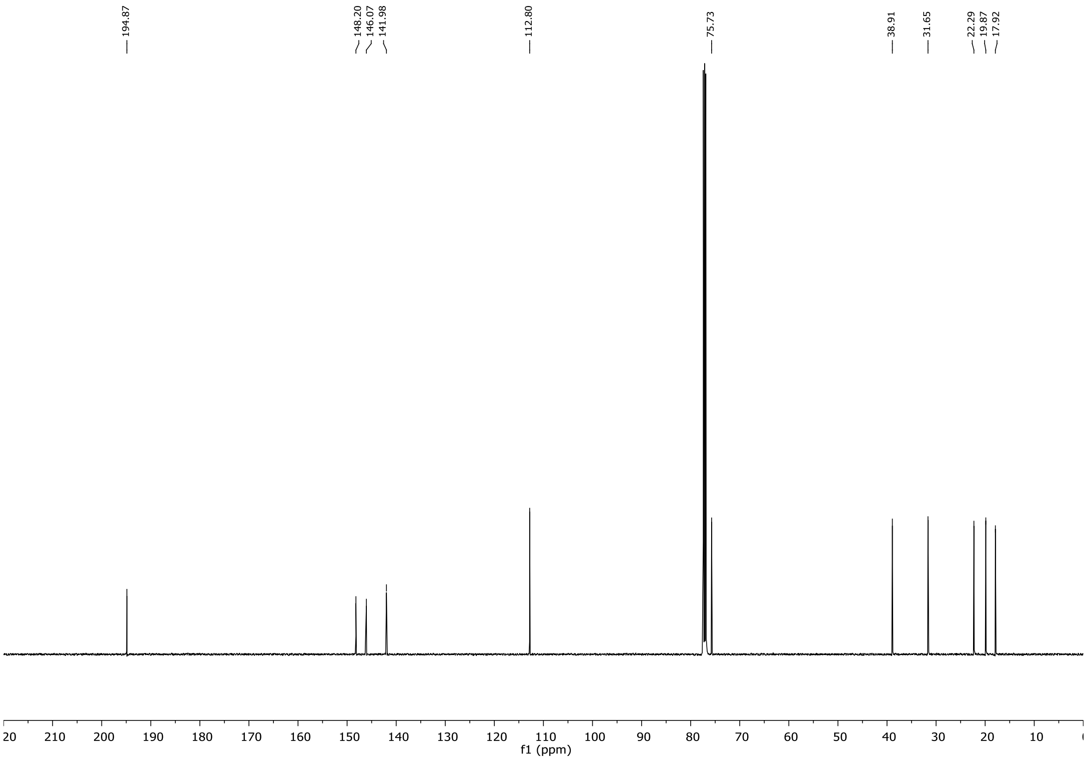
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (75 MHz) spectra of compound **2a** (C_6D_6)



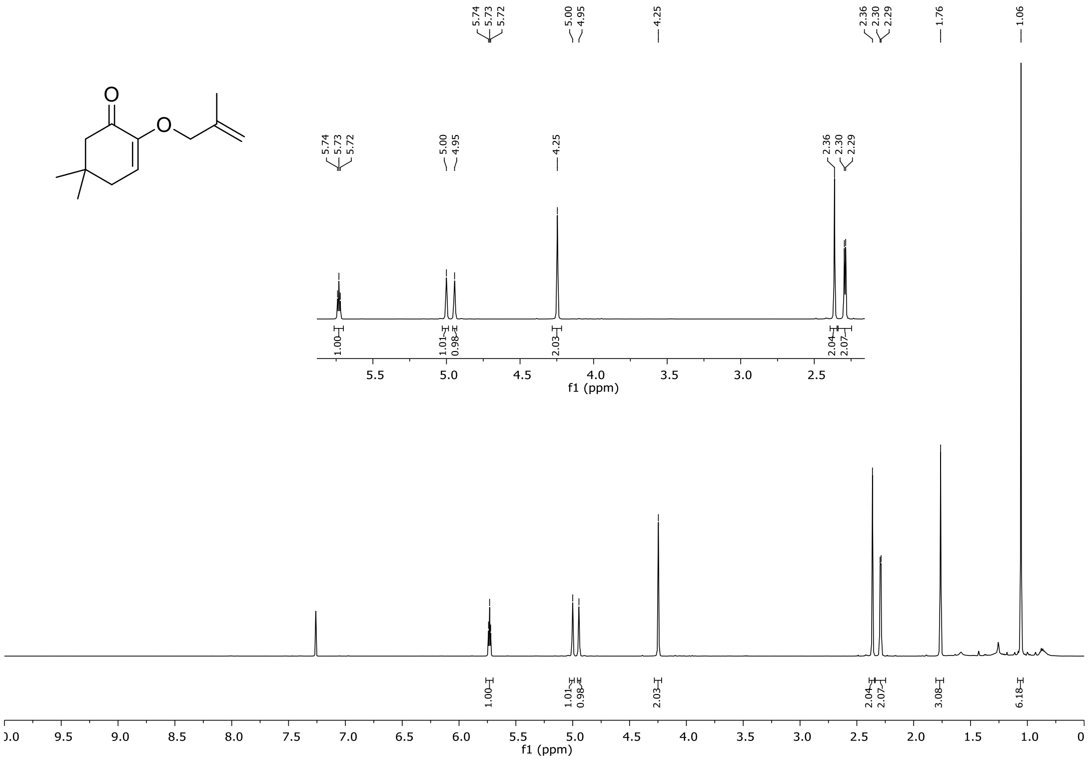


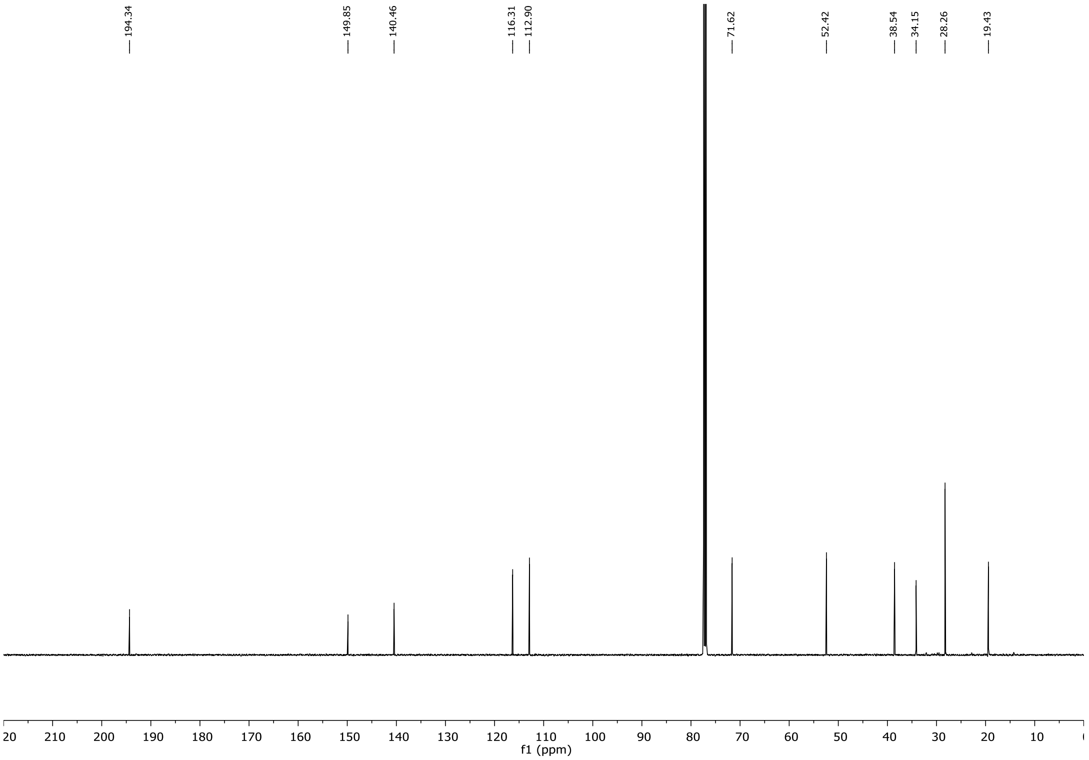
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **2b** (CDCl$_3$)





^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **2c** ($CDCl_3$)





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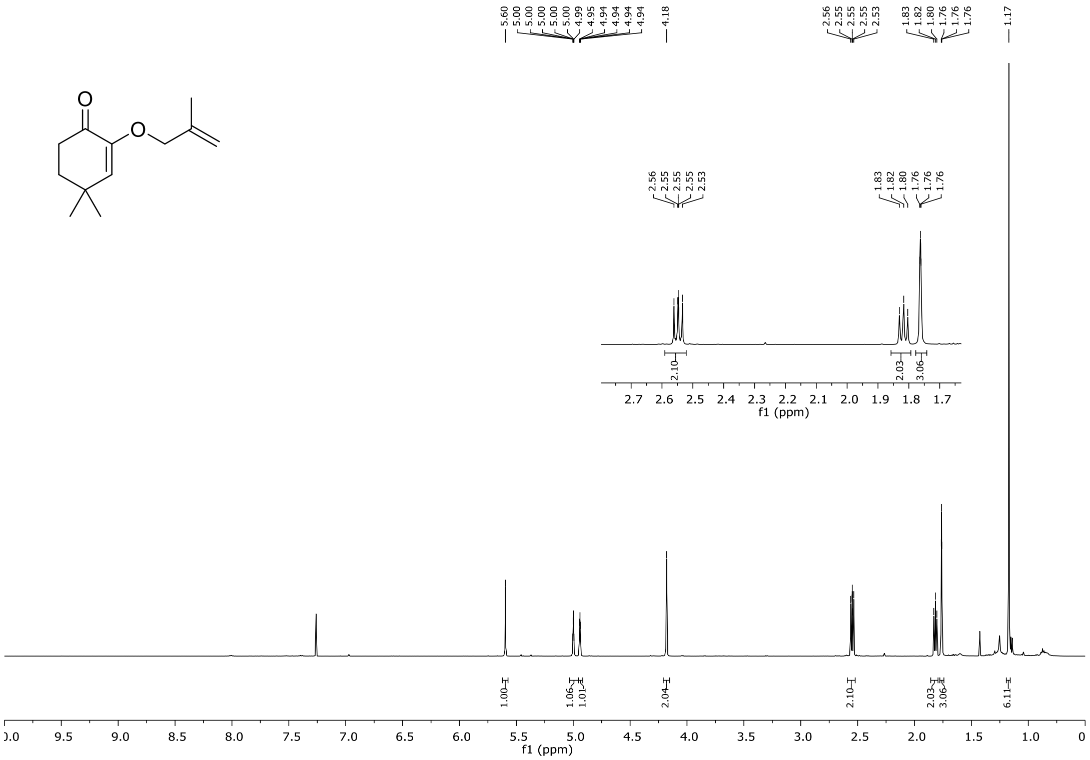

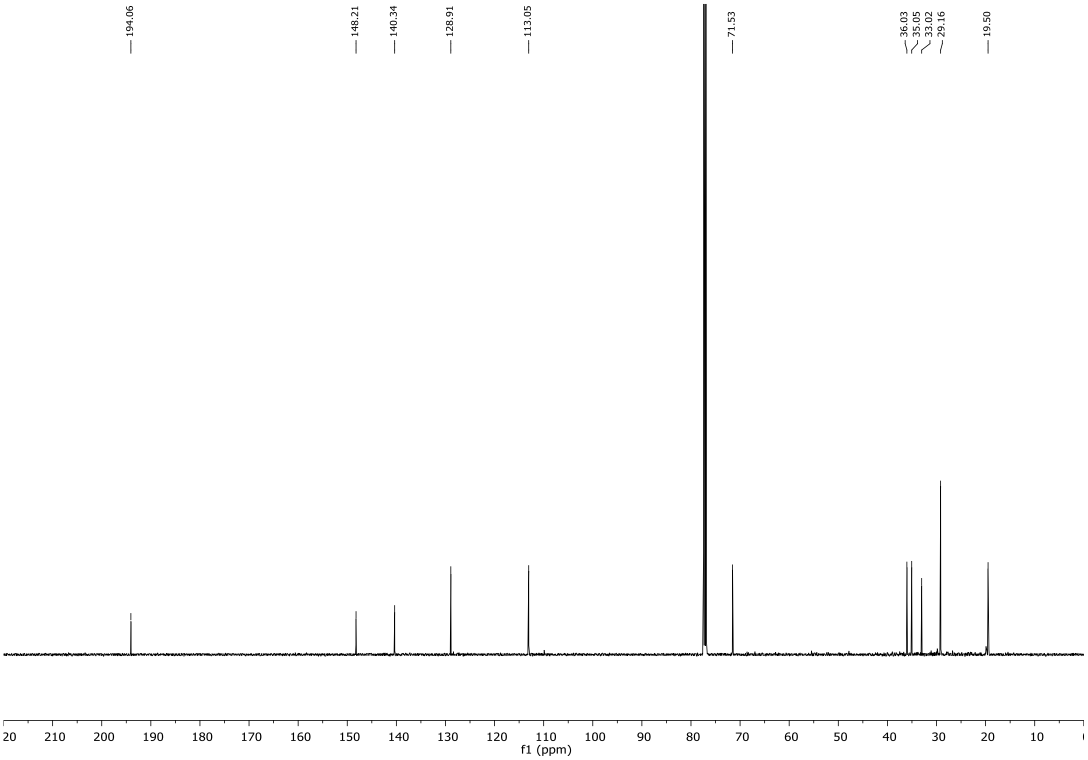

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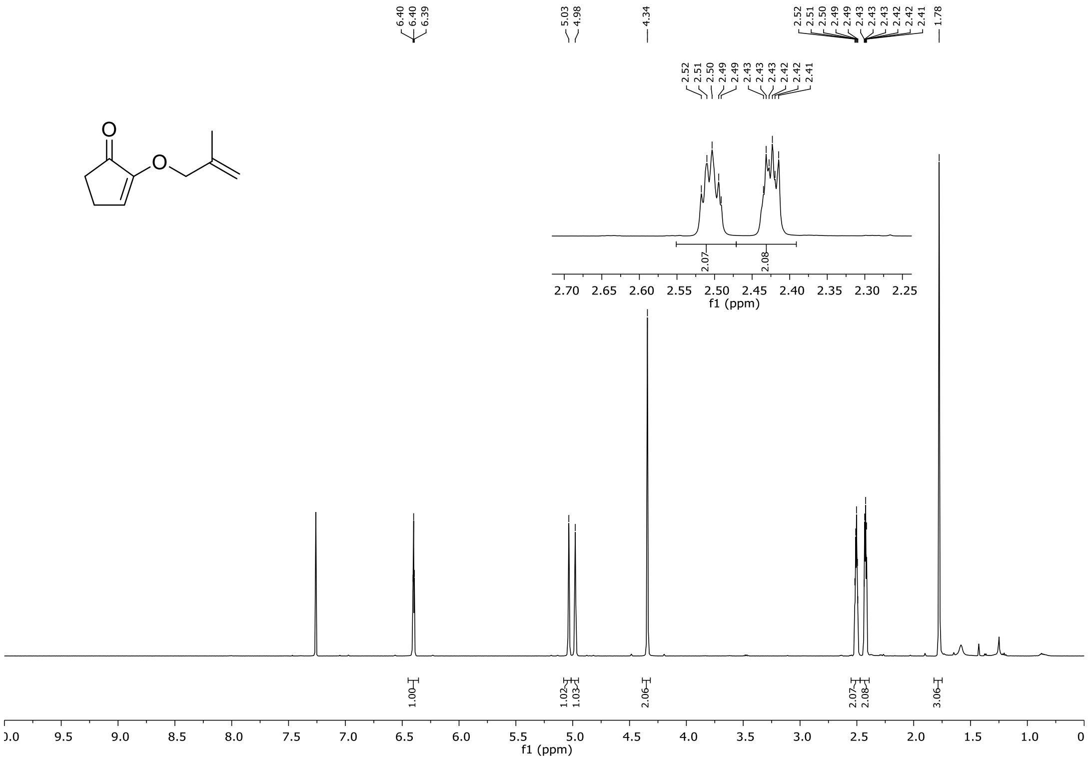
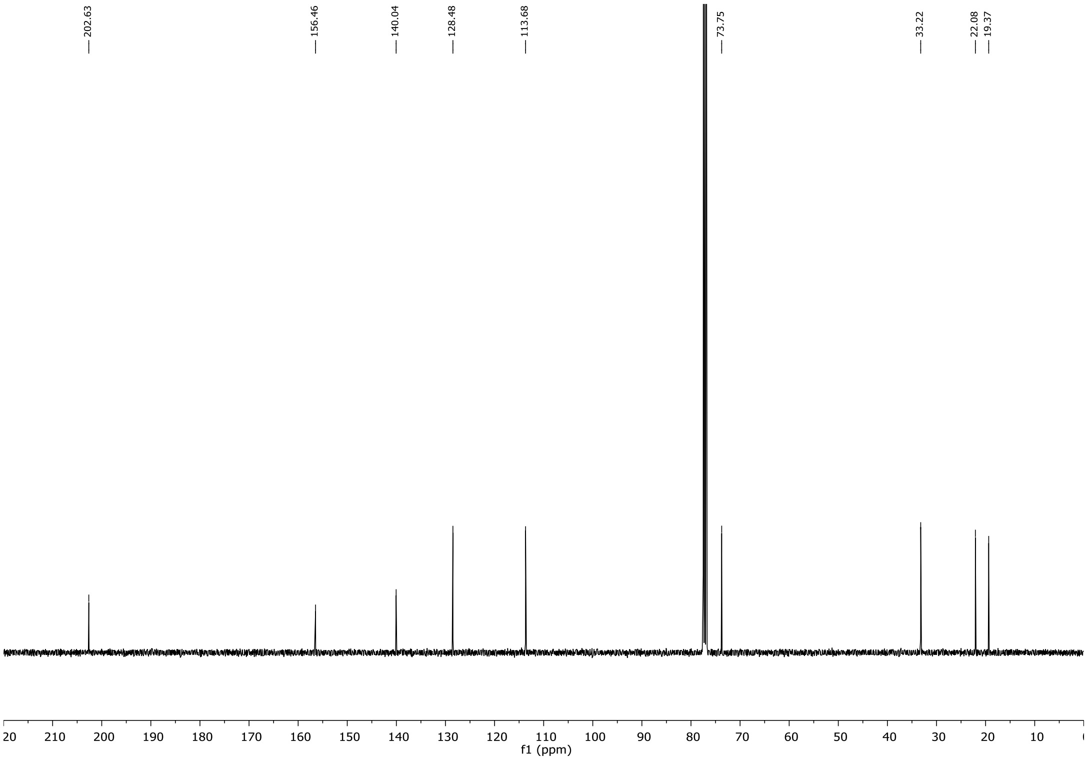
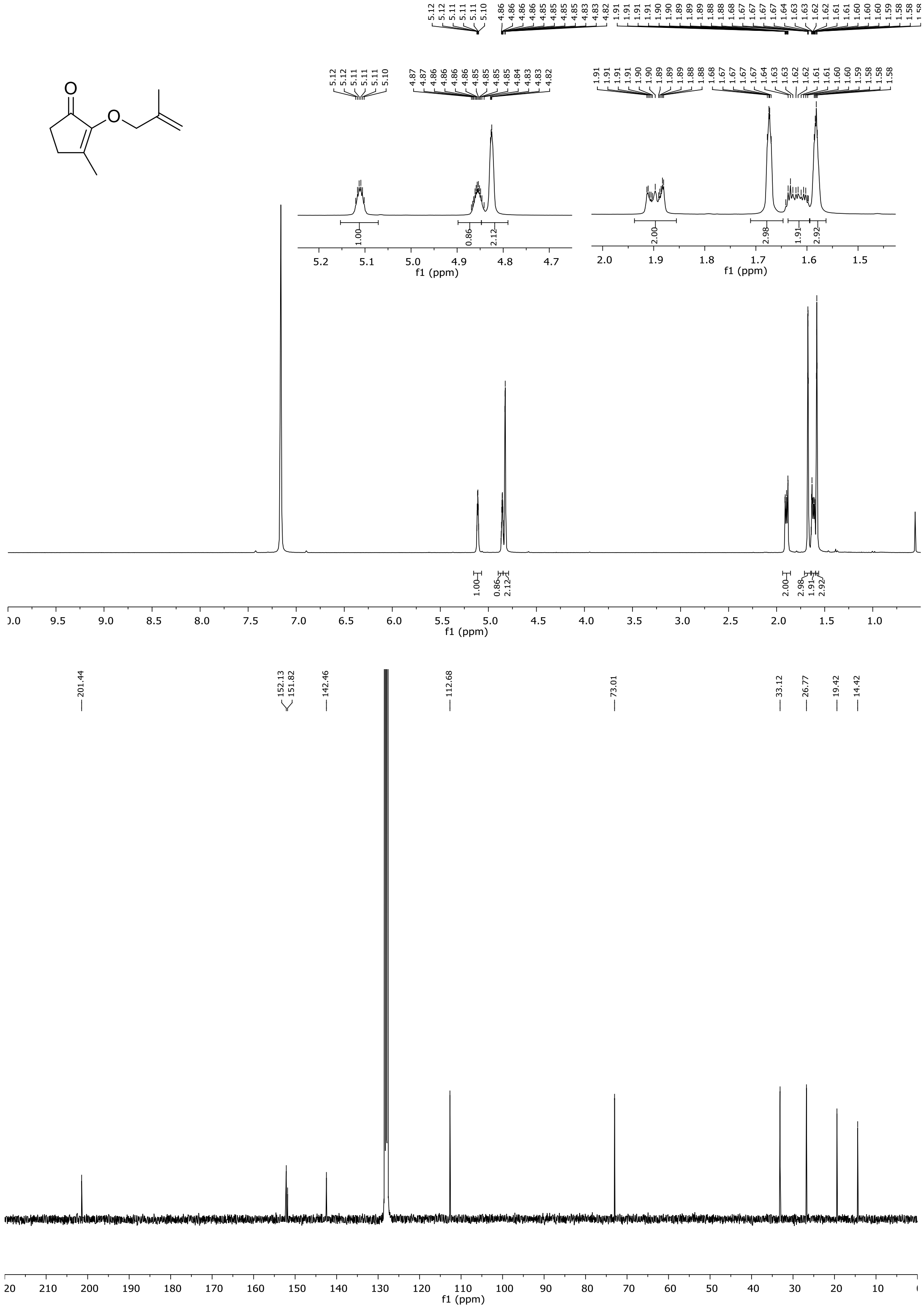
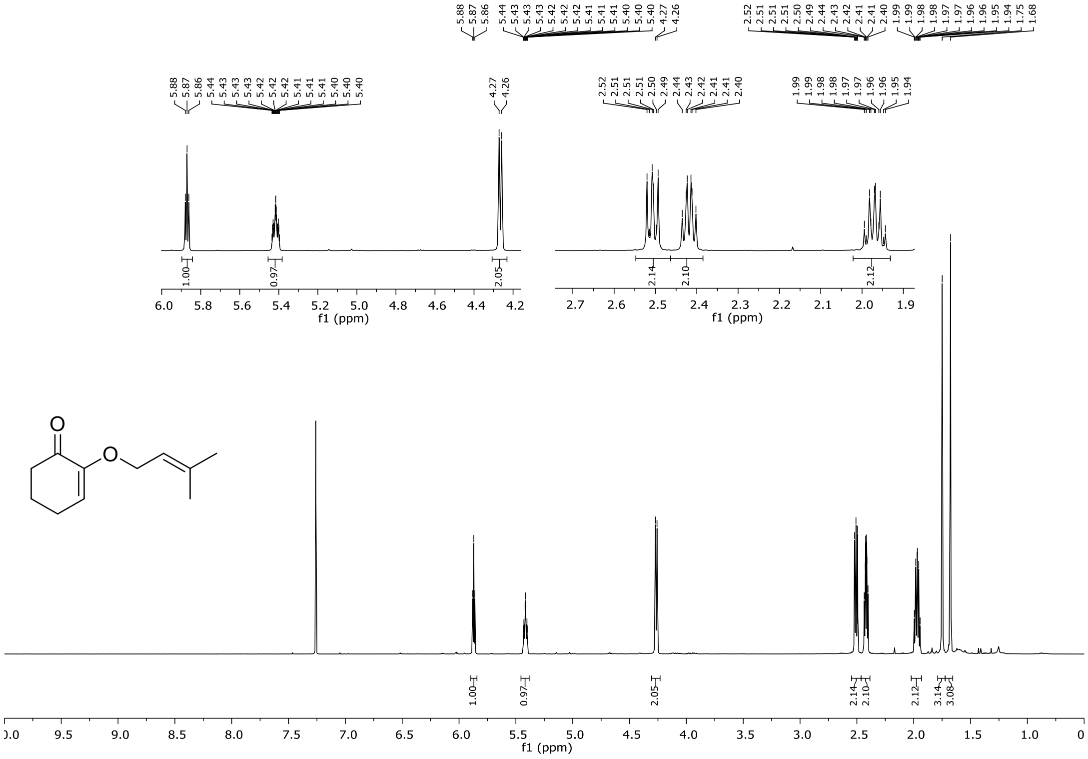


