

Heterogenisation of ketone catalysts within mesoporous supports for asymmetric epoxidation

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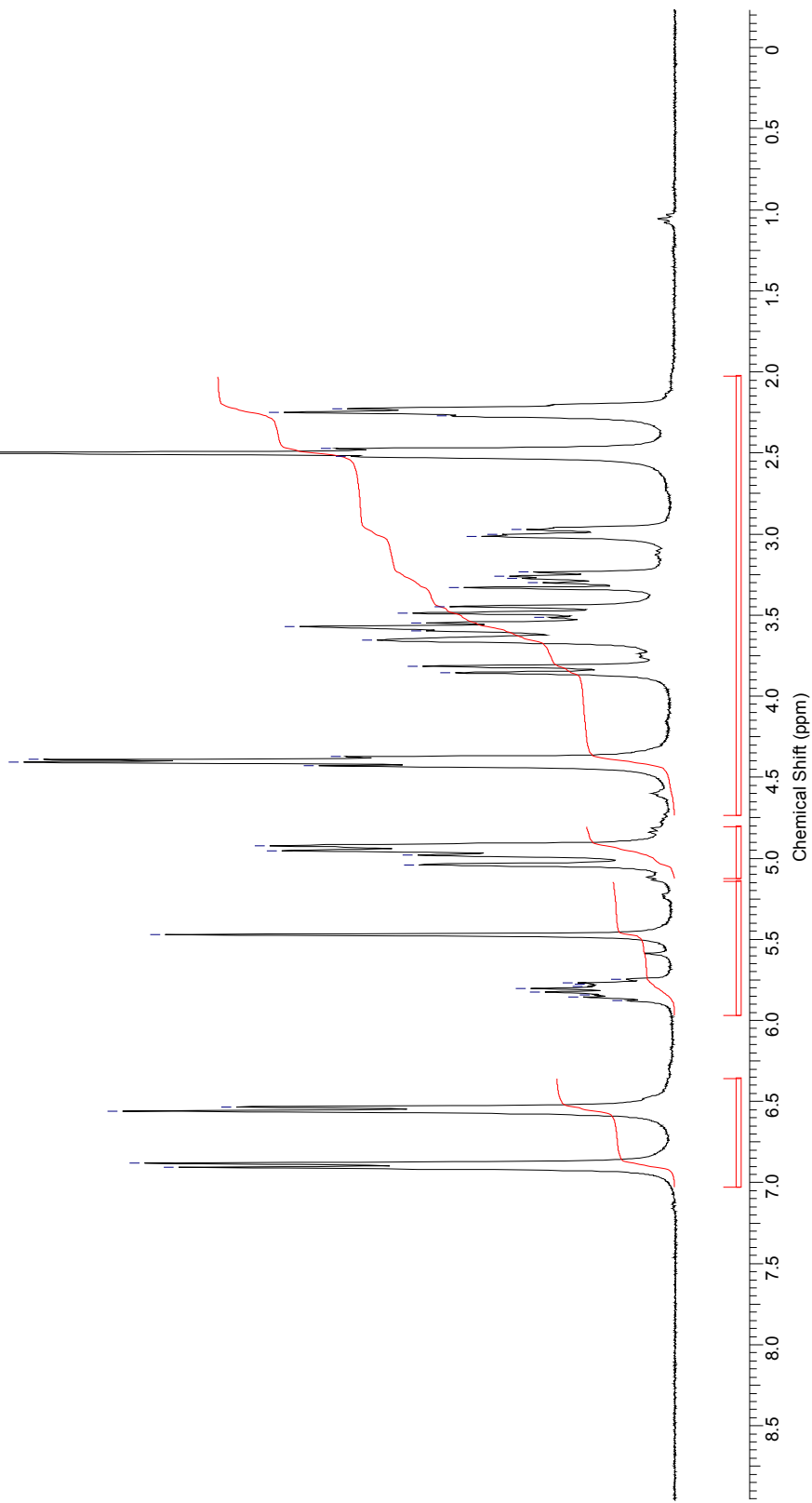
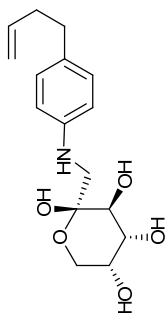
NMR spectroscopic data (catalyst synthesis)

¹H NMR for (2*R*,3*S*,4*R*,5*R*)-2-(((4-(But-3-en-1-yl)phenyl)amino)methyl) tetrahydro-2*H*-pyran-2,3,4,5-tetraol (**2**)

[300 MHz, DMSO]