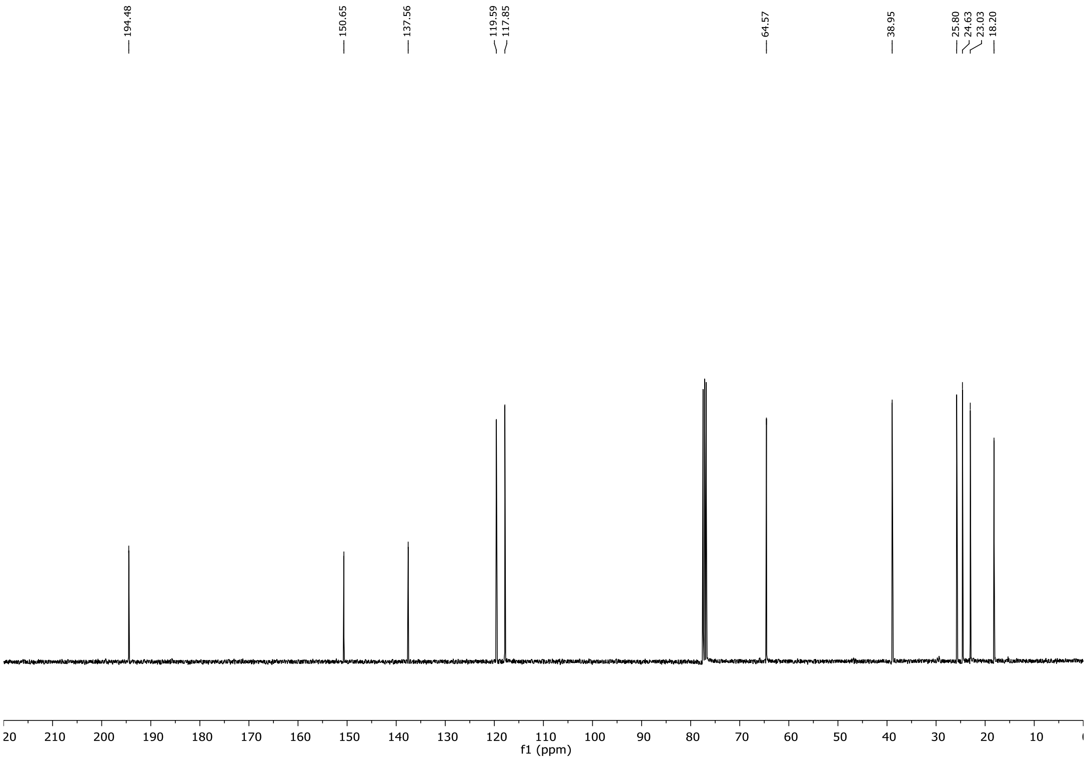


^{1}H (300 MHz) and ^{13}C{^{1}H} NMR (75 MHz) spectra of compound **2f** (C_6D_6)

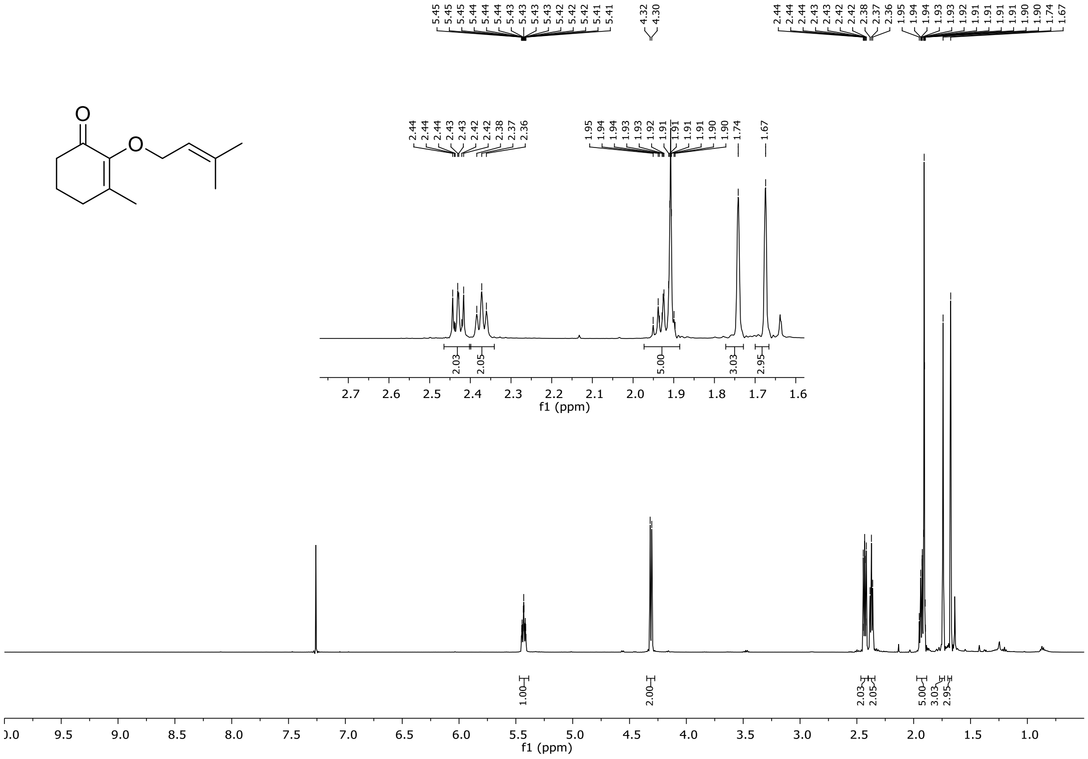


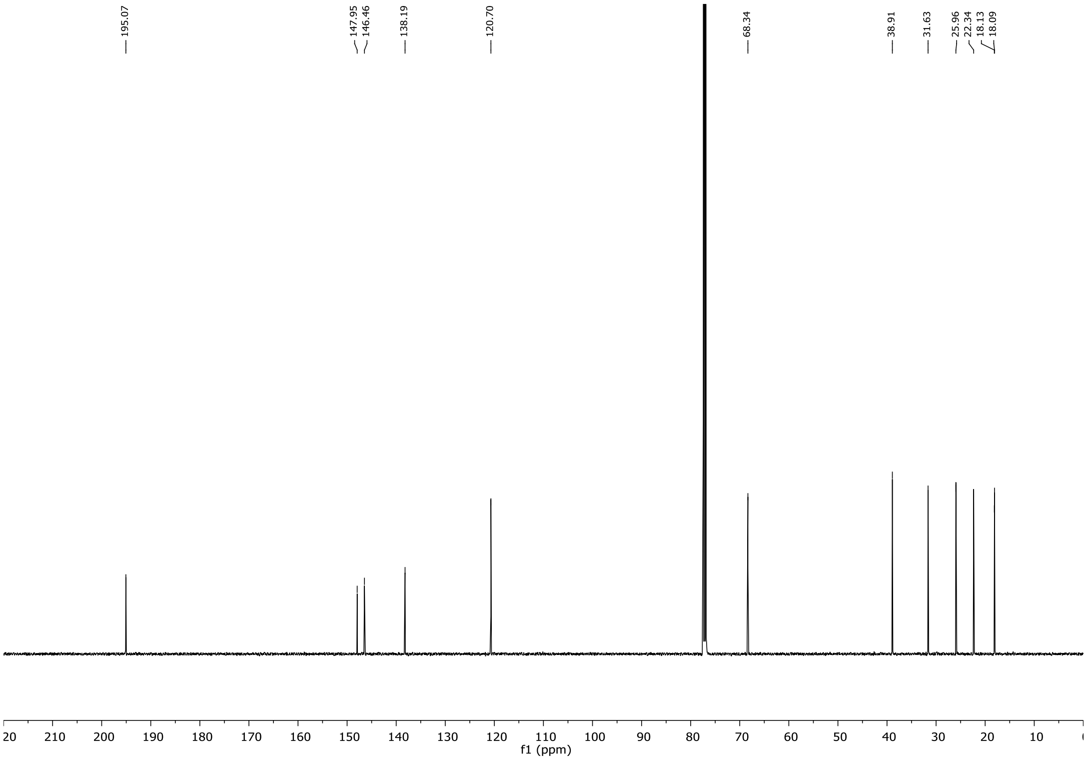
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (101 MHz) spectra of compound **3a** (CDCl$_3$)





^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **3b** (CDCl$_3$)





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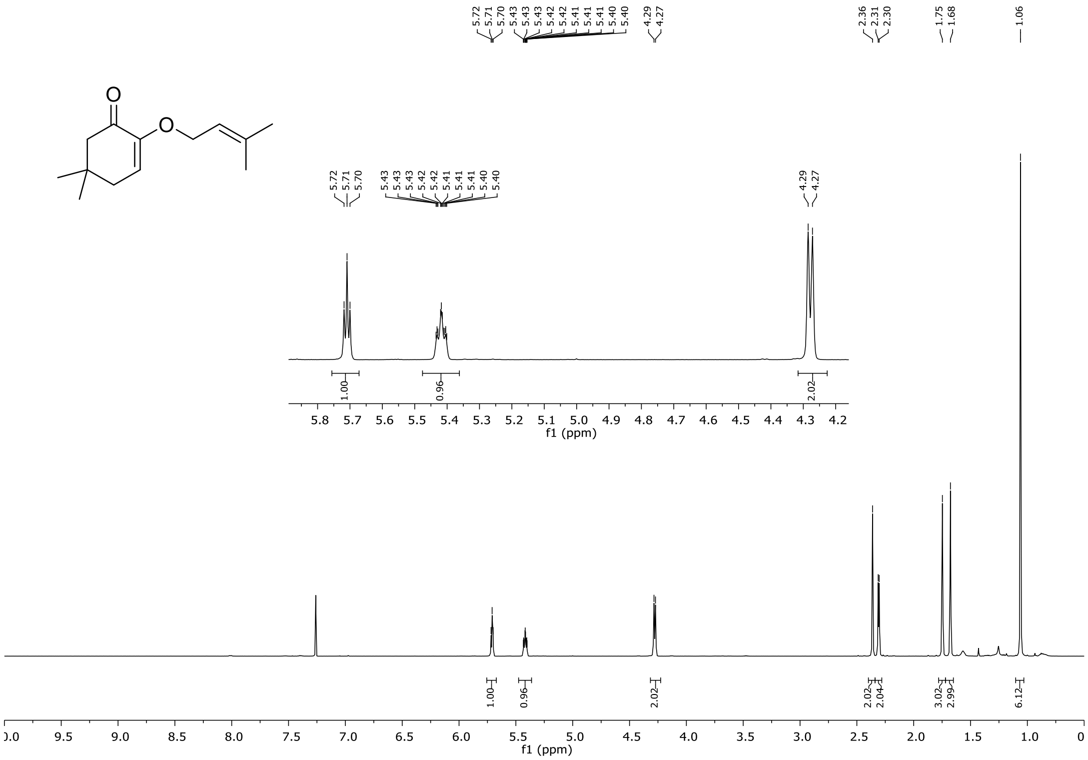
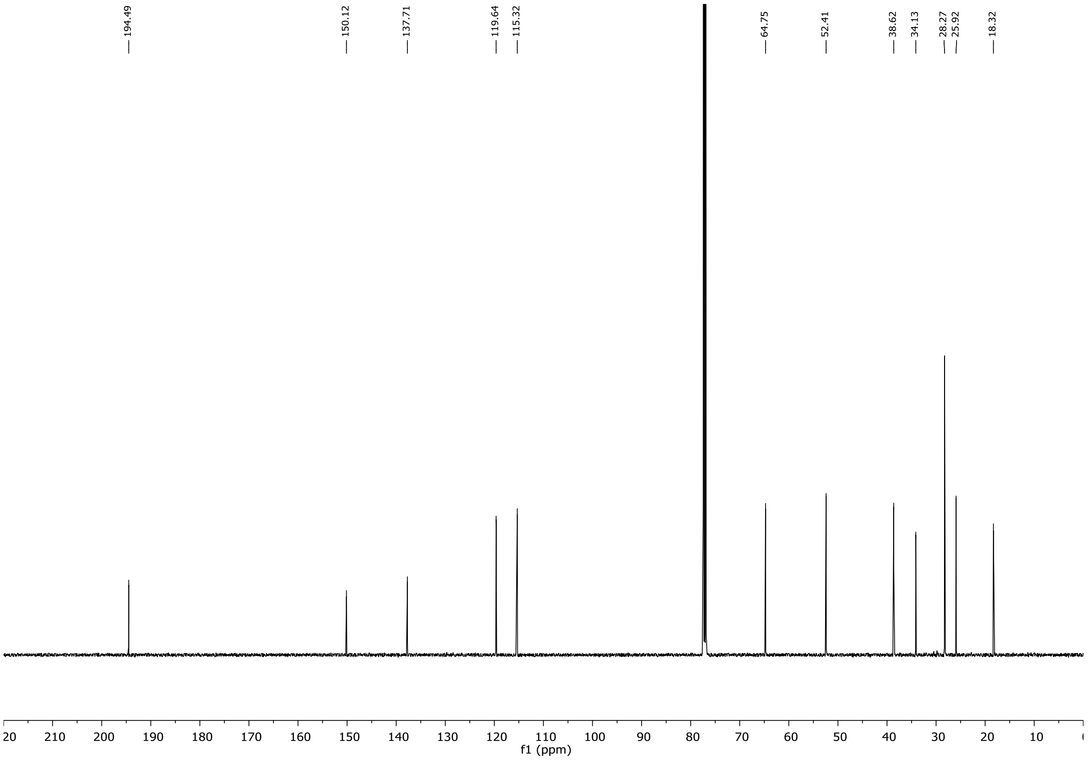
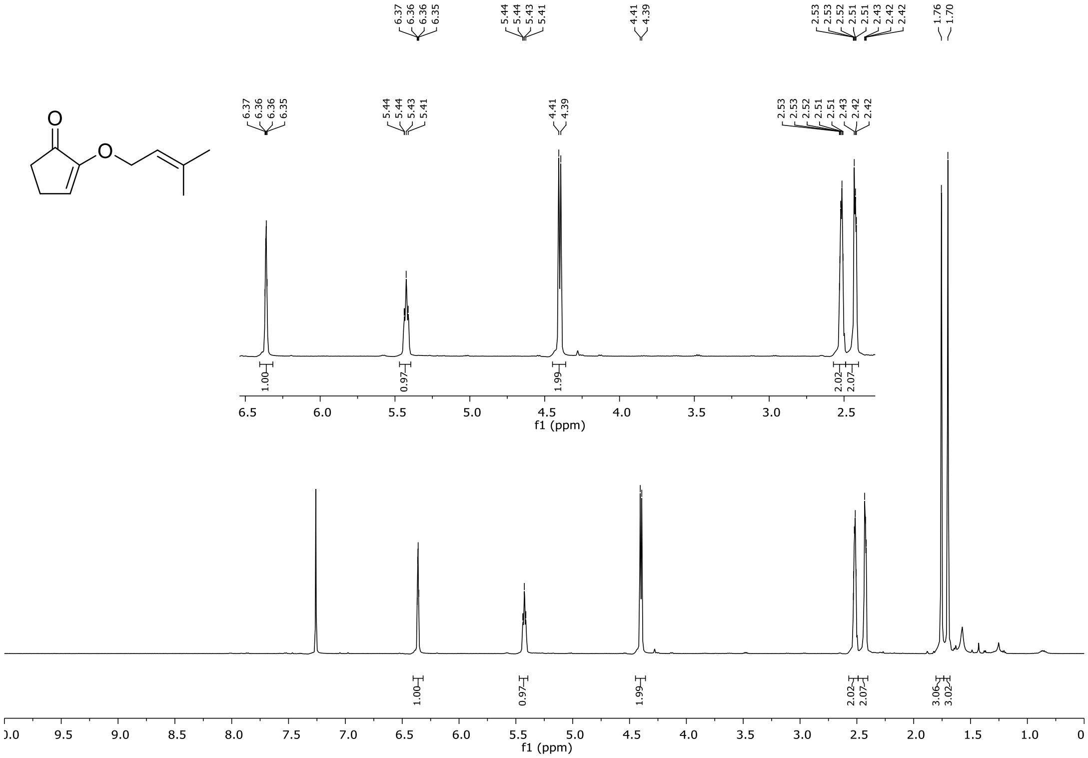
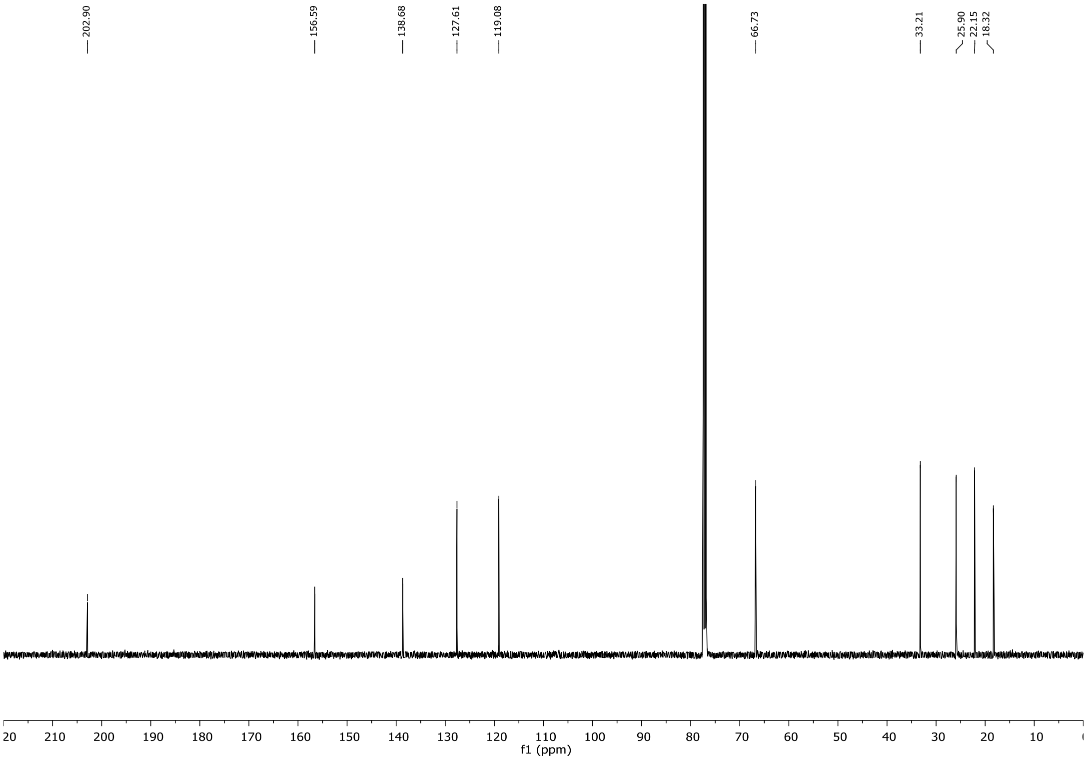


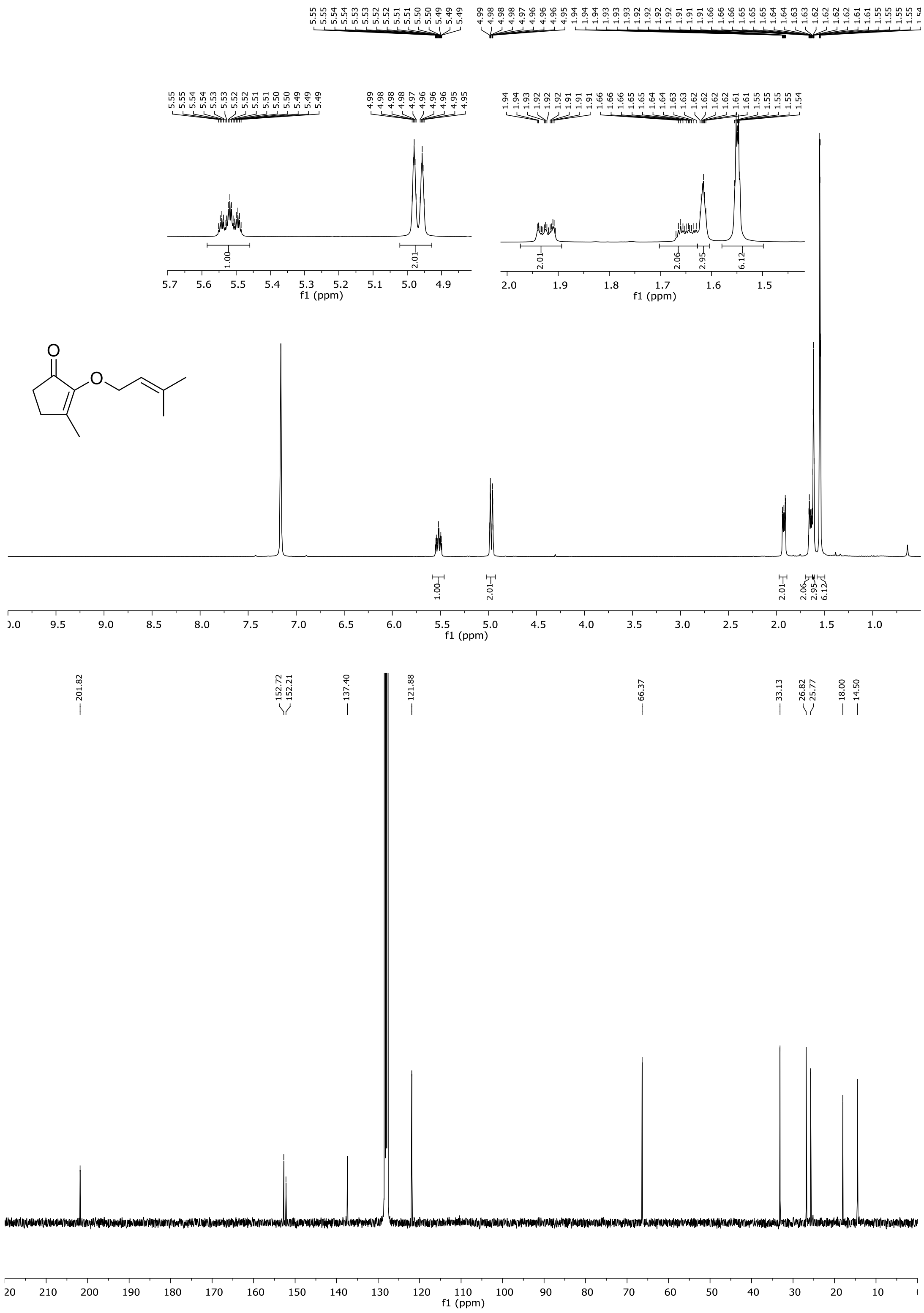


^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **3e** (CDCl$_3$)

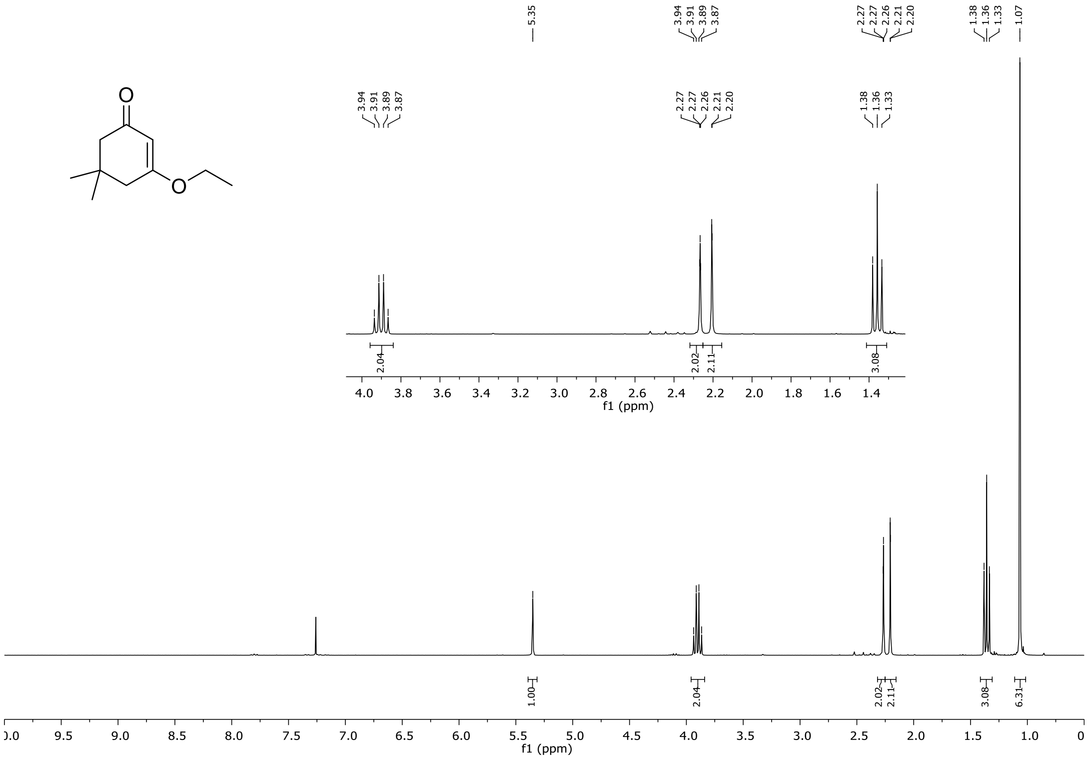


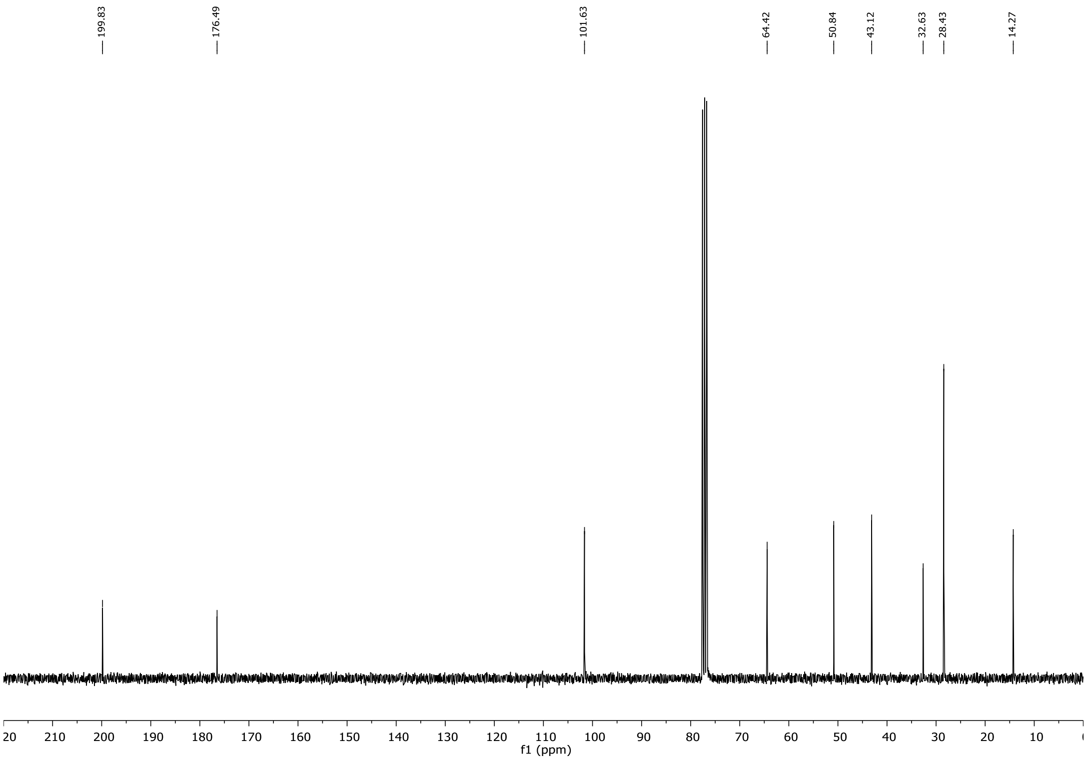


^1H (300 MHz) and $^{13}C\{^1H\}$ NMR (75 MHz) spectra of compound **3f** (C_6D_6)



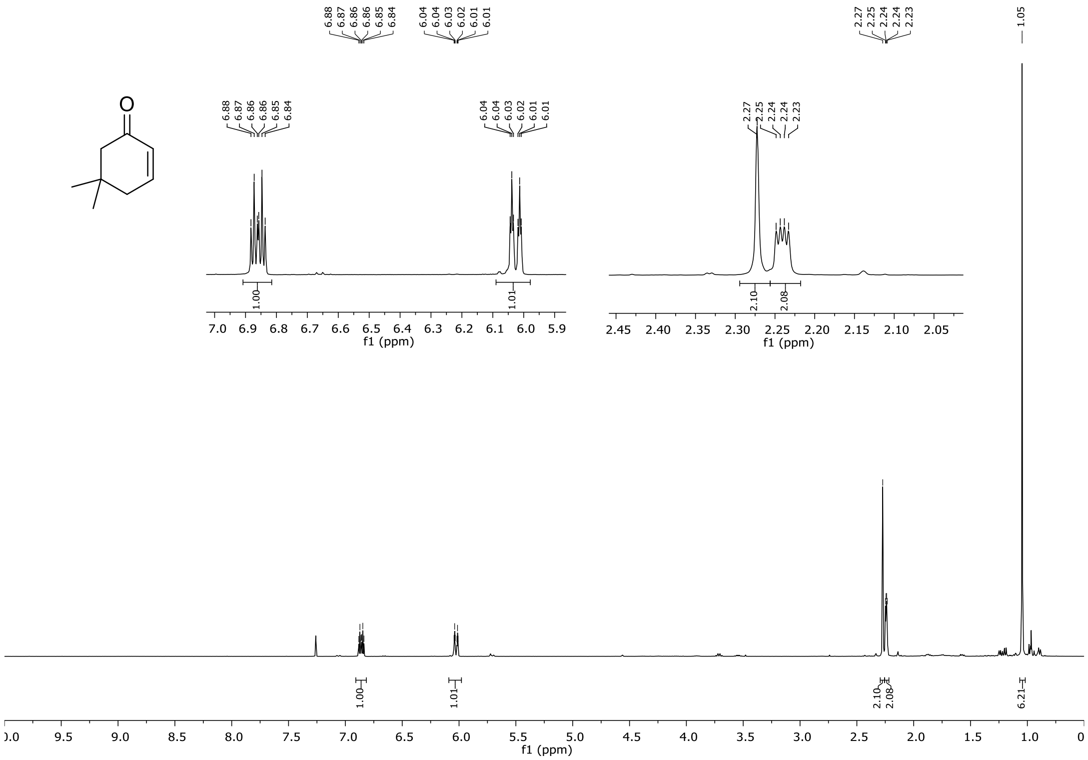
^{1}H (300 MHz) and ^{13}C{^{1}H} NMR (75 MHz) spectra of **3-Ethoxy-5,5-dimethylcyclohex-2-en-1-one** (CDCl$_3$)

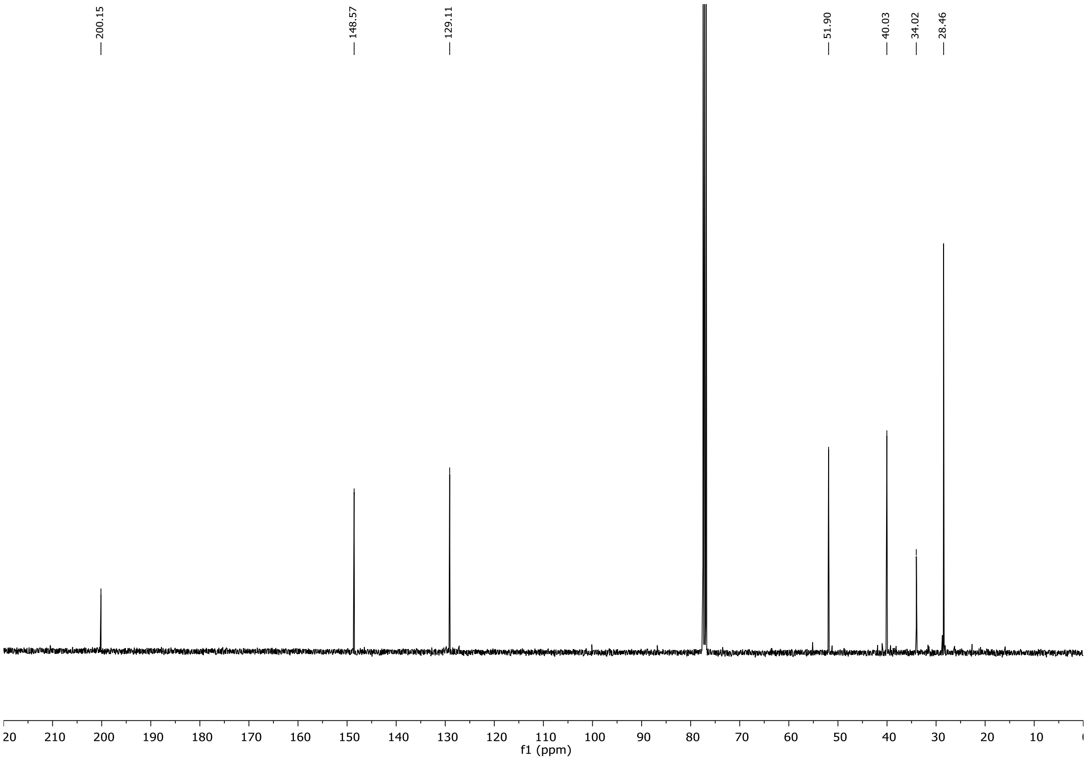




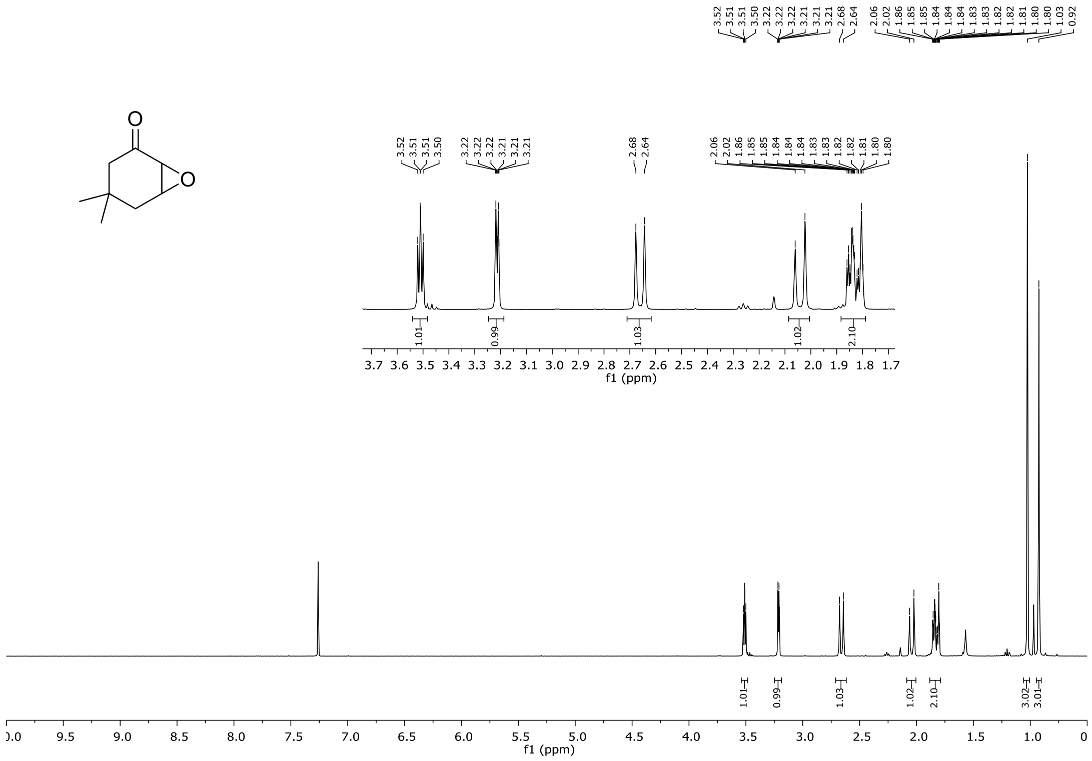
^{1}H (400 MHz) and $^{13}C\{^{1}H\}$ NMR (101 MHz) spectra of **5,5-Dimethylcyclohex-2-en-1-one**

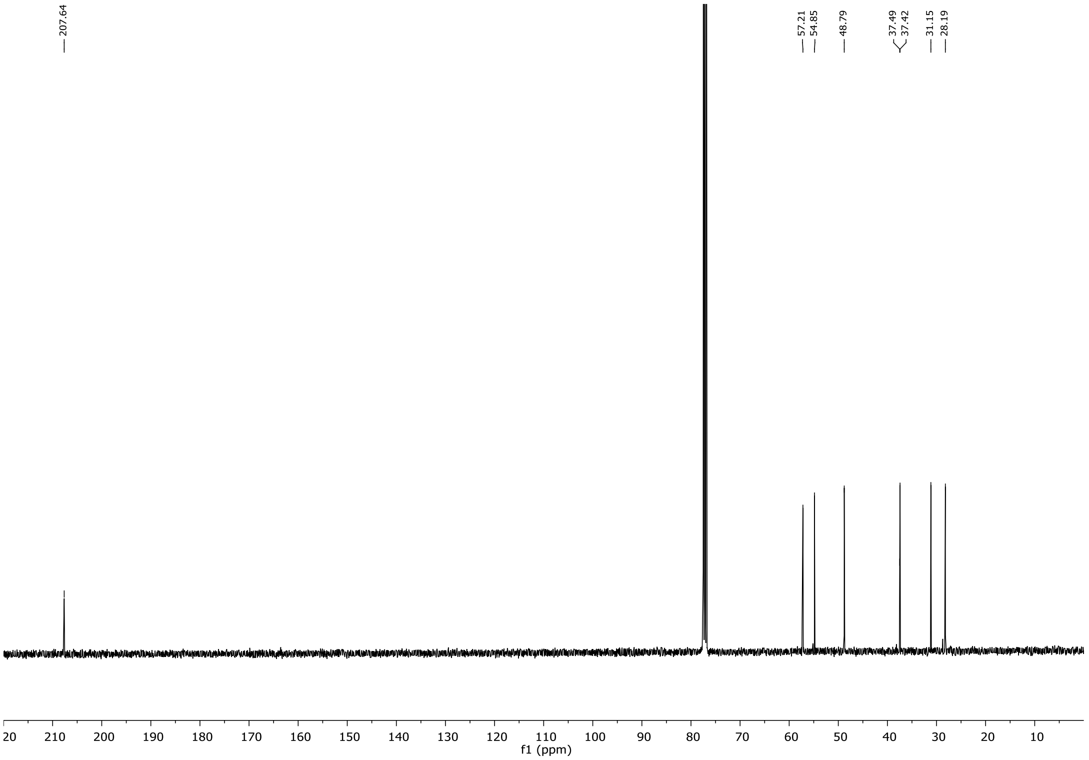
(CDCl$_3$)





^{1}H (400 MHz) and ^{13}C{^{1}H} NMR (101 MHz) spectra of compound **4c** (CDCl$_3$)





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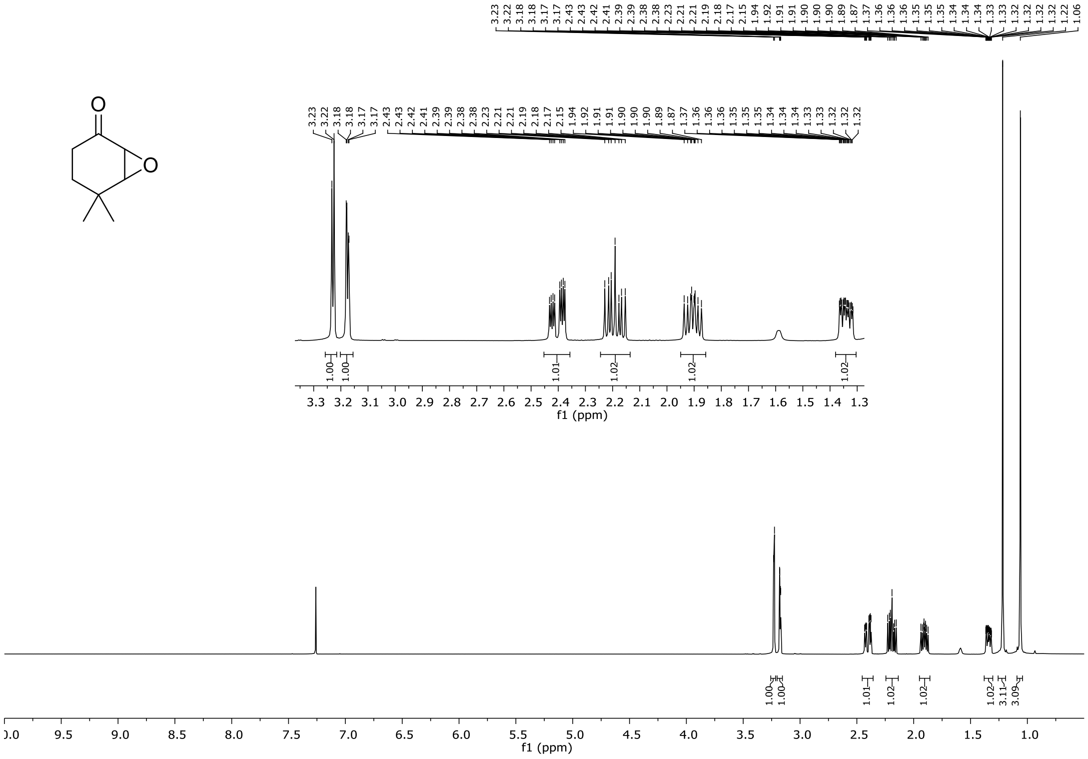
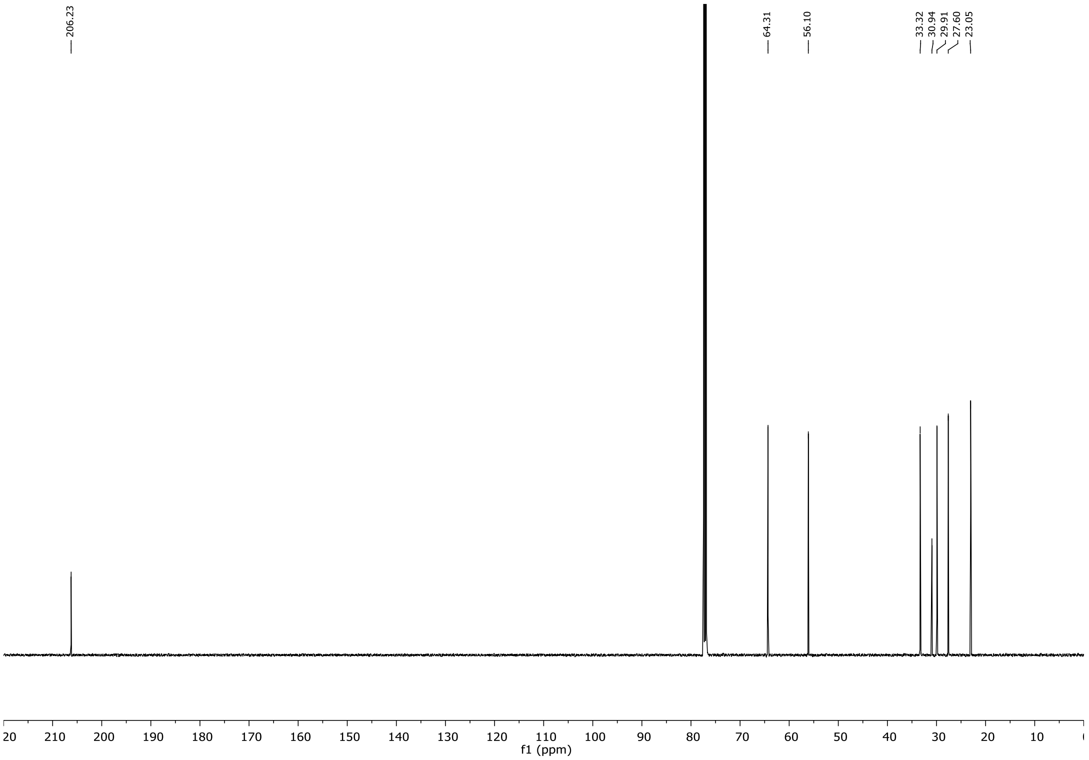
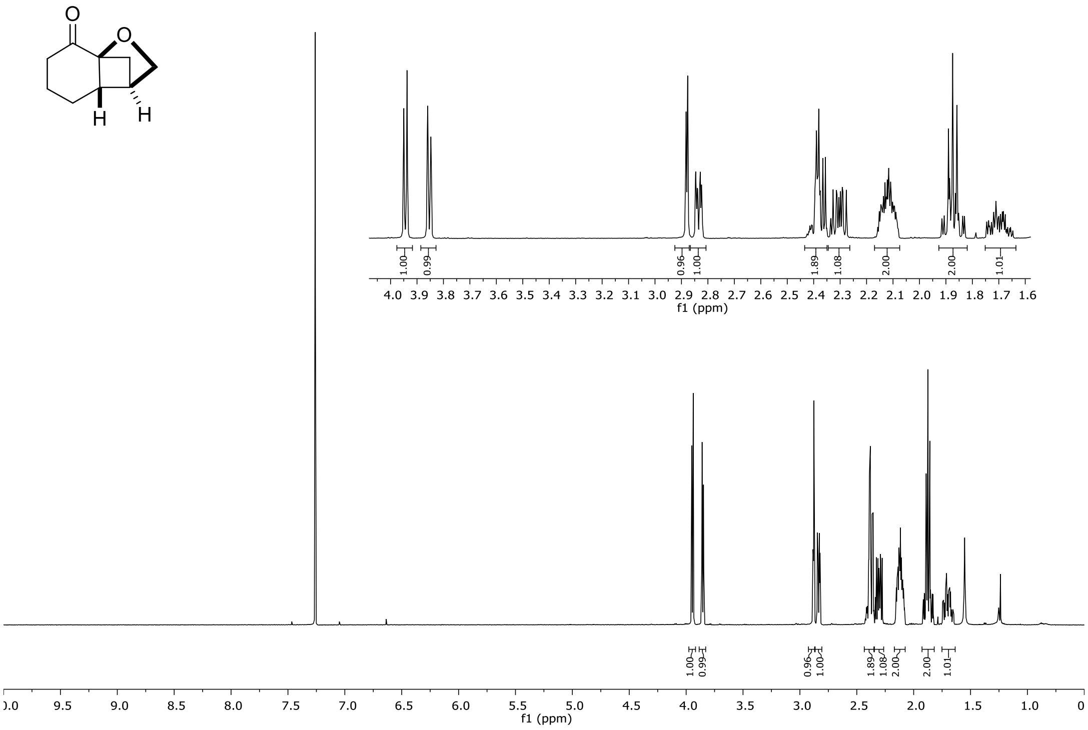
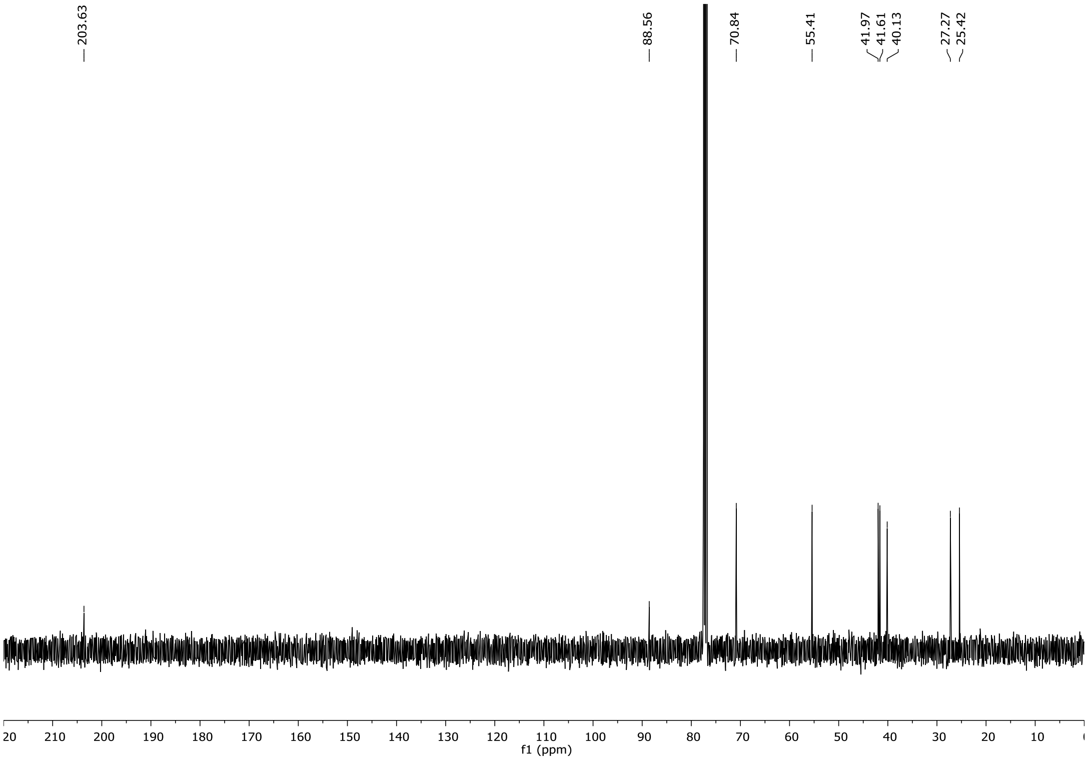


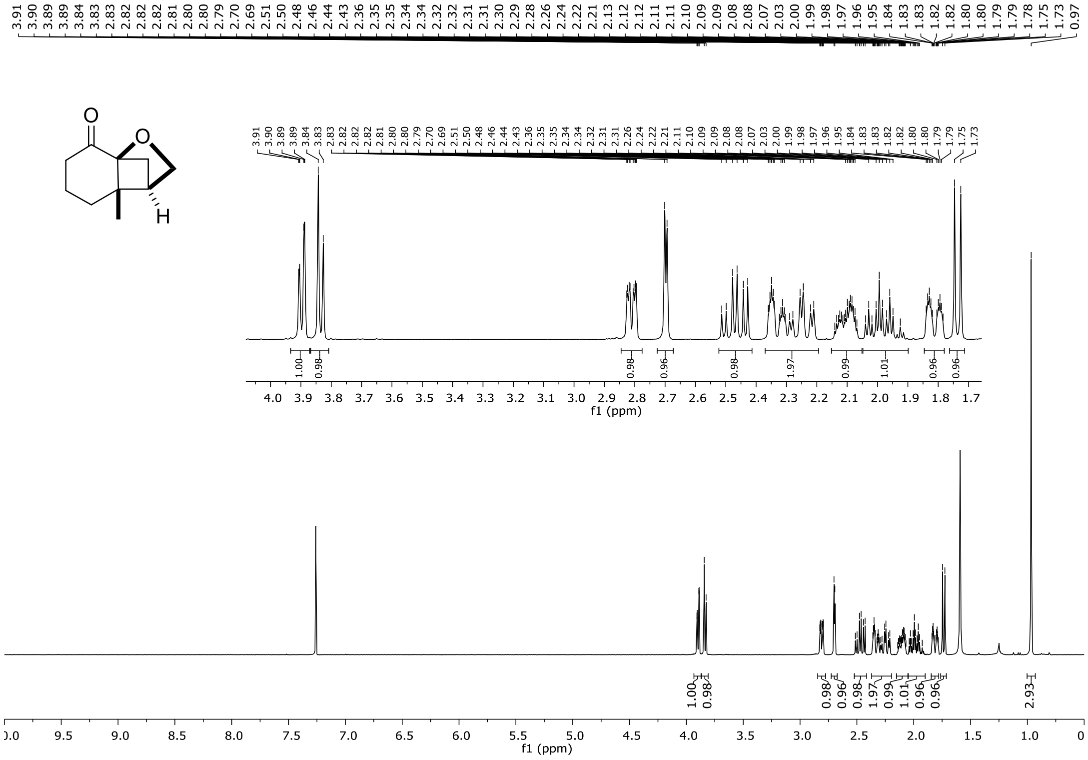


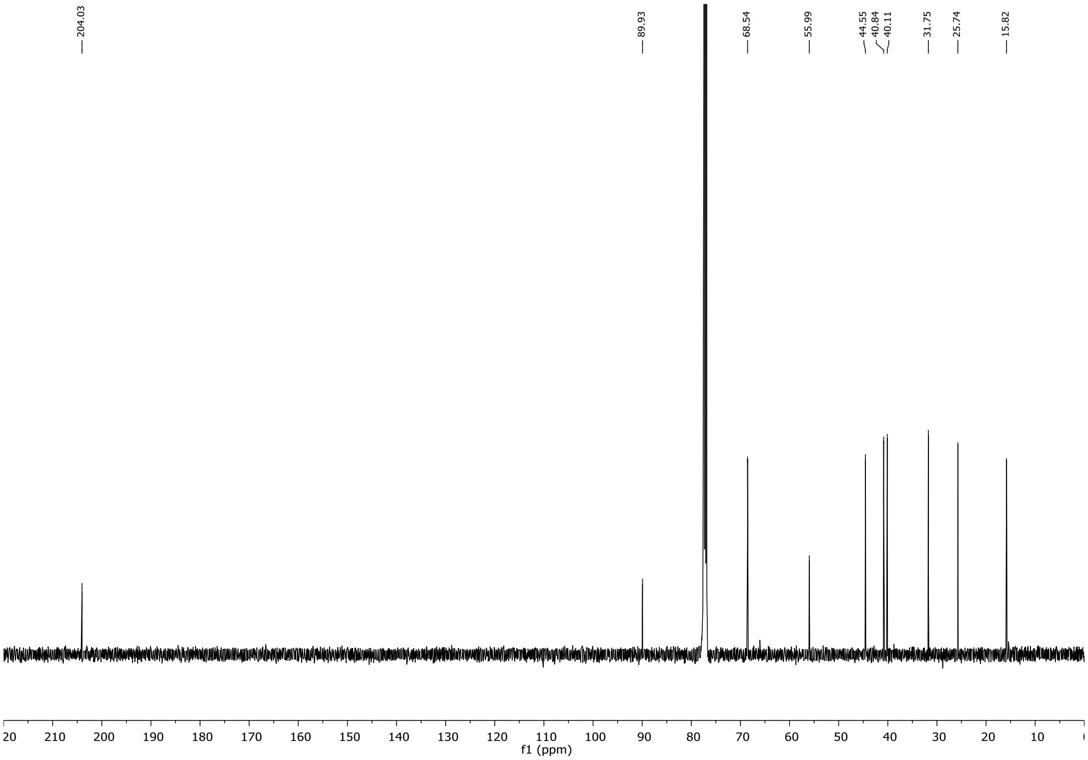
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (101 MHz) spectra of compound **8a** (CDCl$_3$)



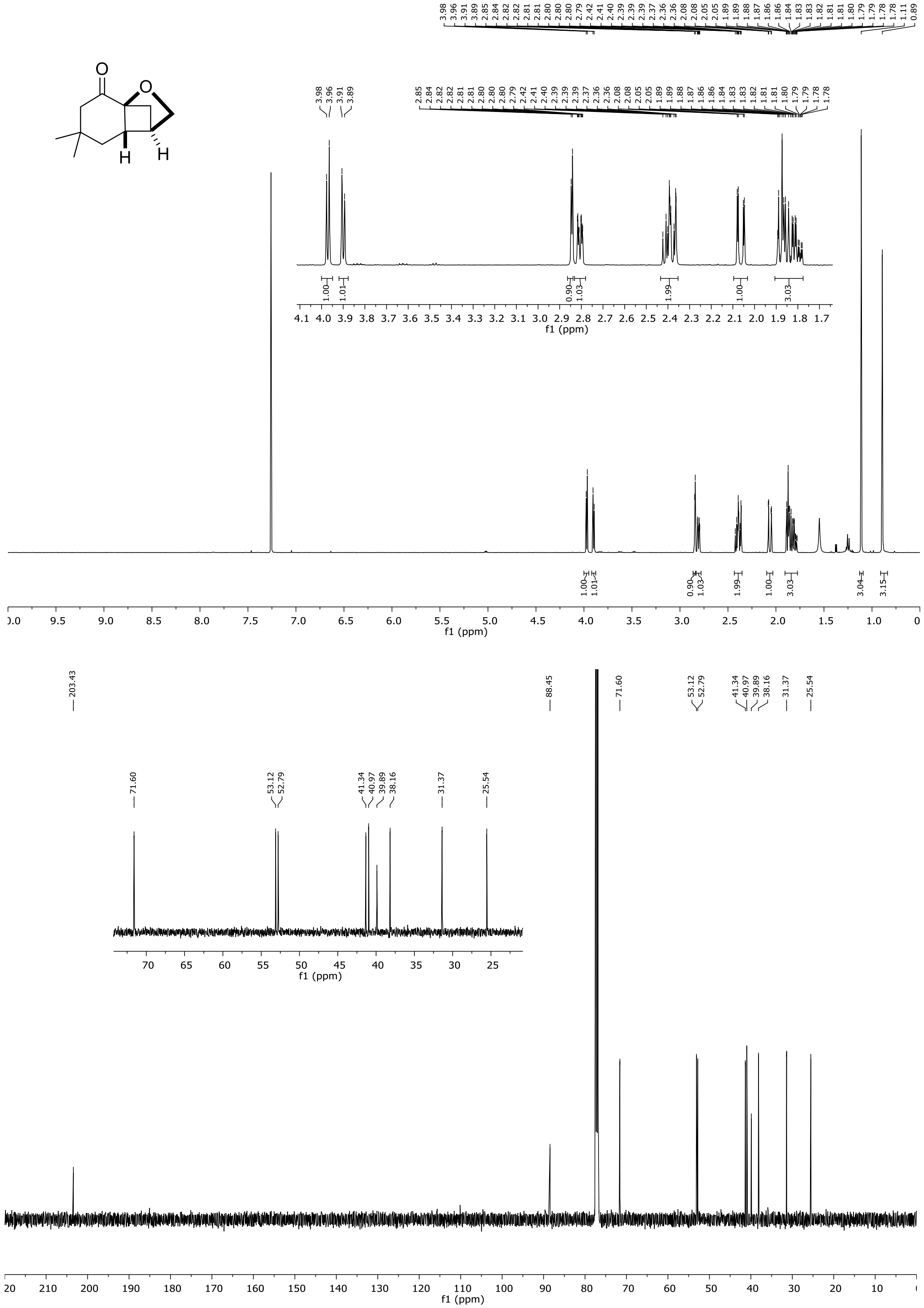


^{1}H (400 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **8b** (CDCl$_3$)

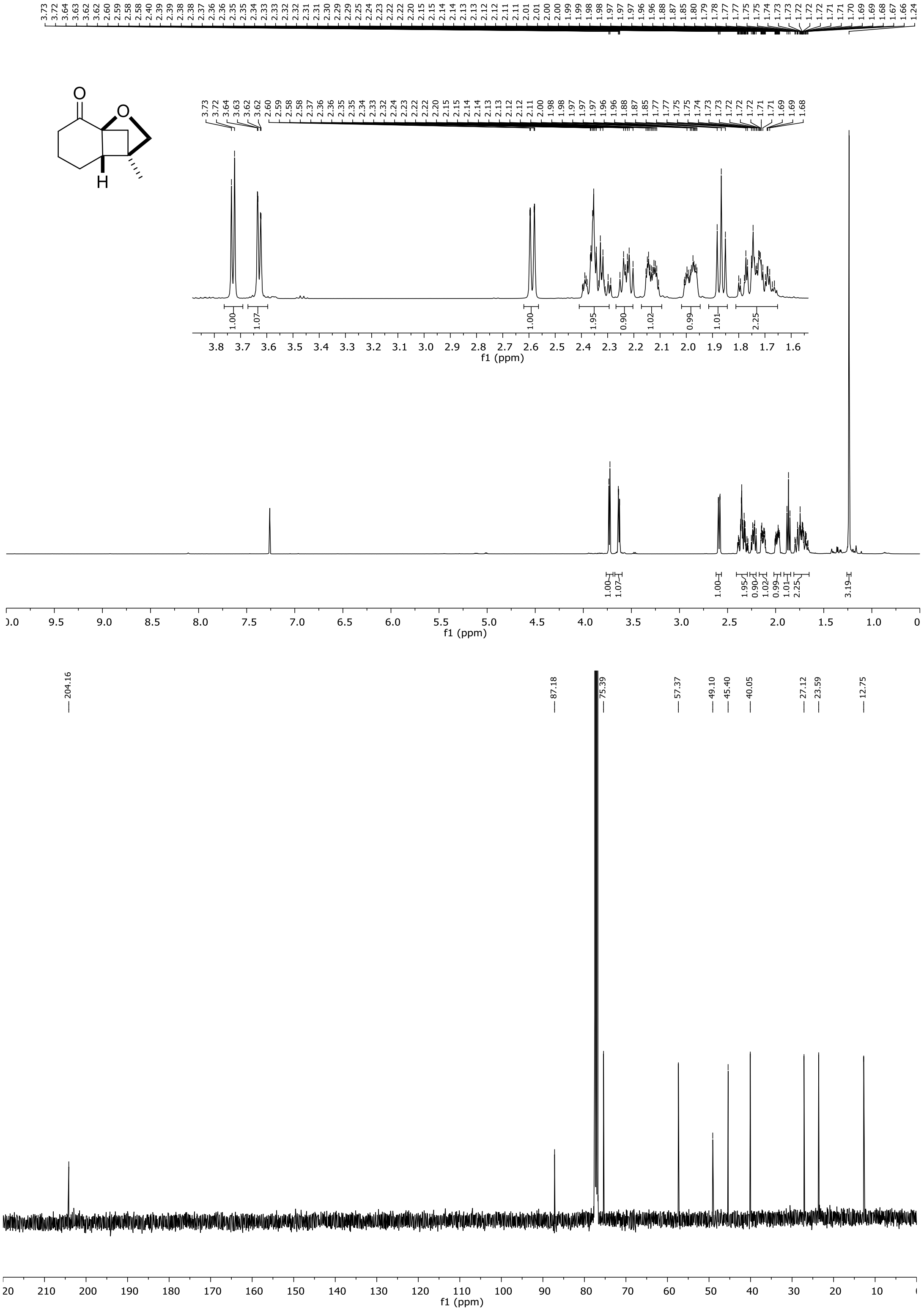




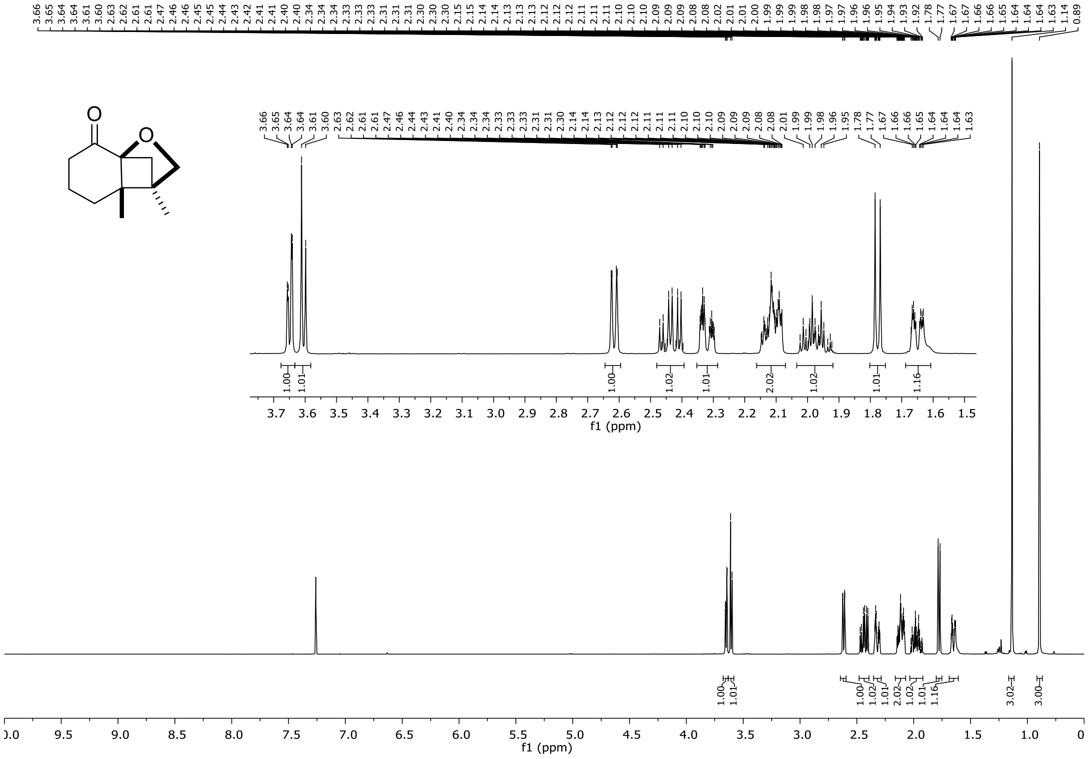
^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ NMR (126 MHz) spectra of compound **8c** ($CDCl_3$)

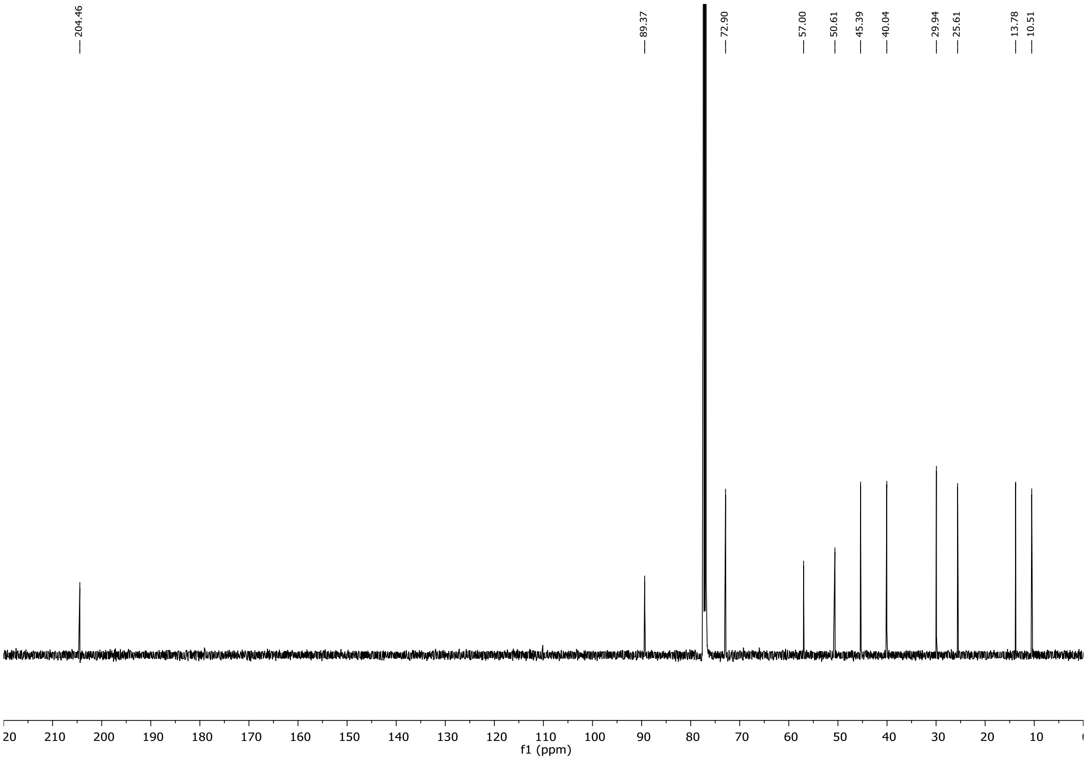


^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ NMR (101 MHz) spectra of compound **9a** ($CDCl_3$)

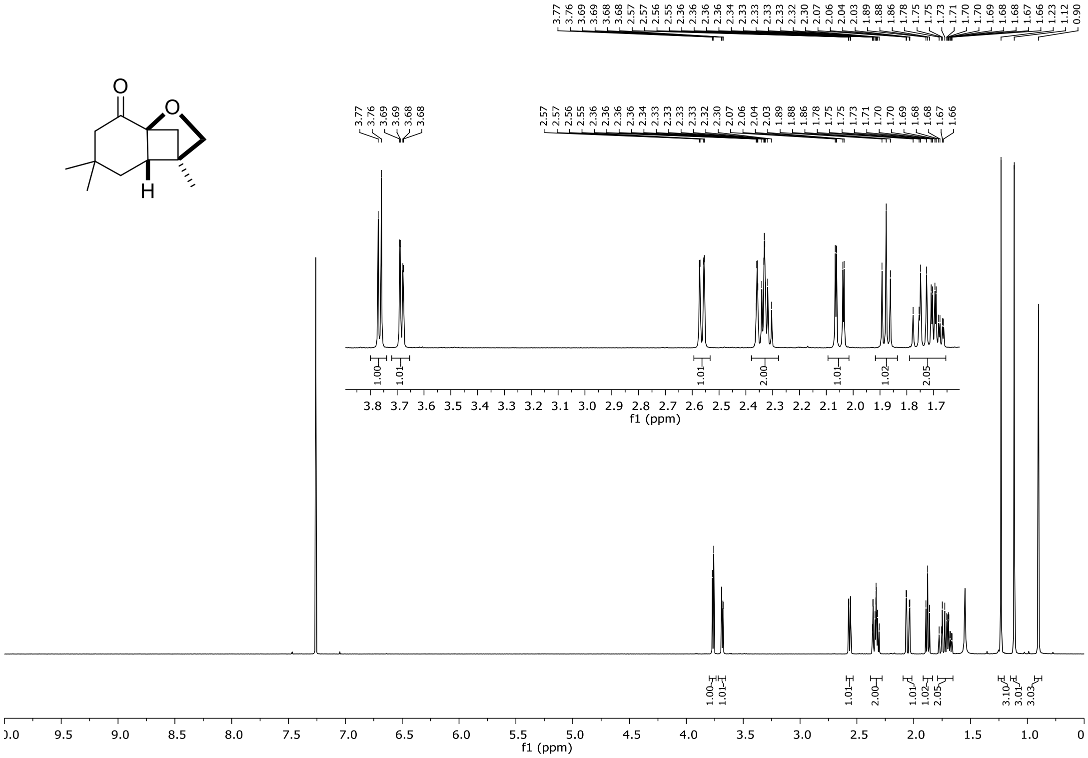


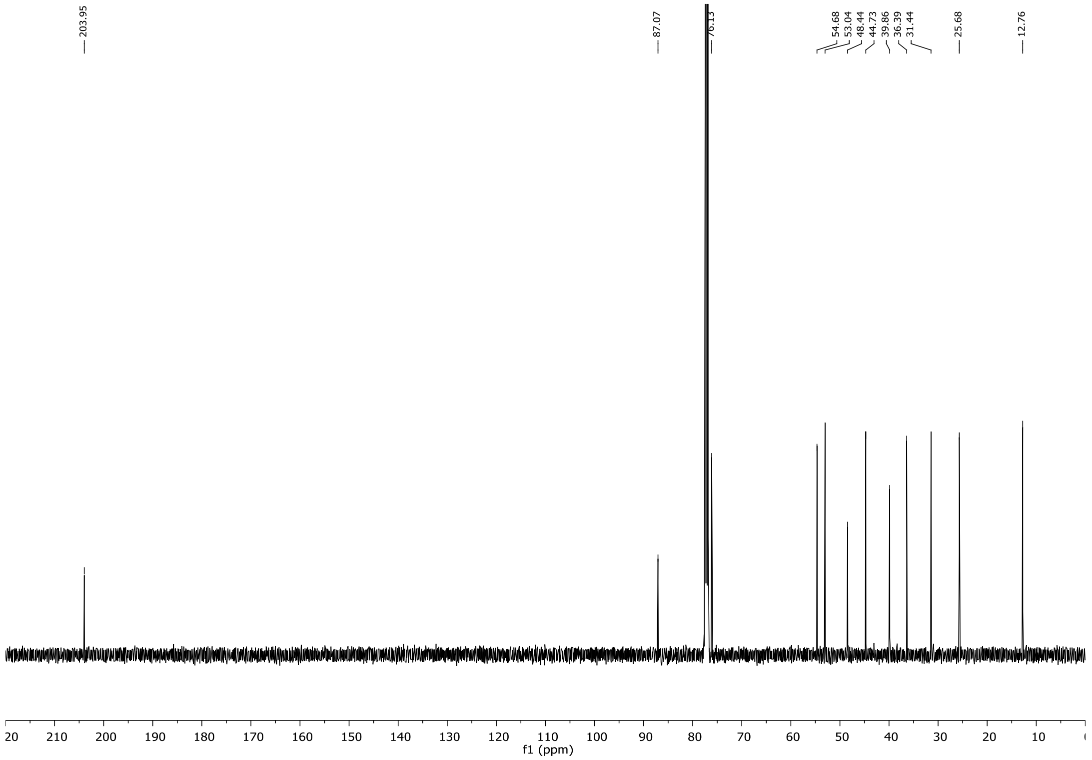
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **9b** (CDCl$_3$)



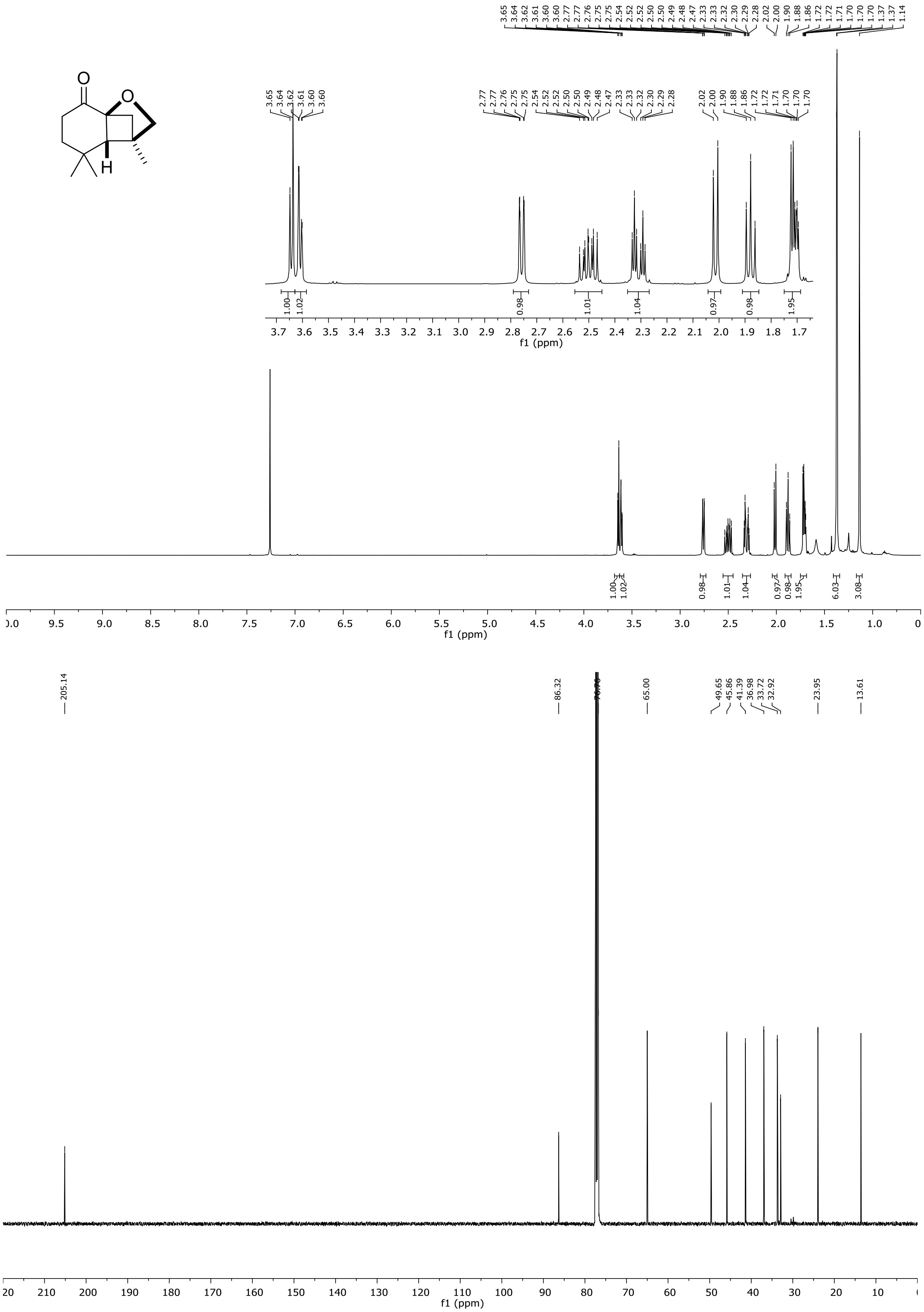


^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **9c** (CDCl$_3$)

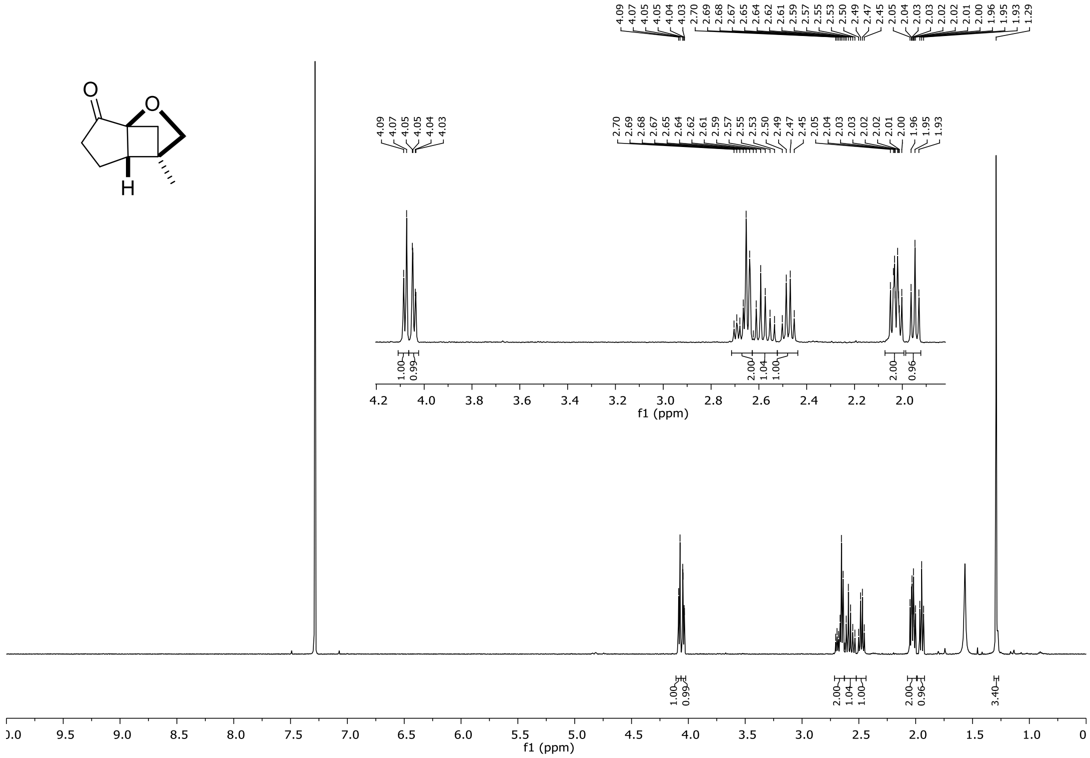


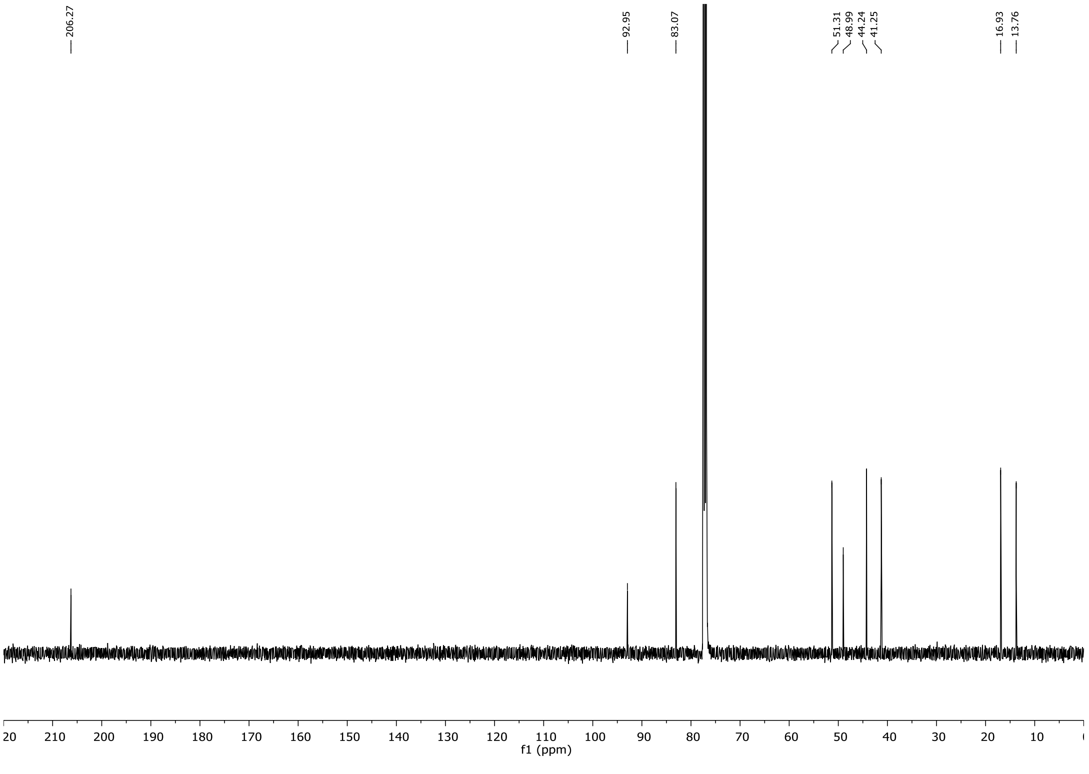


^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ NMR (126 MHz) spectra of compound **9d** ($CDCl_3$)

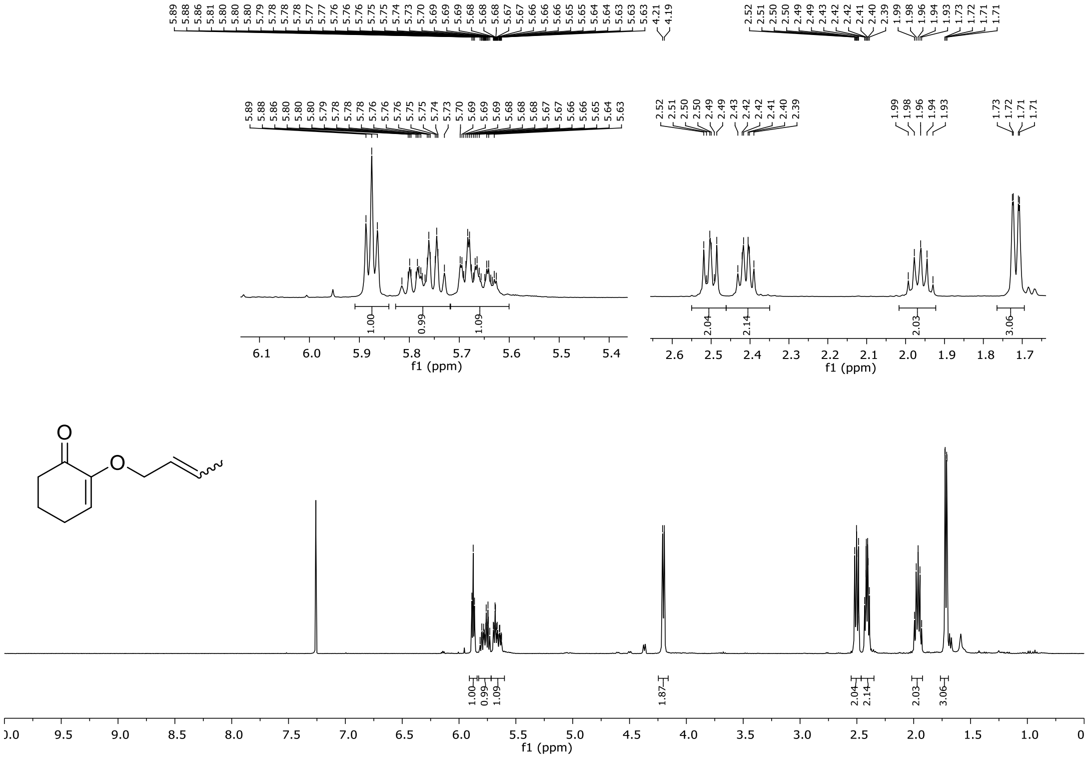


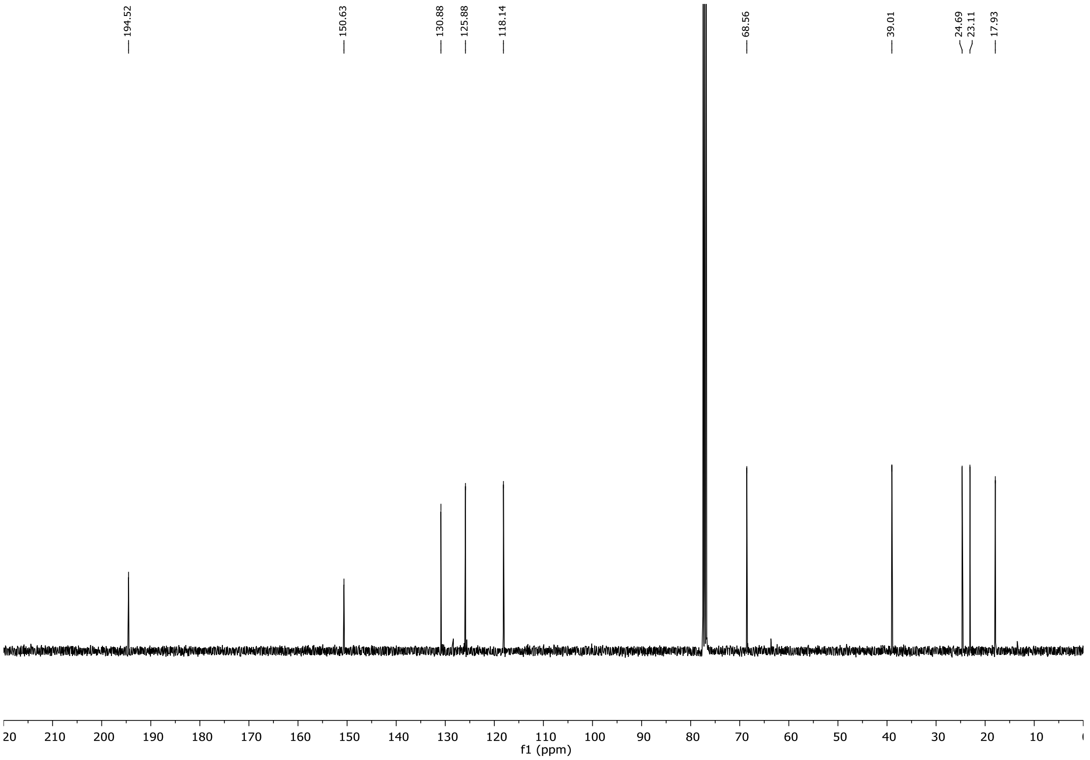
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **9e** ($CDCl_3$)



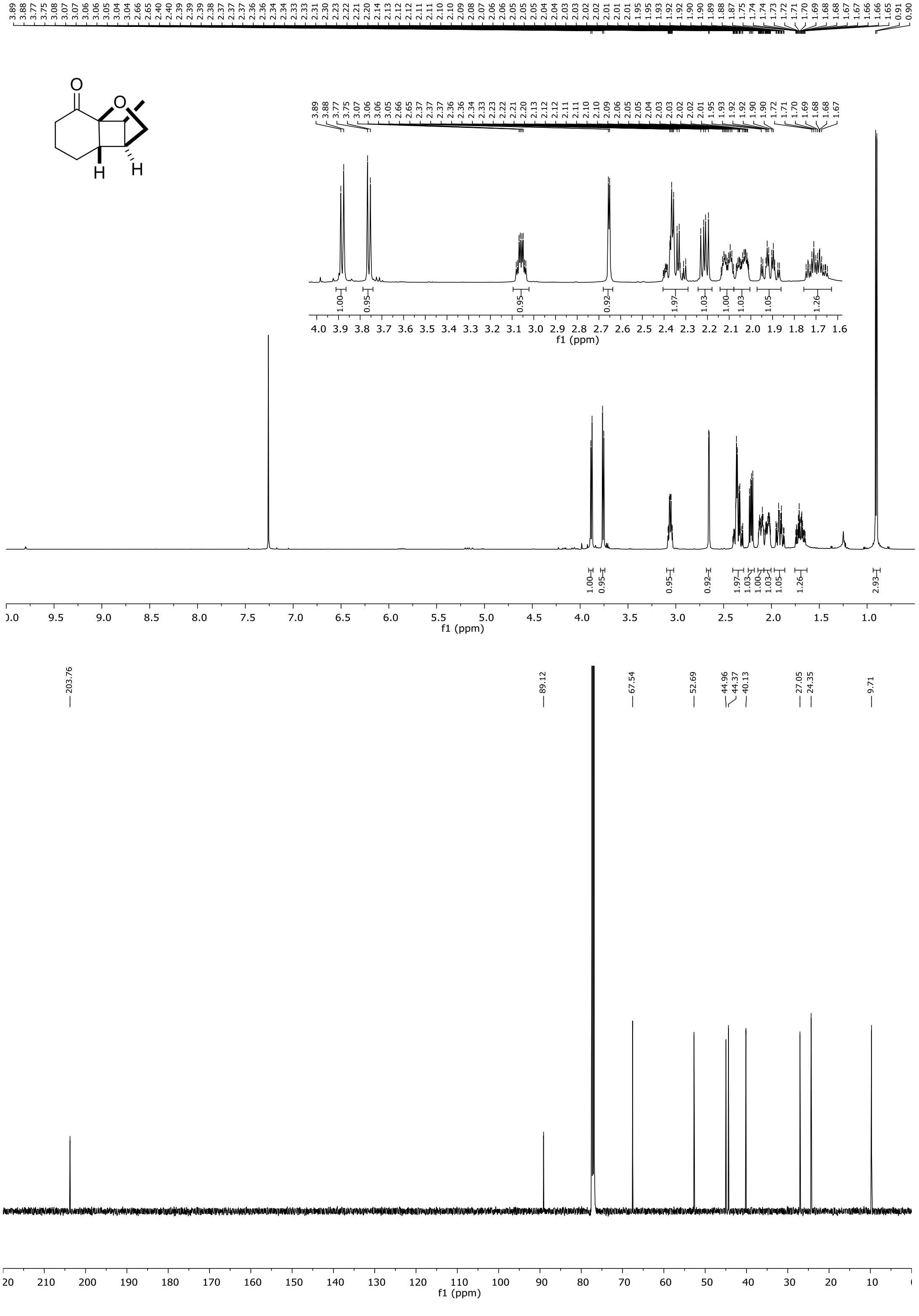


^{1}H (400 MHz) and ^{13}C{^{1}H} NMR (101 MHz) spectra of compound *E*-**10**/*Z*-**10** ($CDCl_3$)

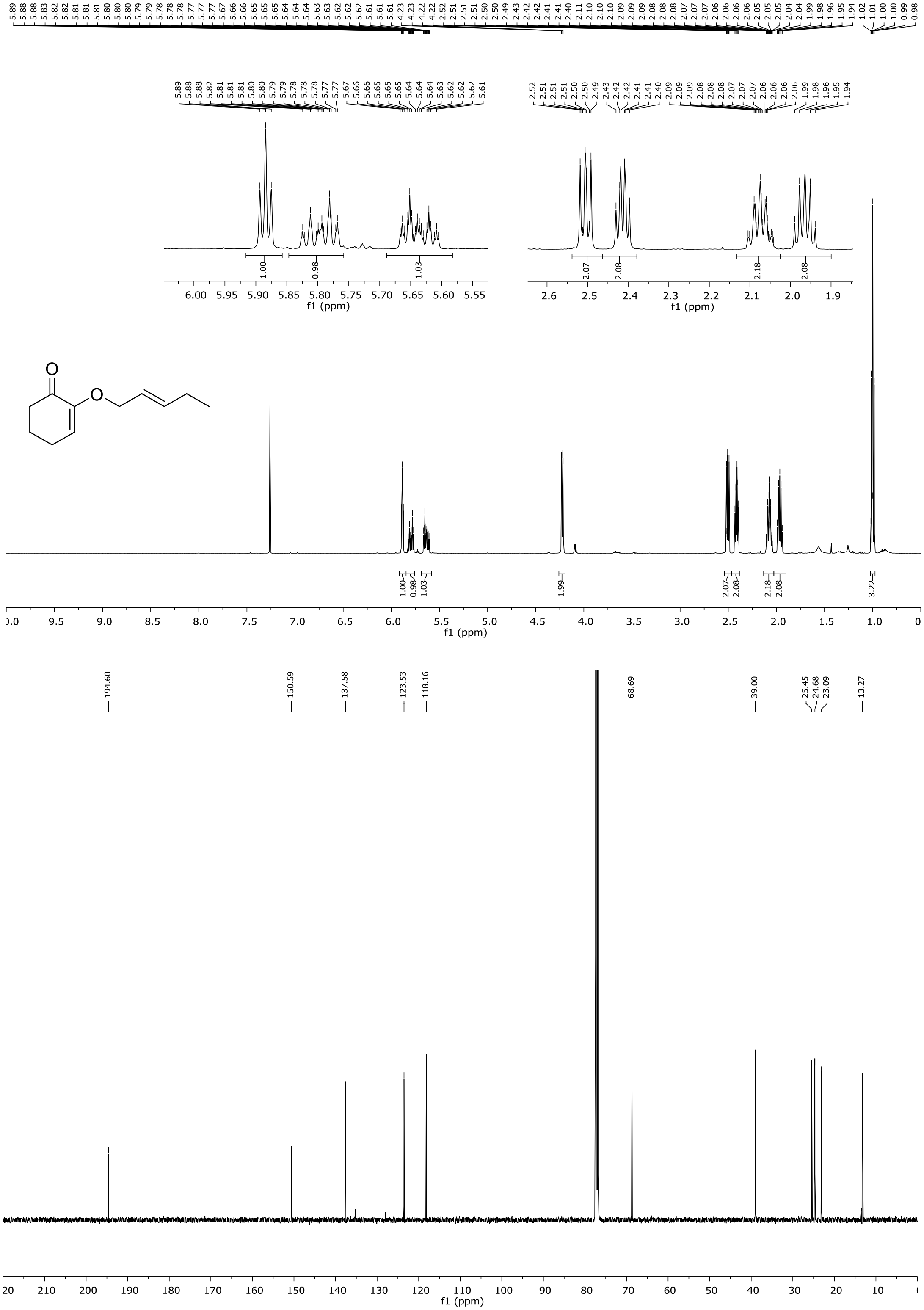




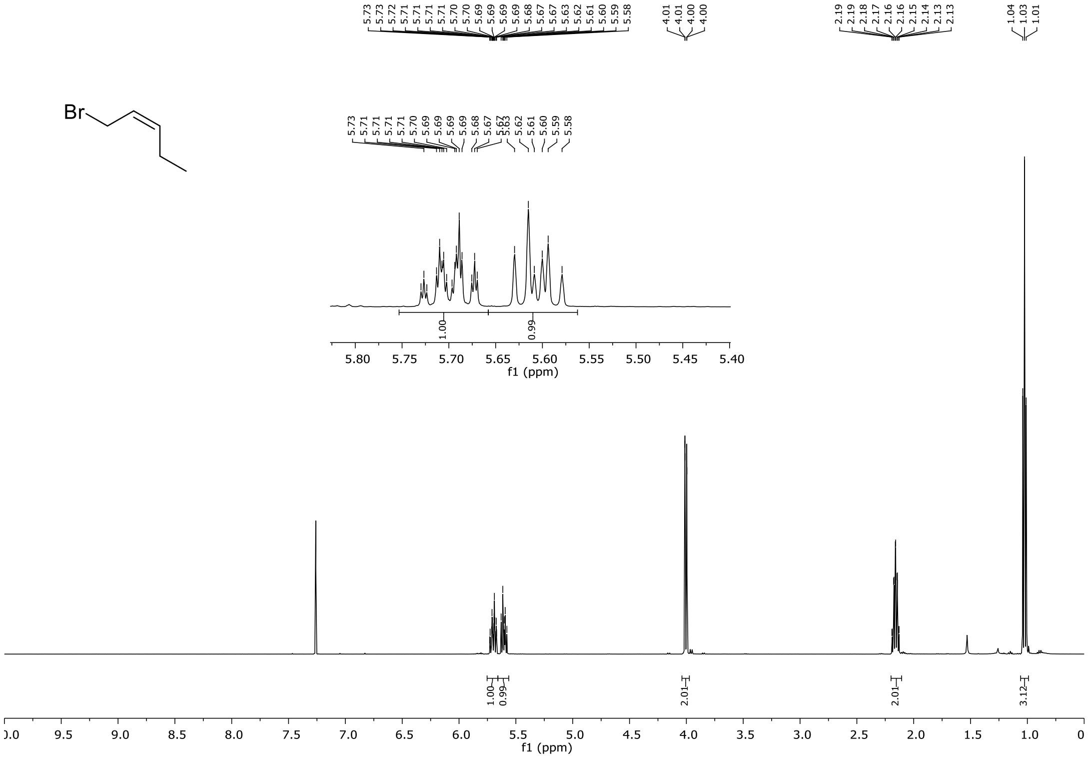
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **11** (CDCl$_3$)

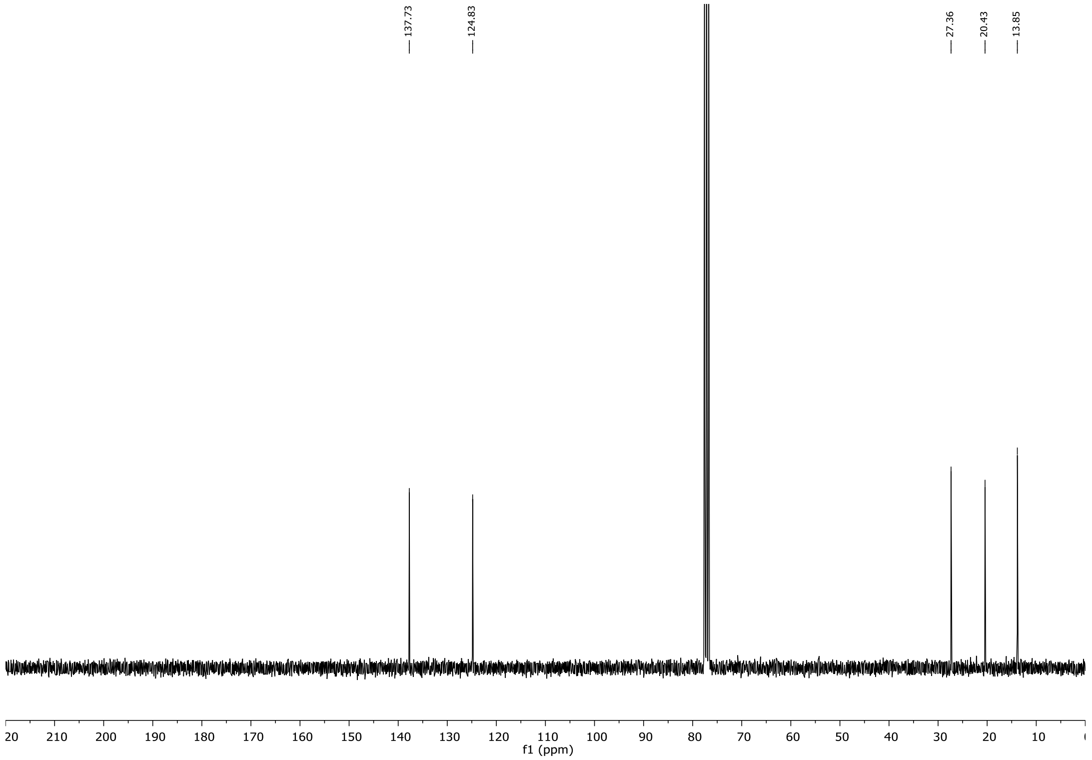


^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound *E*-**12** (CDCl$_3$)

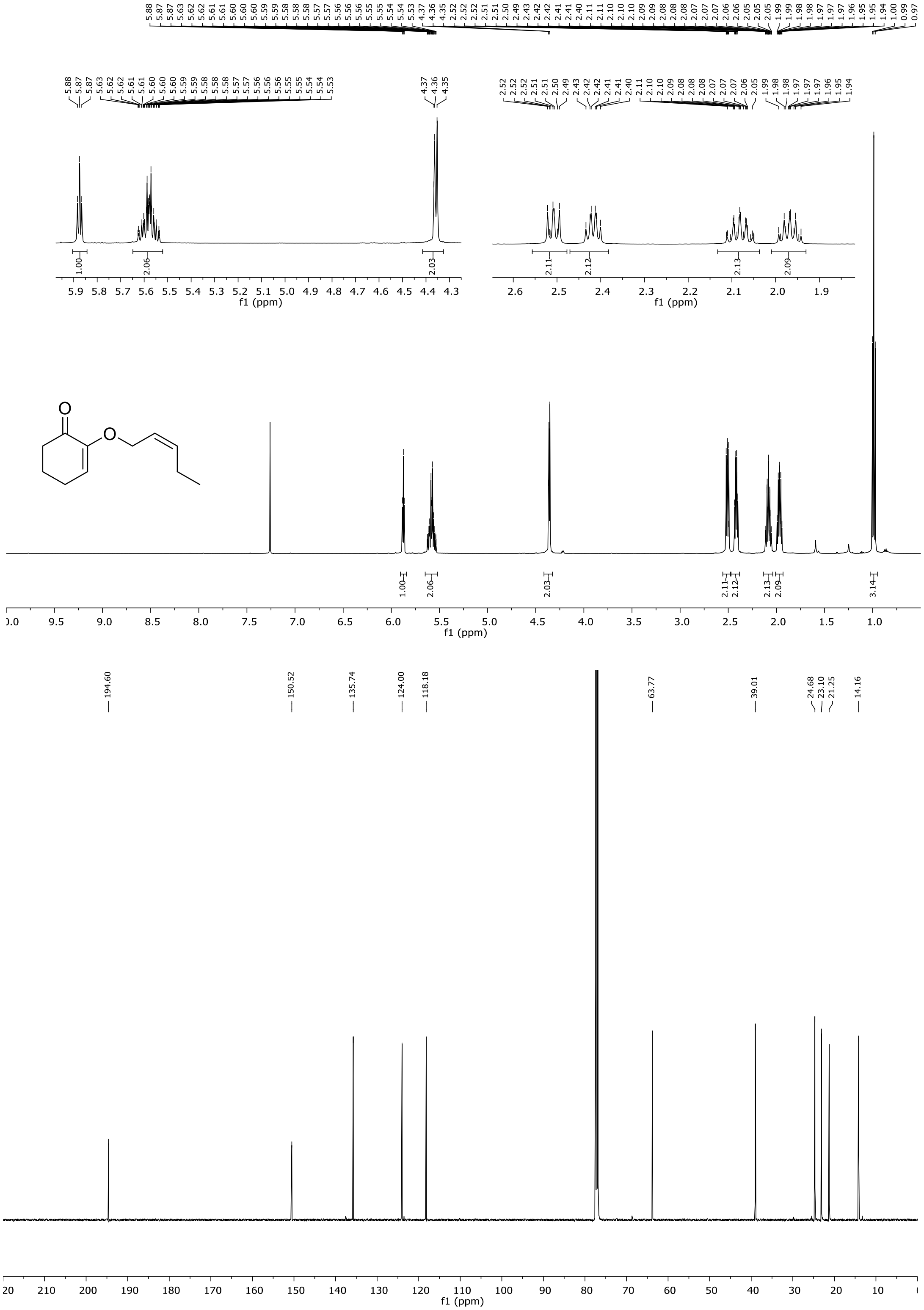


^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (75 MHz) spectra of ***(Z)*-1-Bromopent-2-ene** ($CDCl_3$)

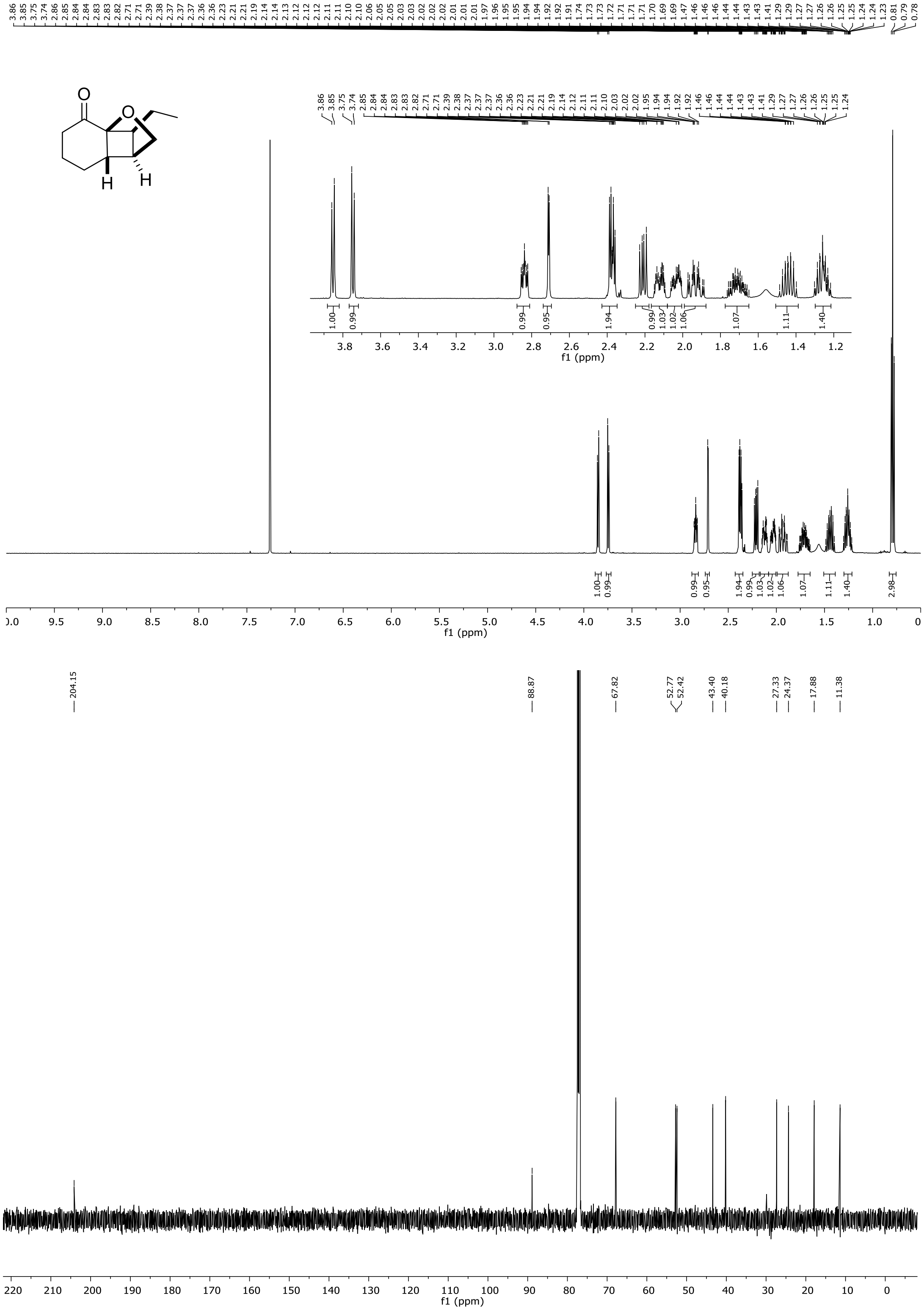




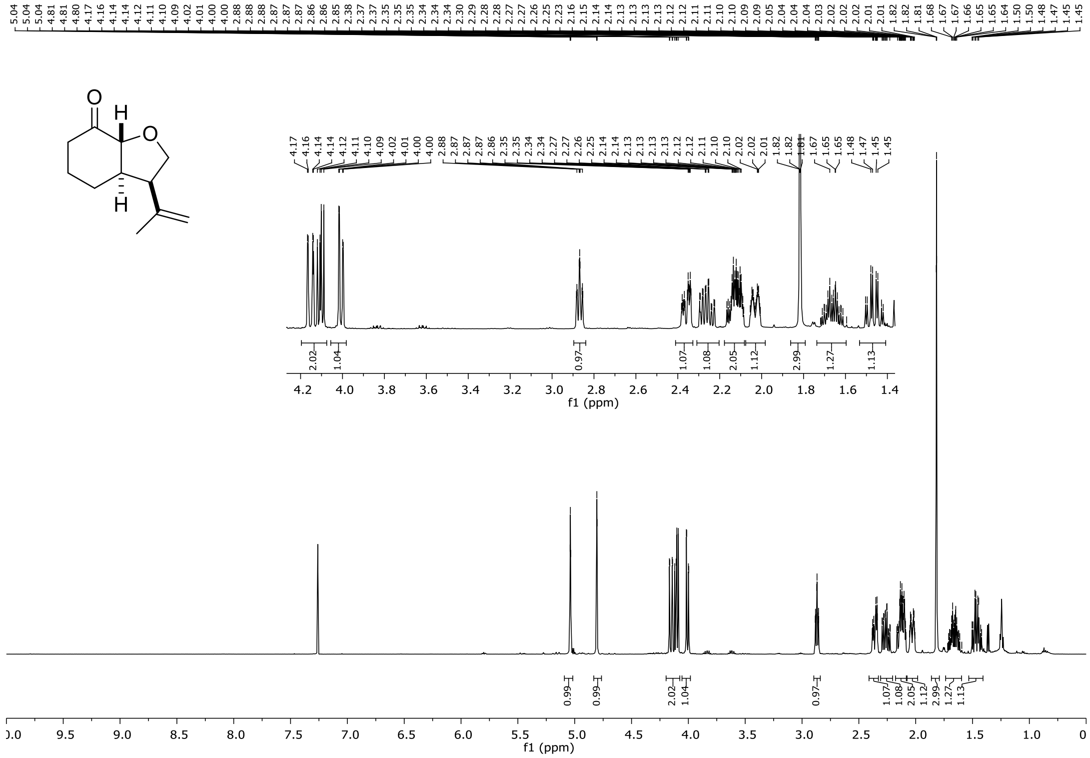
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound *Z*-**12** ($CDCl_3$)

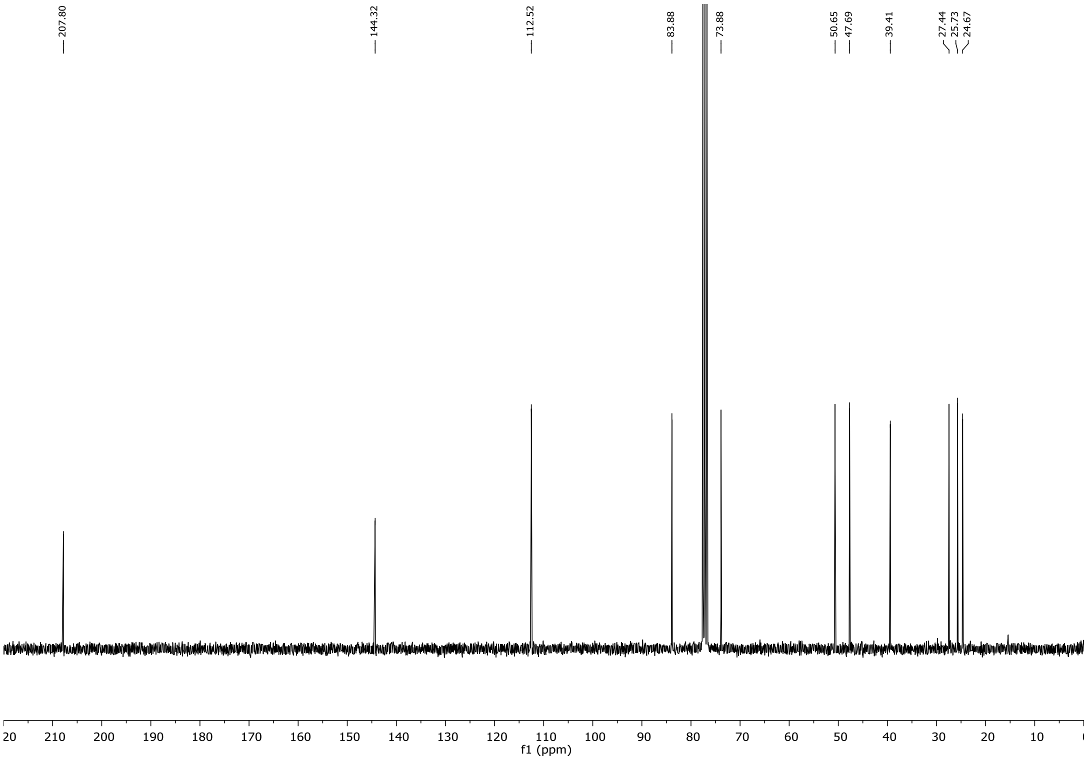


^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **13** (CDCl$_3$)

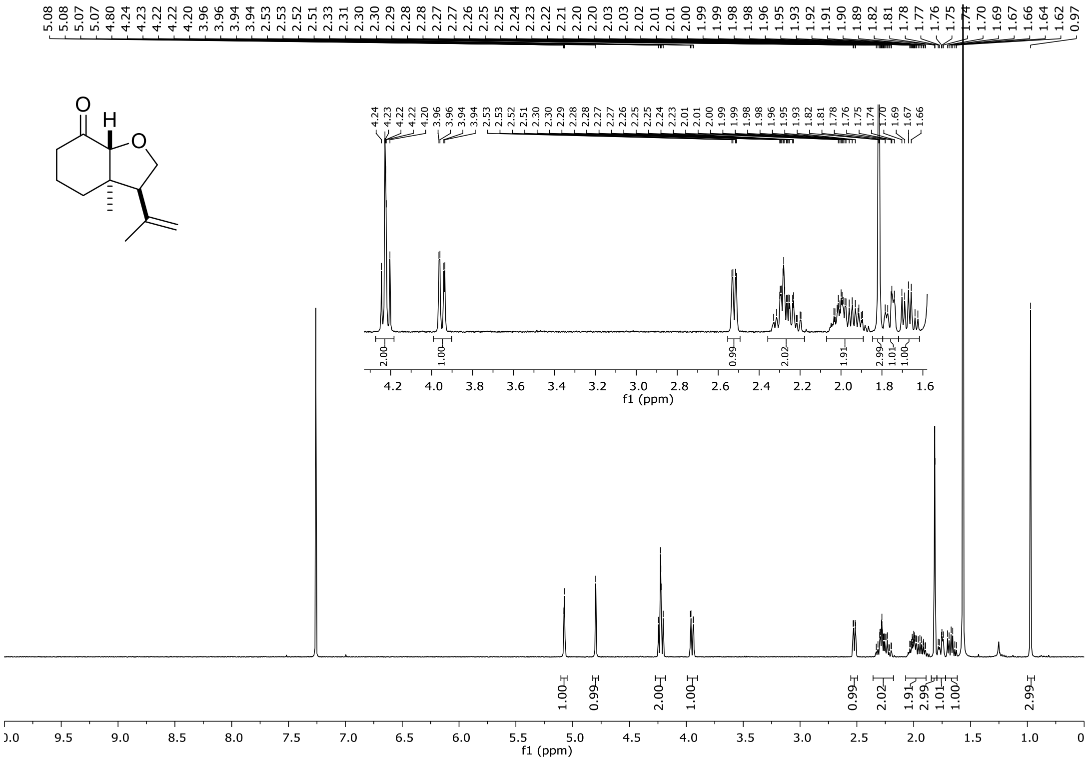


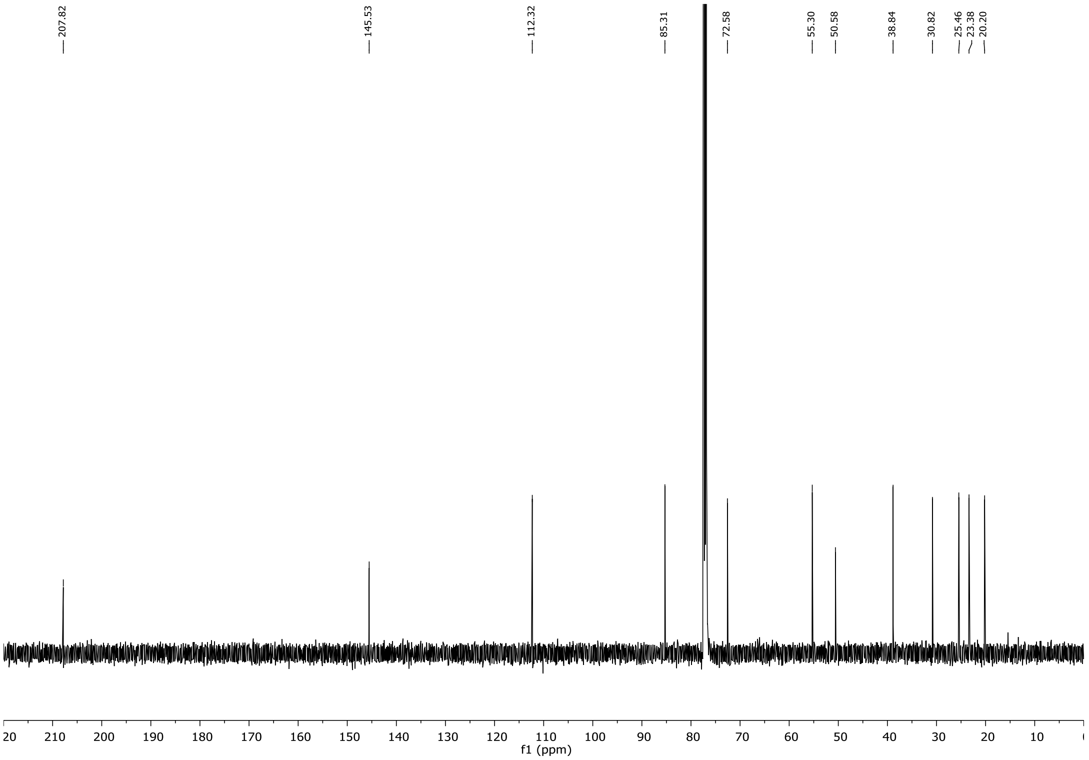
^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (75 MHz) spectra of compound **16a** ($CDCl_3$)



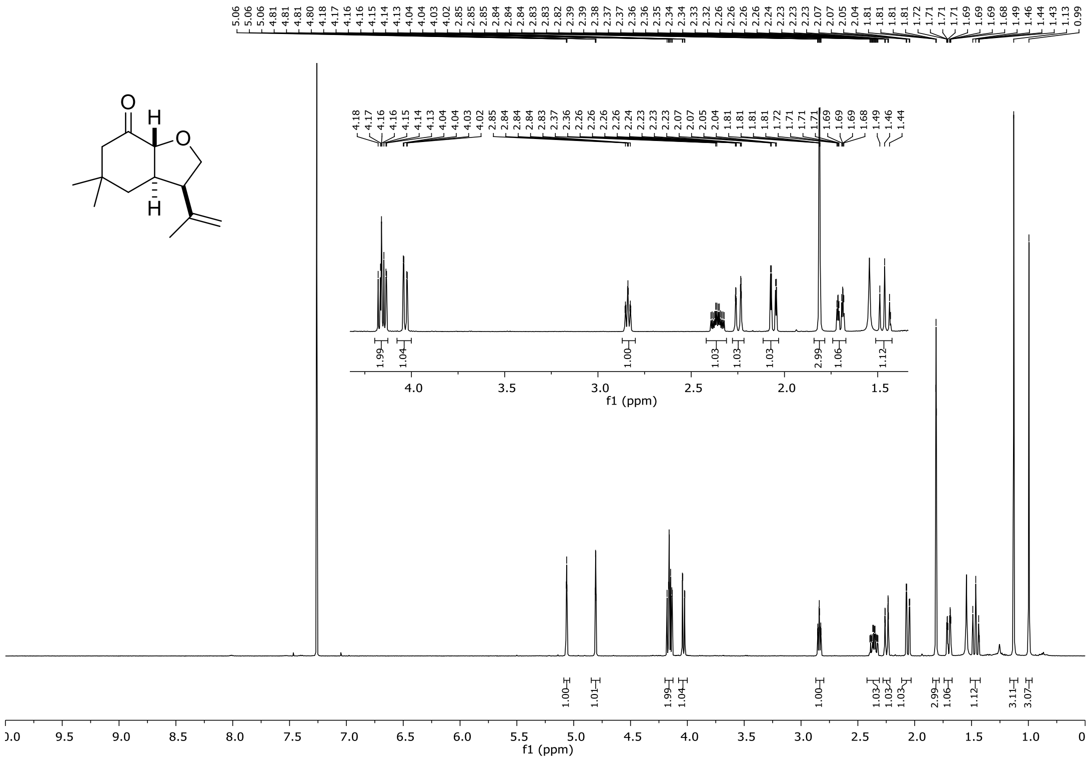


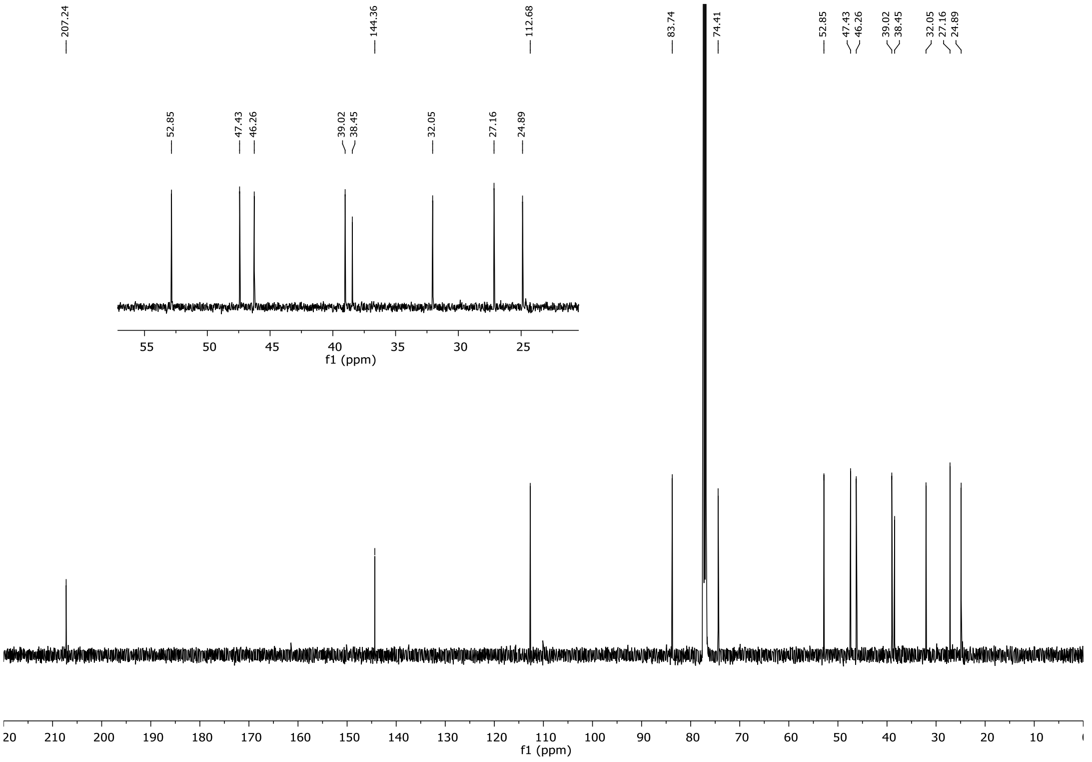
^{1}H (400 MHz) and $^{13}C\{^{1}H\}$ NMR (126 MHz) spectra of compound **16b** ($CDCl_3$)



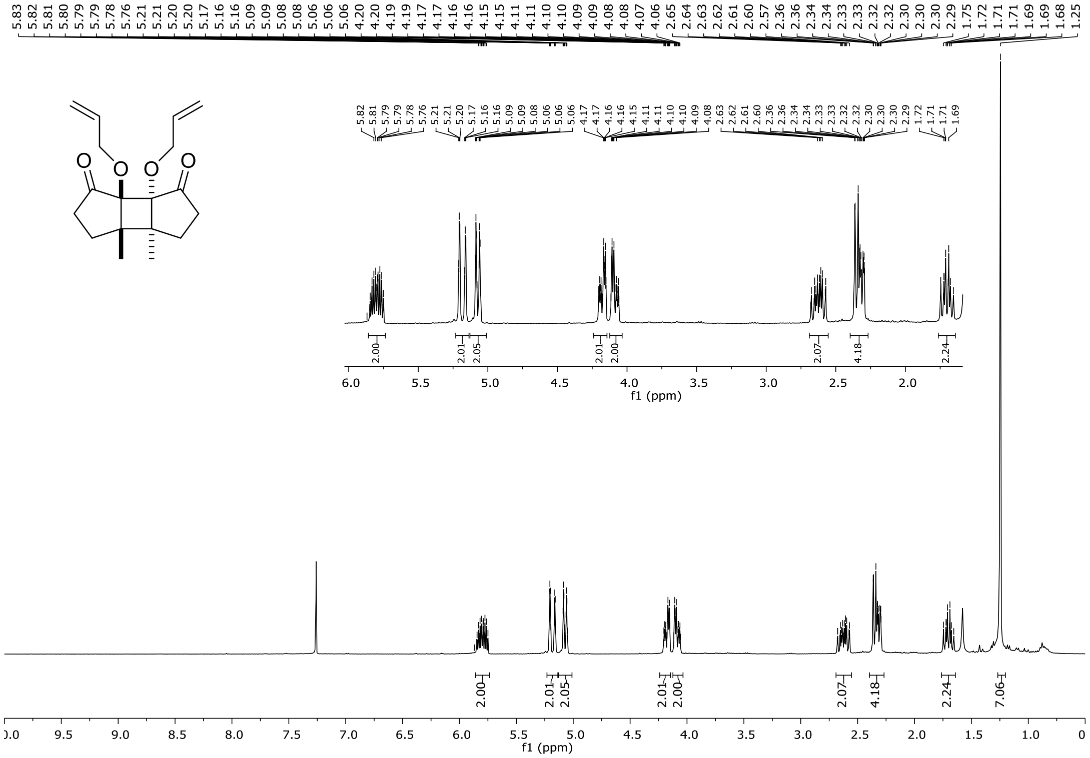


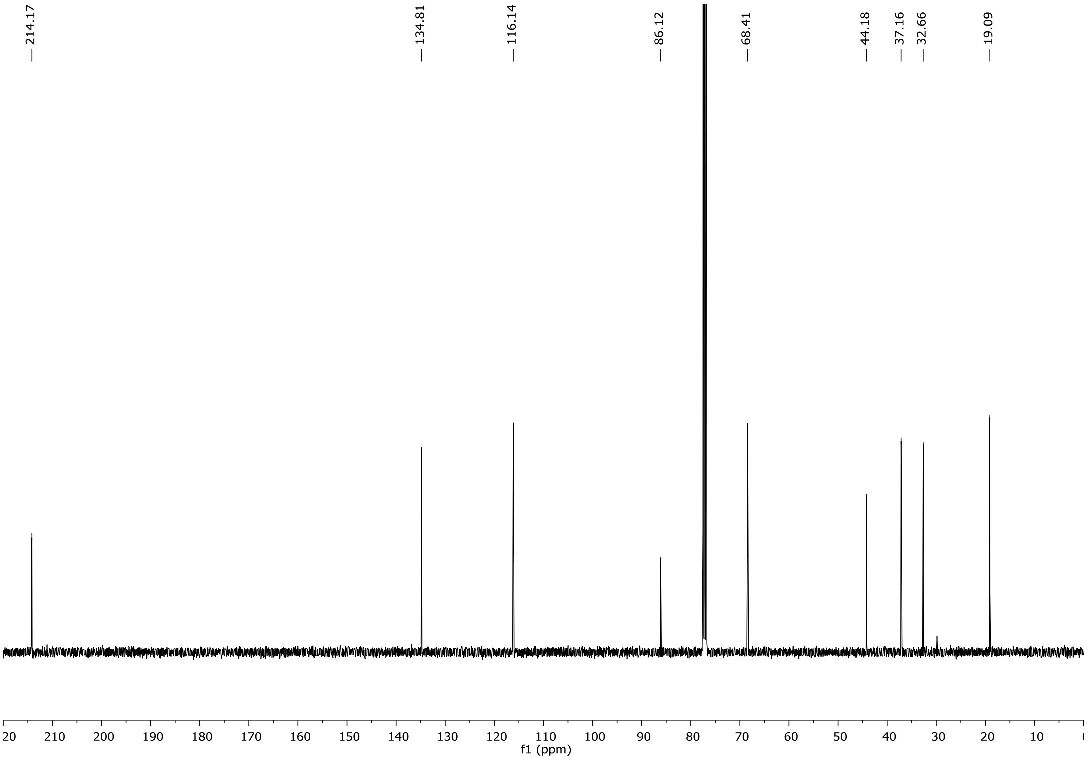
^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ NMR (126 MHz) spectra of compound **16c** (CDCl$_3$)



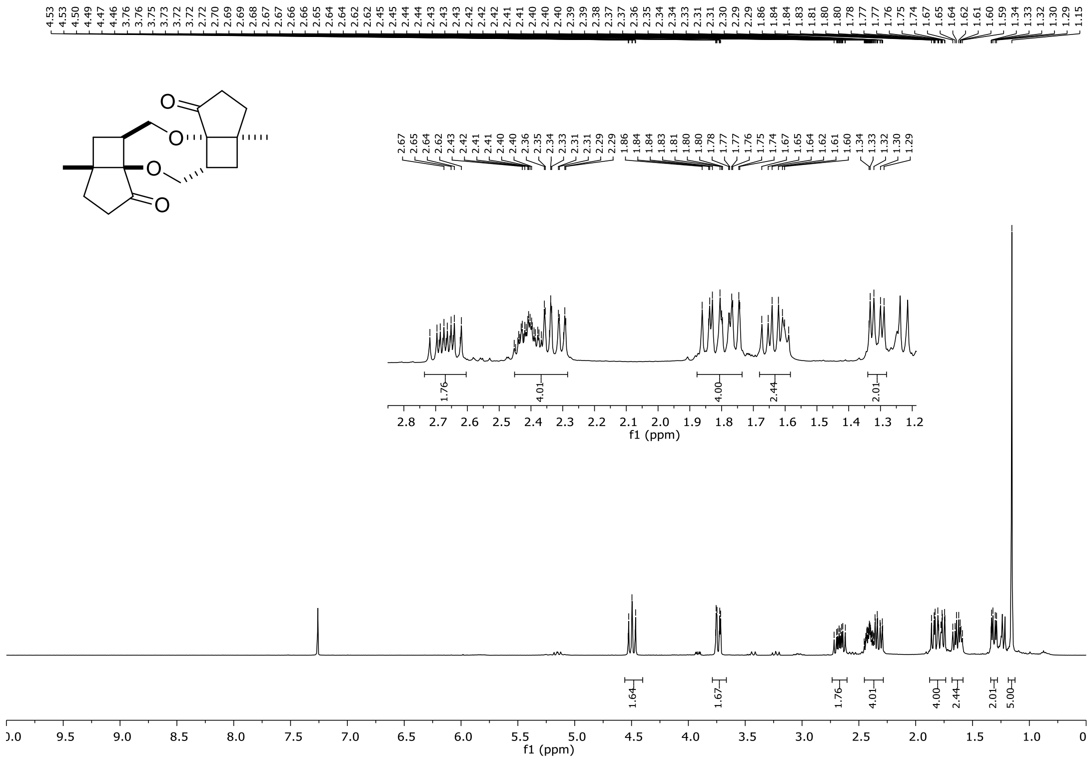


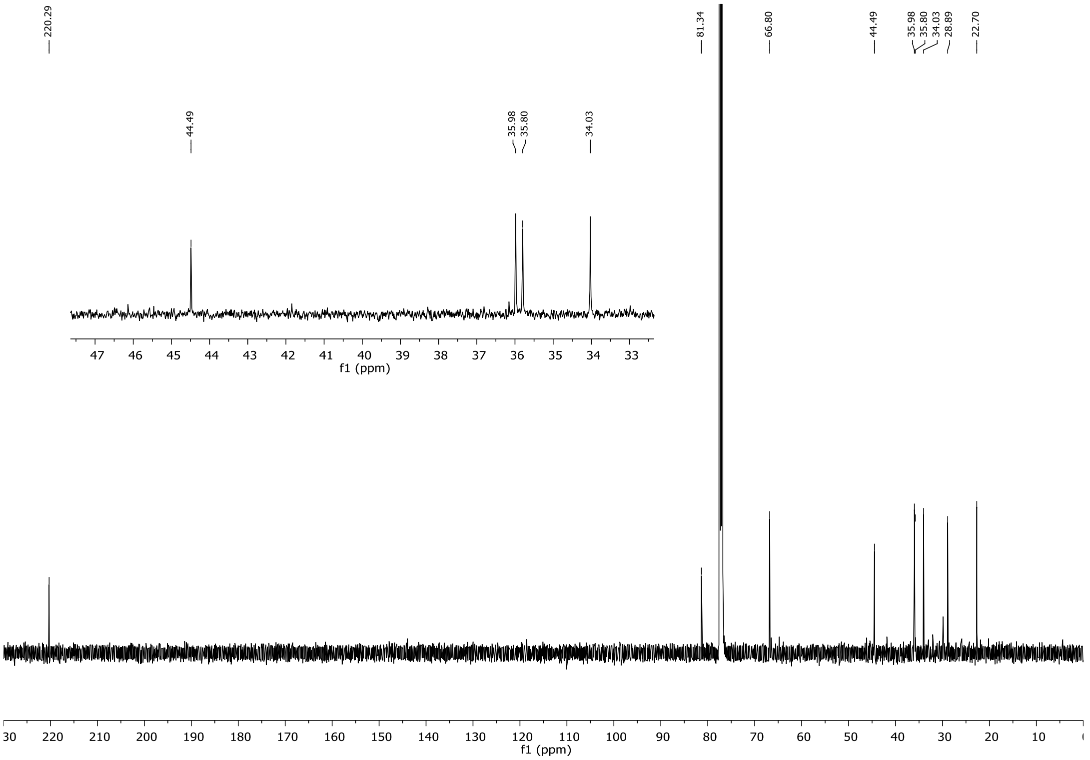
^{1}H (400 MHz) and ^{13}C{^{1}H} NMR (101 MHz) spectra of compound **17** ($CDCl_3$)



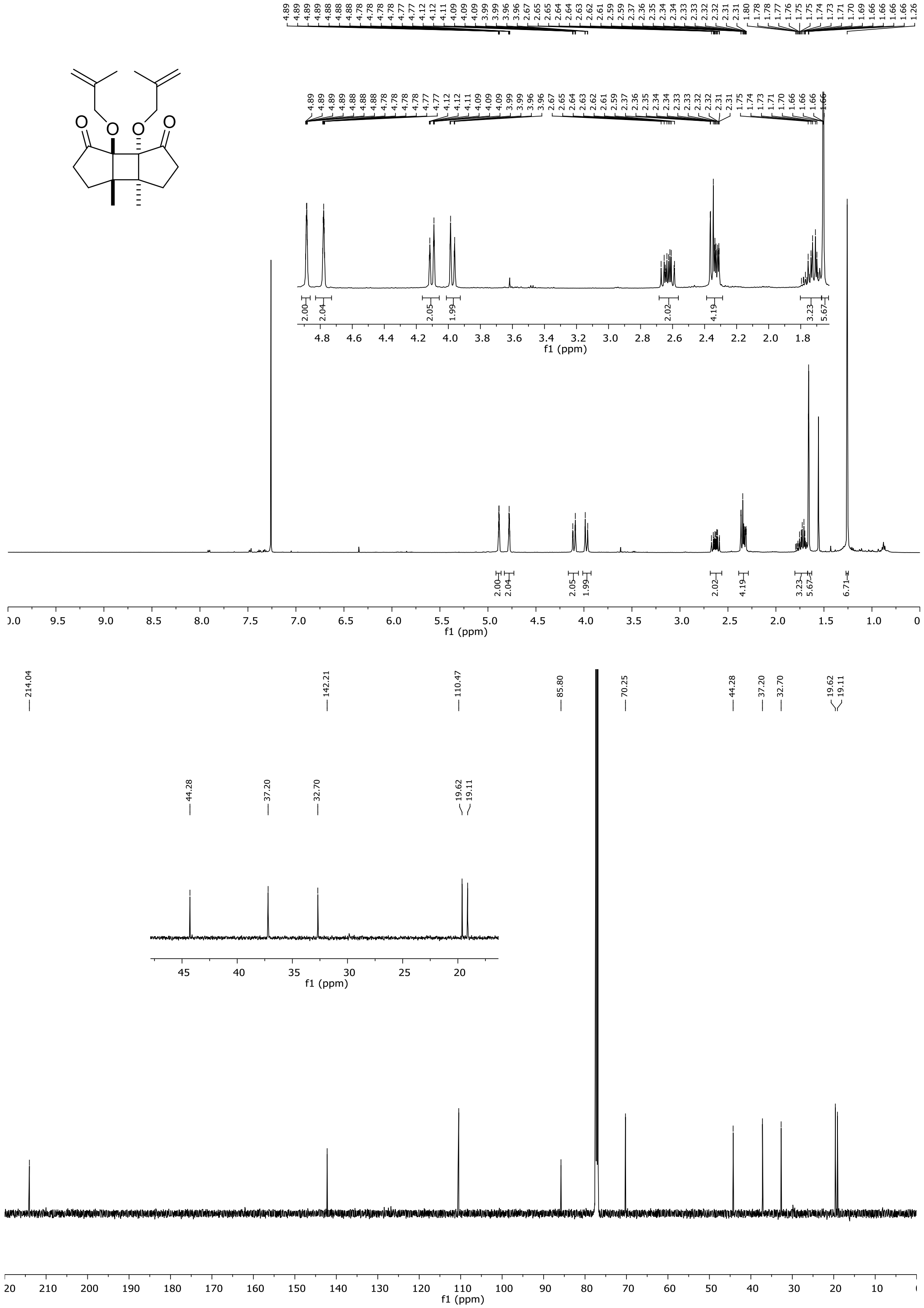


^{1}H (400 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **18** (CDCl$_3$)

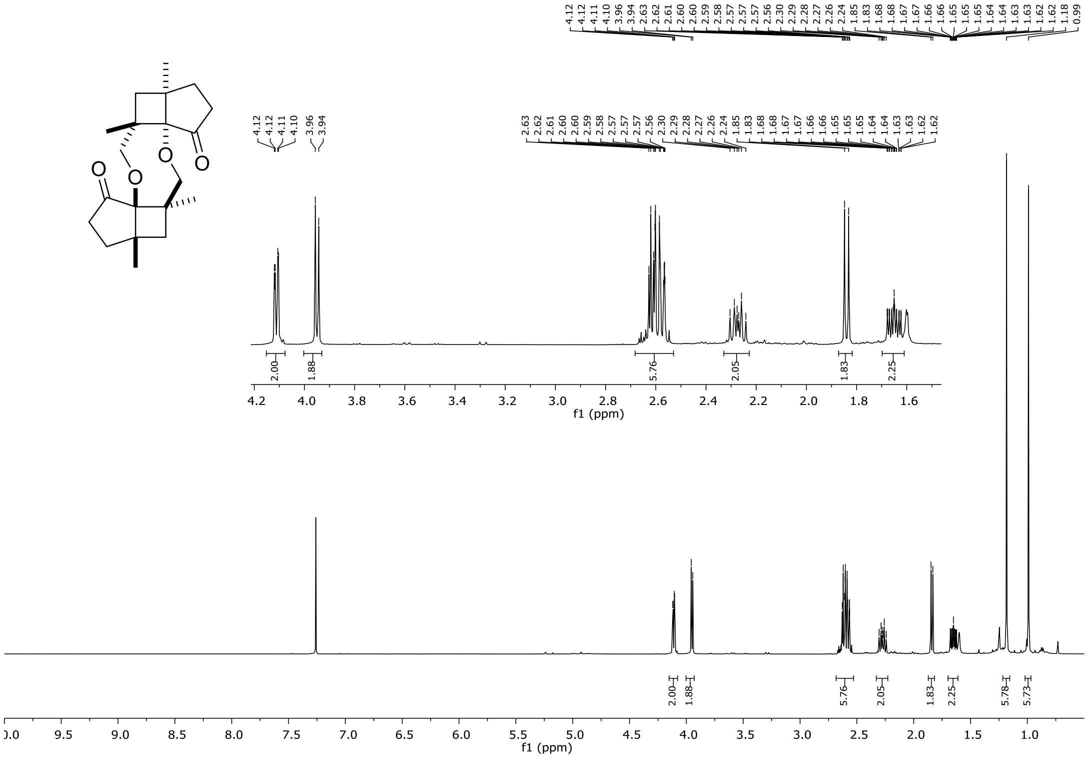


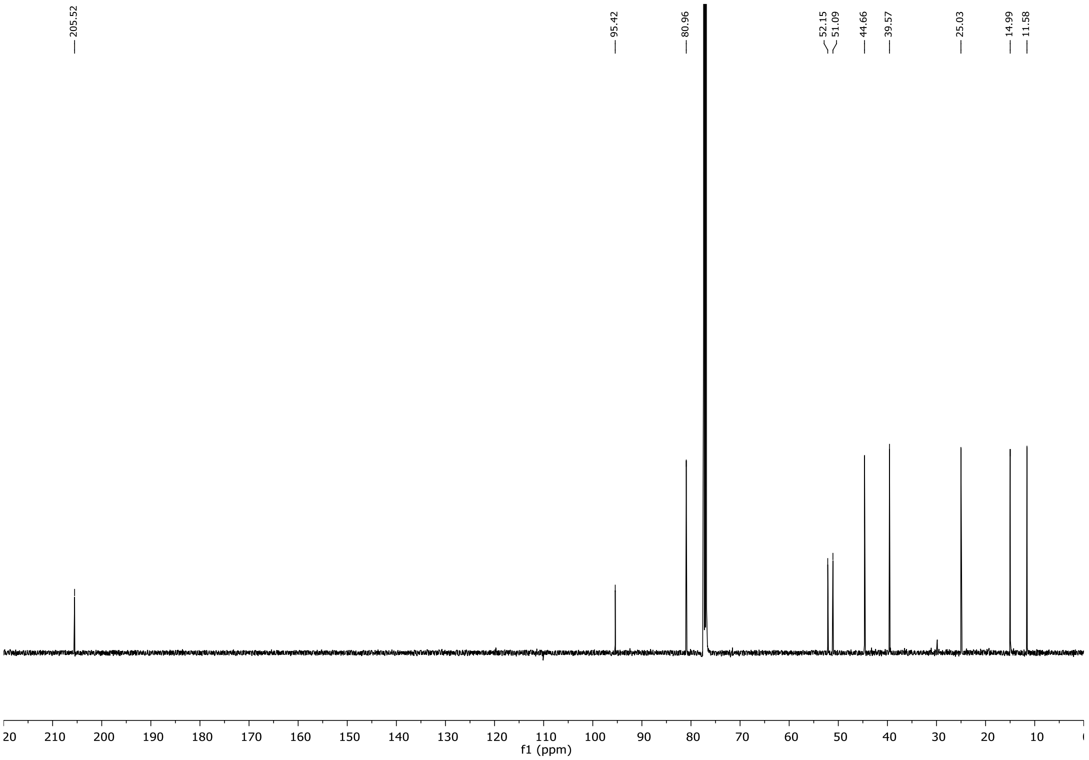


^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ NMR (126 MHz) spectra of compound **19** ($CDCl_3$)

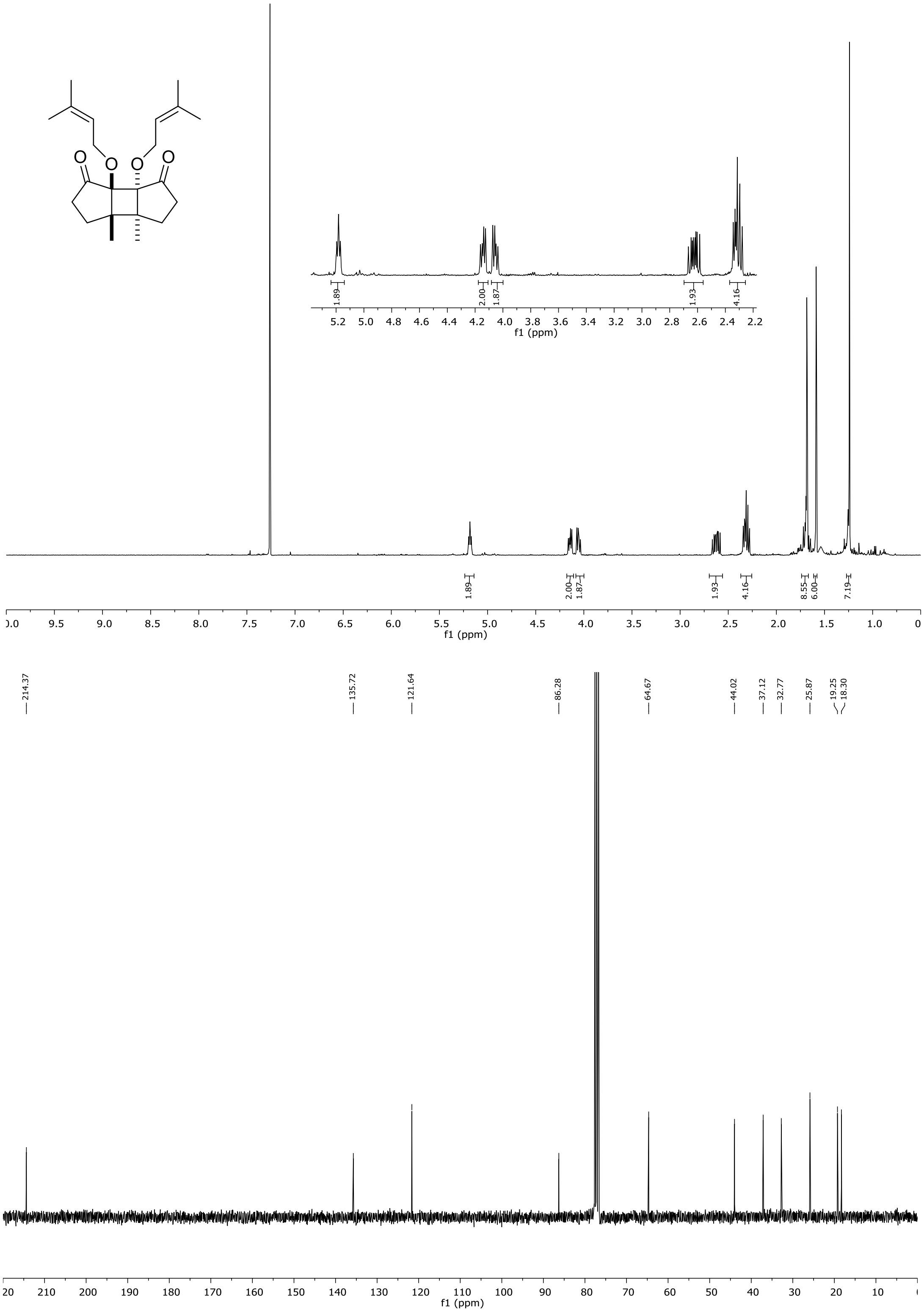


^{1}H (500 MHz) and ^{13}C{^{1}H} NMR (126 MHz) spectra of compound **20** ($CDCl_3$)



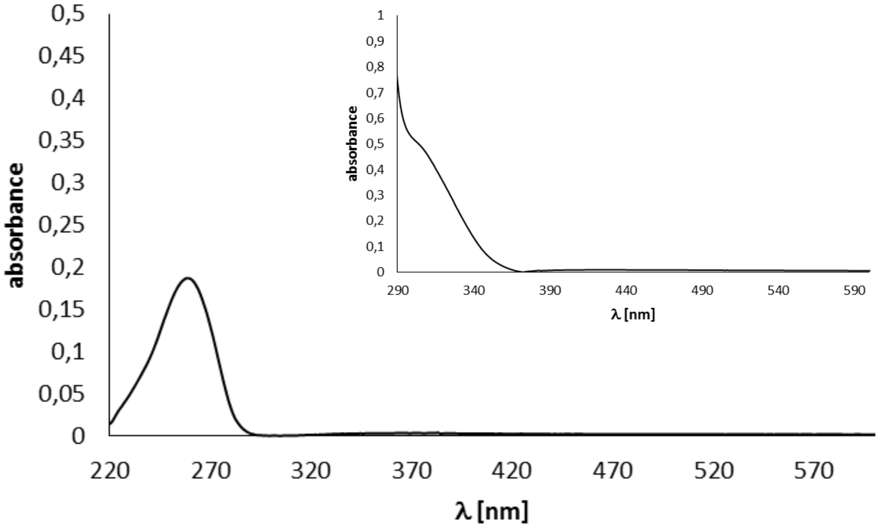


^{1}H (500 MHz) and $^{13}C\{^{1}H\}$ NMR (75 MHz) spectra of compound **21** ($CDCl_3$)

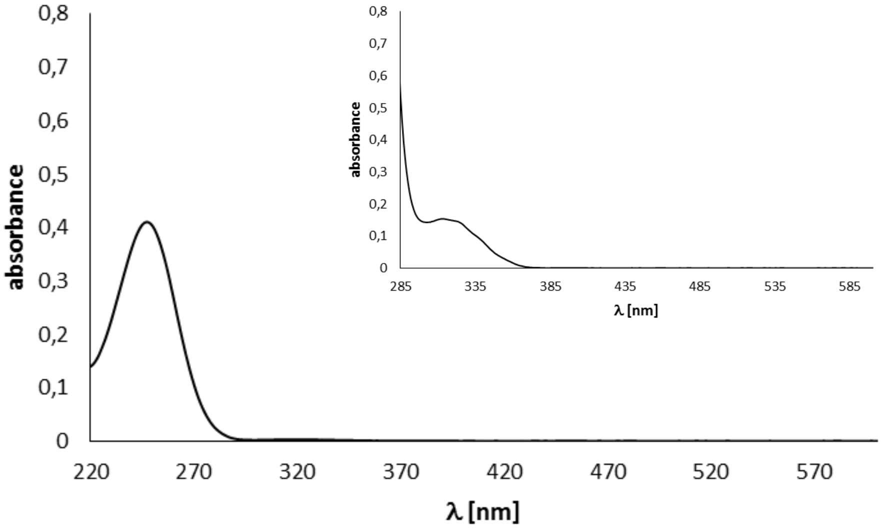


7. UV/vis Spectra of All Irradiation Precursors

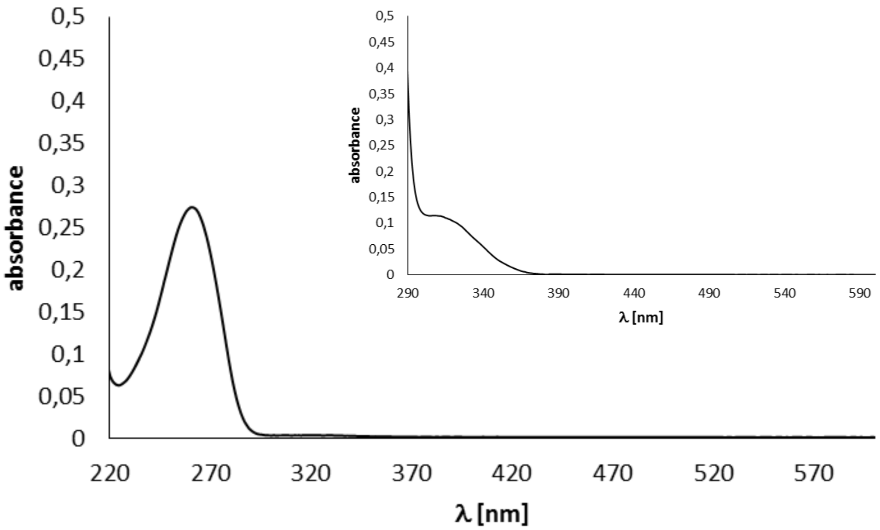
UV/vis spectrum of compound **1a** (0.5 mᴍ, CH_2Cl_2; 50mᴍ, CH_2Cl_2)



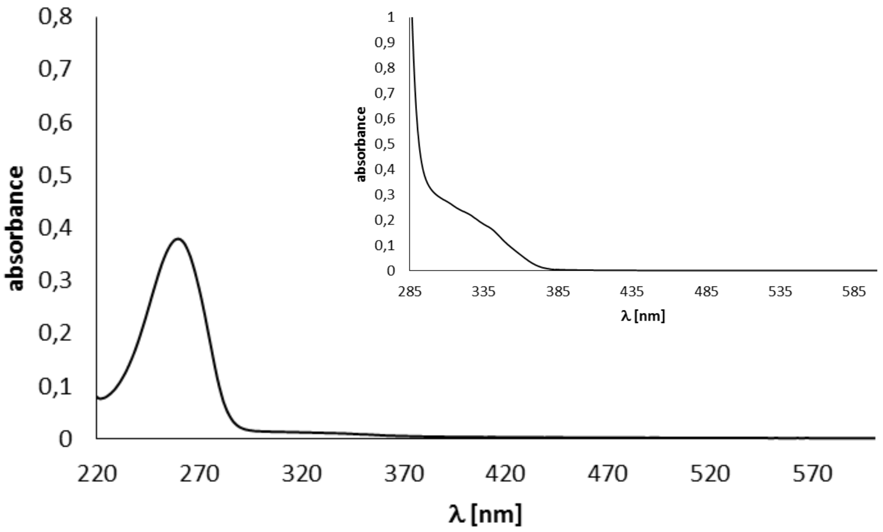
UV/vis spectrum of compound **1b** (0.5 mᴍ, CH_2Cl_2; 20mᴍ, CH_2Cl_2)



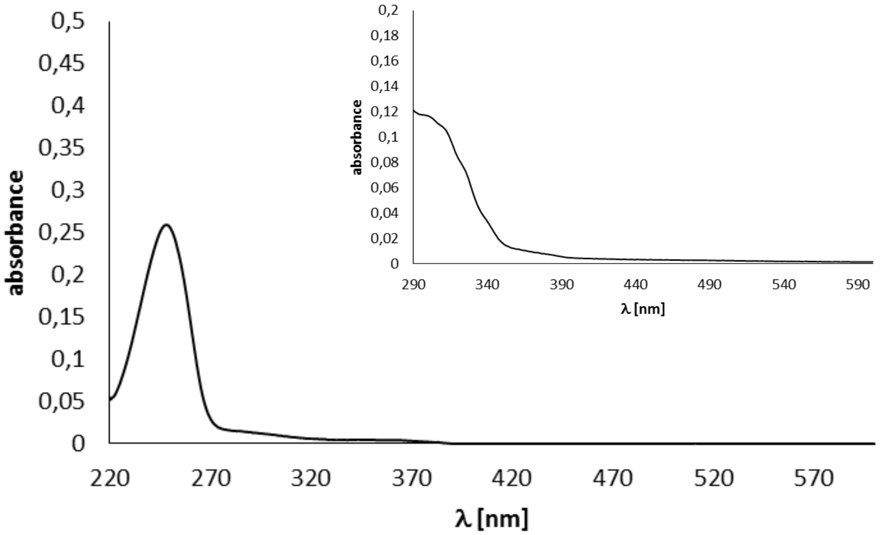
UV/vis spectrum of compound **1c** (0.5 mM, CH_2Cl_2; 20mM, CH_2Cl_2)



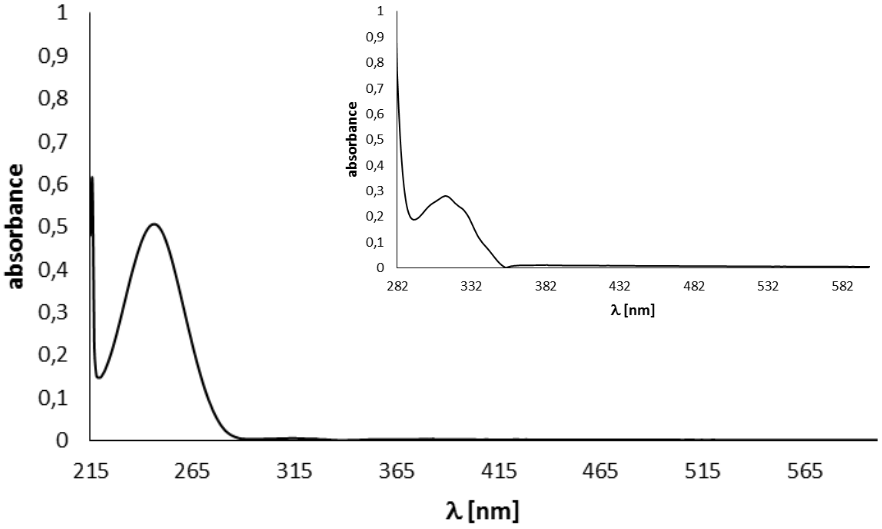
UV/vis spectrum of compound **1d** (0.5 mM, CH_2Cl_2; 20mM, CH_2Cl_2)



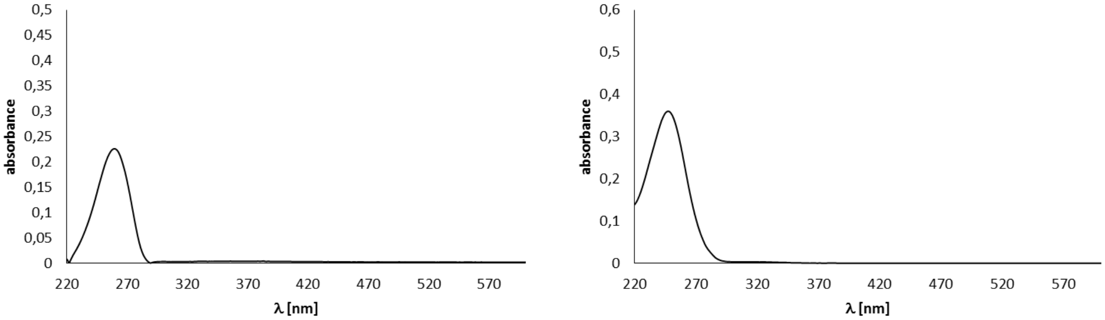
UV/vis spectrum of compound **1e** (0.5 mᴍ, CH_2Cl_2; 20mᴍ, CH_2Cl_2)



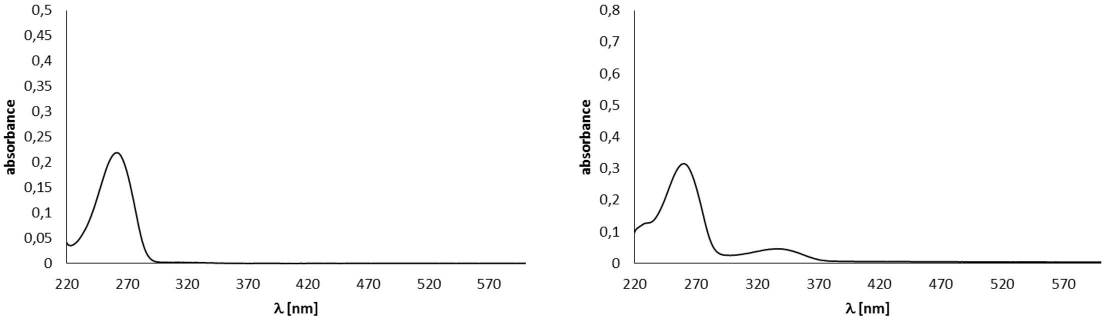
UV/vis spectrum of compound **1f** (0.5 mᴍ, CH_2Cl_2; 20mᴍ, CH_2Cl_2)



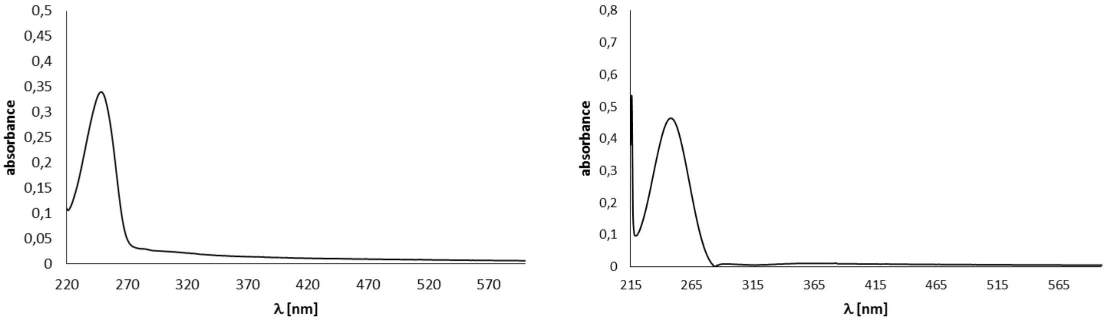
UV/vis spectrum of compound **2a** (left) and **2b** (right) (0.5 mM, CH_2Cl_2)



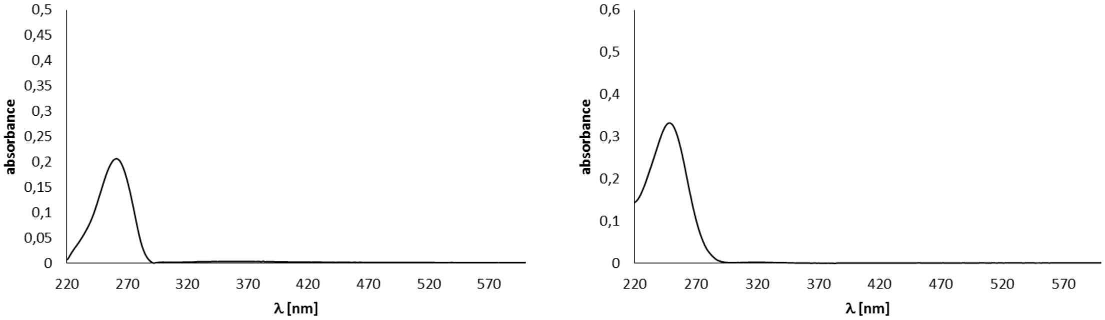
UV/vis spectrum of compound **2c** (left) and **2d** (right) (0.5 mM, CH_2Cl_2)



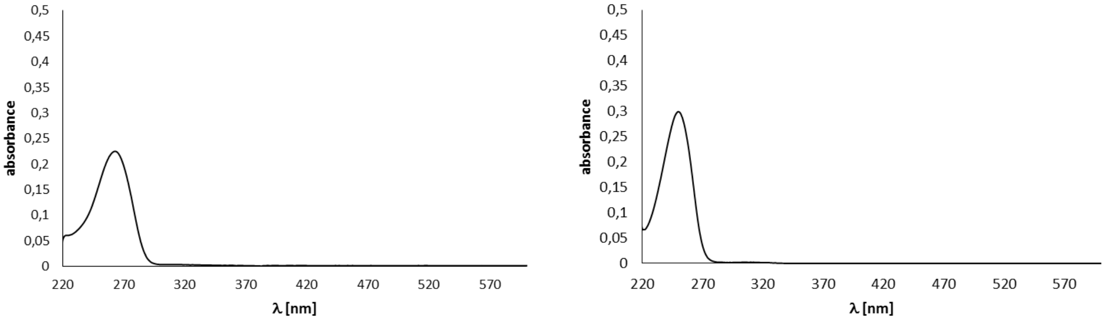
UV/vis spectrum of compound **2e** (left) and **2f** (right) (0.5 mM, CH_2Cl_2)



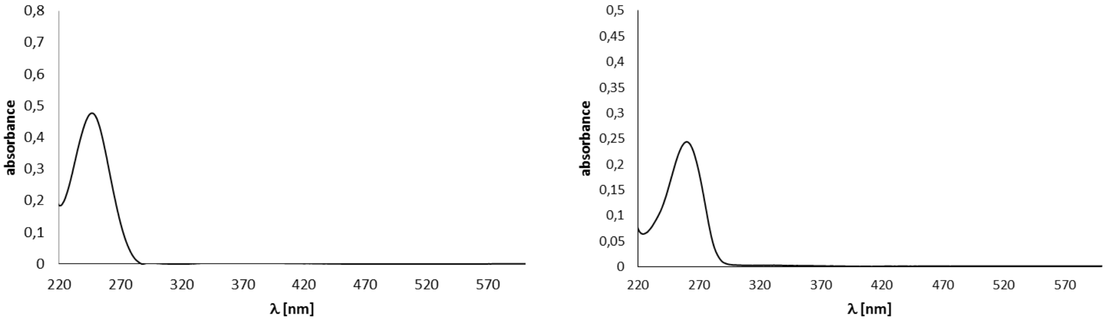
UV/vis spectrum of compound **3a** (left) and **3b** (right) (0.5 mᴍ, CH_2Cl_2)



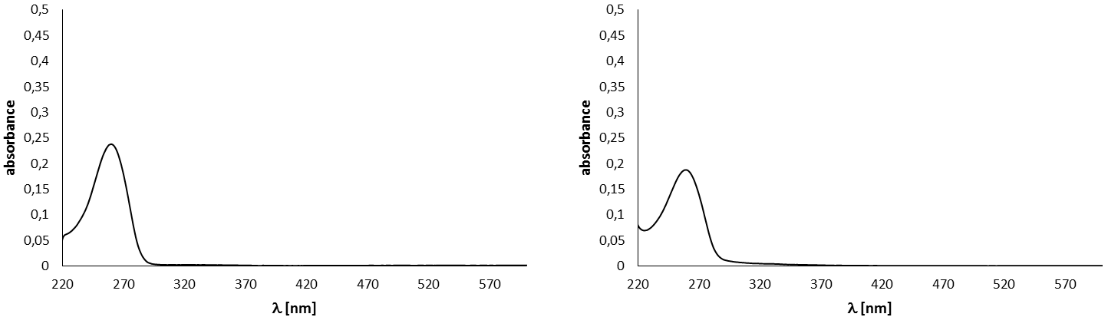
UV/vis spectrum of compound **3c** (left) and **3e** (right) (0.5 mᴍ, CH_2Cl_2)



UV/vis spectrum of compound **3f** (left) and *E*-**10**/*Z*-**10** (right) (0.5 mᴍ, CH_2Cl_2)



UV/vis spectrum of compound *E*-**12** (left) and *Z*-**12** (right) (0.5 mM, CH_2Cl_2)



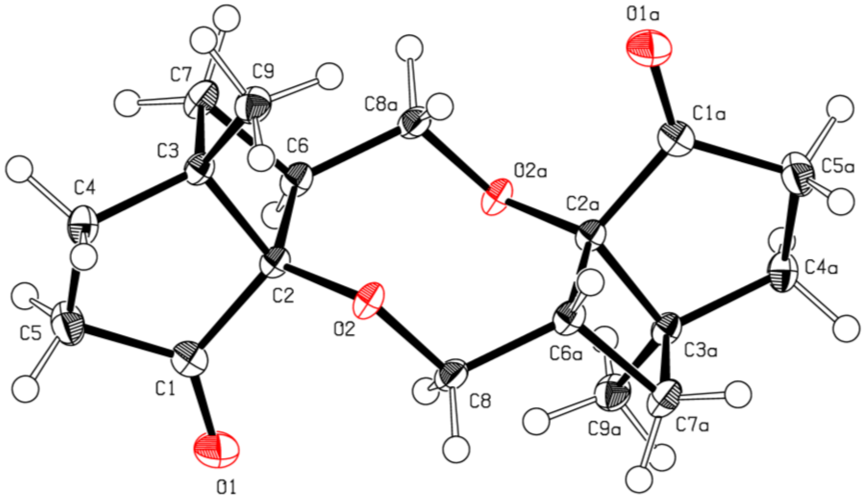
8. X-ray Crystallographic Details

Data were collected on Bruker D8 Venture single crystal x-ray diffractometer equipped with a CPAD detector (Bruker Photon II) (compounds **18**, **S1**) or a CMOS detector (Bruker Photon-100) (compound **21**), an IMS micro source (compounds **18**, **S1**) or a TXS rotating anode (compound **21**) with MoK$_\alpha$ radiation (λ = 0.71073 Å) and a Helios optic using the APEX3 software package.[6] Measurements were performed on single crystals coated with perfluorinated ether. The crystals were fixed on top of a kapton micro sampler and frozen under a stream of cold nitrogen. A matrix scan was used to determine the initial lattice parameters. Reflections were corrected for Lorentz and polarisation effects, scan speed, and background using SAINT.[7] Absorption correction, including odd and even ordered spherical harmonics was performed using SADABS.[7] Space group assignments were based upon systematic absences, E statistics, and successful refinement of the structures. The structures were solved using SHELXT with the aid of successive difference Fourier maps, and were refined against all data using SHELXL in conjunction with SHELXLE.[8,9,10] Hydrogen atoms were calculated in ideal positions as follows: Methyl hydrogen atoms were refined as part of rigid rotating groups, with a C–H distance of 0.98 Å and $U_{iso(H)} = 1.5 \cdot U_{eq(C)}$. Other H atoms were placed in calculated positions and refined using a riding model, with methylene and aromatic C–H distances of 0.99 Å and 0.95 Å, respectively, other C–H distances of 1.00 Å, all with $U_{iso(H)} = 1.2 \cdot U_{eq(C)}$. Non-hydrogen atoms were refined with anisotropic displacement parameters. Full-matrix least-squares refinements were carried out by minimizing $\Sigma w(F_o^2 - F_c^2)^2$ with the SHELXL weighting scheme.[8] Neutral atom scattering factors for all atoms and anomalous dispersion corrections for the non-hydrogen atoms were taken from *International Tables for Crystallography*.[11] Images of the crystal structures were generated with Mercury (main article) and PLATON (SI).[12,13] CCDC 2010591-2010593 contains the supplementary crystallographic data for this paper. These data are provided free of charge by The Cambridge Crystallographic Data Centre.

Compound 18 (CCDC 2010593)

Single crystals were obtained by solvent evaporation from a saturated solution of **18** in Et_2O.

Ellipsoids in the ORTEP structure are displayed at the 50% probability level.



Diffractometer operator C. Jandl
scanspeed 1-10 s per frame
dx 37 mm
3415 frames measured in 13 data sets
phi-scans with delta_phi = 0.5
omega-scans with delta_omega = 0.5
shutterless mode

Crystal data

$C_{18}H_{24}O_4$

M_r = 304.37

Monoclinic, *C*2/*c*

Hall symbol: -C 2yc

a = 18.1075 (17) Å

b = 11.052 (1) Å

c = 8.2387 (8) Å

β = 111.686 (3)°

D_x = 1.320 Mg m^{-3}

Melting point: > 493 K

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 9504 reflections

θ = 2.2–28.3°

μ = 0.09 mm^{-1}

T = 100 K

V = 1532.1 (3) Å^3

Z = 4

F(000) = 656

Fragment, colourless

0.26 × 0.16 × 0.13 mm

Data collection

Bruker D8 Venture diffractometer

Radiation source: IMS microsource

Helios optic monochromator

Detector resolution: 7.5 pixels mm^{-1}

phi– and ω–rotation scans

Absorption correction: multi-scan *SADABS* 2016/2, Bruker

$T_{\min}$ = 0.725, $T_{\max}$ = 0.746

49708 measured reflections

1827 independent reflections

1720 reflections with $I > 2\sigma(I)$

R_{int} = 0.029

$\theta_{\max}$ = 27.9°, $\theta_{\min}$ = 2.2°

$h = -23 \quad 23$

$k = -14 \quad 14$

$l = -10 \quad 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.034

$wR(F^2)$ = 0.093

S = 1.09

1827 reflections

101 parameters

0 restraints

0 constraints

Primary atom site location: intrinsic phasing

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

W = $1/[\Sigma^2(F\text{O}^2) + (0.0421P)^2 + 1.3923P]$
WHERE $P = (F\text{O}^2 + 2F\text{C}^2)/3$

$(\Delta/\sigma)_{\max}$ < 0.001

$\Delta\rho_{\max}$ = 0.39 e Å^{-3}

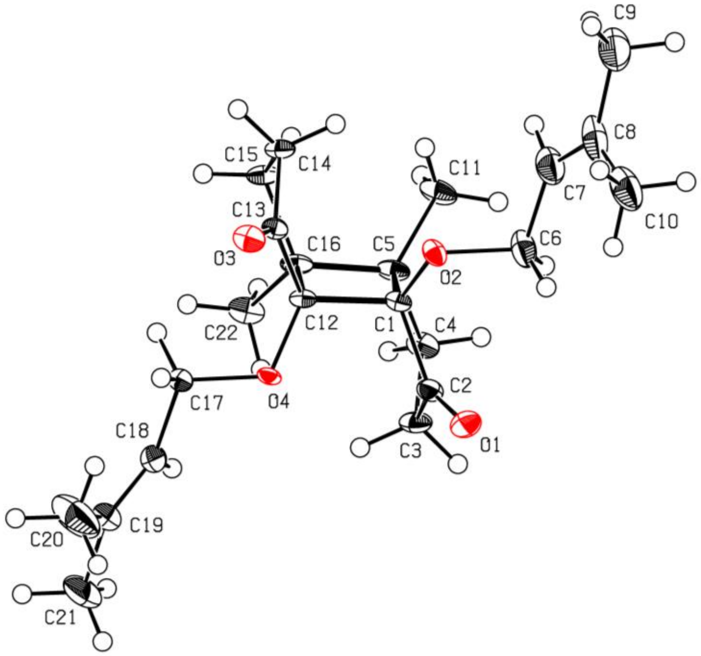
$\Delta\rho_{\min}$ = −0.21 e Å^{-3}

Extinction correction: none

Extinction coefficient: -

Compound 21 (CCDC 2010591)

Single crystals were obtained by diffusion of pentane into a saturated solution of **21** in CH_2Cl_2. Ellipsoids in the ORTEP structure are displayed at the 50% probability level.



Diffractometer operator C. Jandl
scanspeed 10-60 s per frame
dx 50 mm
1488 frames measured in 6 data sets
phi-scans with delta_phi = 0.5
omega-scans with delta_omega = 0.5
shutterless mode

Crystal data

$C_{22}H_{32}O_4$

M_r = 360.47

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

a = 11.8018 (14) Å

b = 6.7440 (8) Å

c = 25.405 (3) Å

β = 100.493 (4)°

V = 1988.2 (4) Å^3

D_x = 1.204 Mg m^{-3}

Melting point: 358–371 K

Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 8914 reflections

θ = 3.1–25.5°

μ = 0.08 mm^{-1}

T = 100 K

Fragment, colourless

$Z = 4$

$F(000) = 784$

0.15 × 0.12 × 0.04 mm

Data collection

Bruker D8 Venture diffractometer

Radiation source: TXS rotating anode

Helios optic monochromator

Detector resolution: 7.5 pixels mm^{-1}

phi– and ω–rotation scans

Absorption correction: multi-scan *SADABS* 2016/2, Bruker

$T_{\min} = 0.647$, $T_{\max} = 0.745$

28402 measured reflections

3647 independent reflections

2817 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.066$

$\theta_{\max} = 25.4°$, $\theta_{\min} = 2.7°$

$h = -14$ 14

$k = -8$ 7

$l = -28$ 30

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.065$

$wR(F^2) = 0.188$

$S = 1.10$

3647 reflections

489 parameters

602 restraints

0 constraints

Primary atom site location: intrinsic phasing

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$W = 1/[\Sigma^2(FO^2) + (0.0982P)^2 + 1.8965P]$ WHERE $P = (FO^2 + 2FC^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.47$ e Å^{-3}

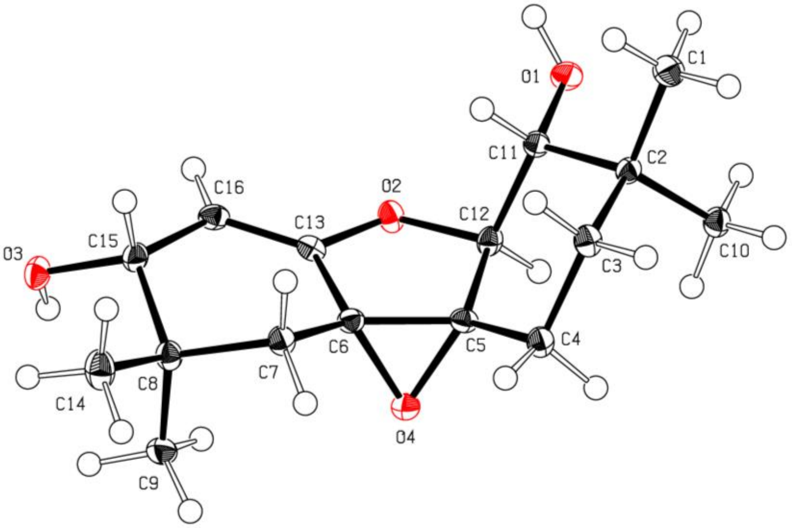
$\Delta\rho_{\min} = -0.46$ e Å^{-3}

Extinction correction: none

Extinction coefficient: -

Compound S1 (CCDC 2010592)

Single crystals were obtained by solvent evaporation from a saturated solution of **S1** in CH_2Cl_2. Ellipsoids in the ORTEP structure are displayed at the 50% probability level.



Diffractometer operator T. Pickl & A. Poethig
phi-scans with delta_phi = 0.5
omega-scans with delta_omega = 0.5
shutterless mode

Crystal data

$C_{16}H_{24}O_4$

M_r = 280.35

D_x = 1.291 Mg m^{-3}

Monoclinic, *P*2$_1$/*n*

Hall symbol: -P 2yn

Mo *K*α radiation, λ = 0.71073 Å

a = 6.1182 (3) Å

Cell parameters from 6447 reflections

b = 10.6025 (5) Å

θ = 2.7–26.4°

c = 22.3764 (12) Å

μ = 0.09 mm^{-1}

β = 96.280 (3)°

T = 100 K

V = 1442.81 (13) Å^3

Needle, clear colourless

Z = 4

0.74 × 0.09 × 0.06 mm

$F(000) = 608$

Data collection

Bruker D8 Venture diffractometer

Radiation source: IMS microsource

HELIOS optic monochromator

Detector resolution: 7.5 pixels mm^{-1}

phi– and ω–rotation scans

Absorption correction: multi-scan *SADABS* 2016/2, Bruker

$T_{\min} = 0.655$, $T_{\max} = 0.745$

28565 measured reflections

2960 independent reflections

2598 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.065$

$\theta_{\max} = 26.4°$, $\theta_{\min} = 2.1°$

$h = -7 \quad 7$

$k = -13 \quad 13$

$l = -27 \quad 27$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.053$

$wR(F^2) = 0.128$

$S = 1.14$

2960 reflections

193 parameters

0 restraints

0 constraints

Primary atom site location: intrinsic phasing

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

$W = 1/[\Sigma^2(F\text{O}^2) + (0.0351P)^2 + 1.9676P]$ WHERE $P = (F\text{O}^2 + 2F\text{C}^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.30$ e Å^{-3}

$\Delta\rho_{\min} = -0.24$ e Å^{-3}

Extinction correction: none

Extinction coefficient: -

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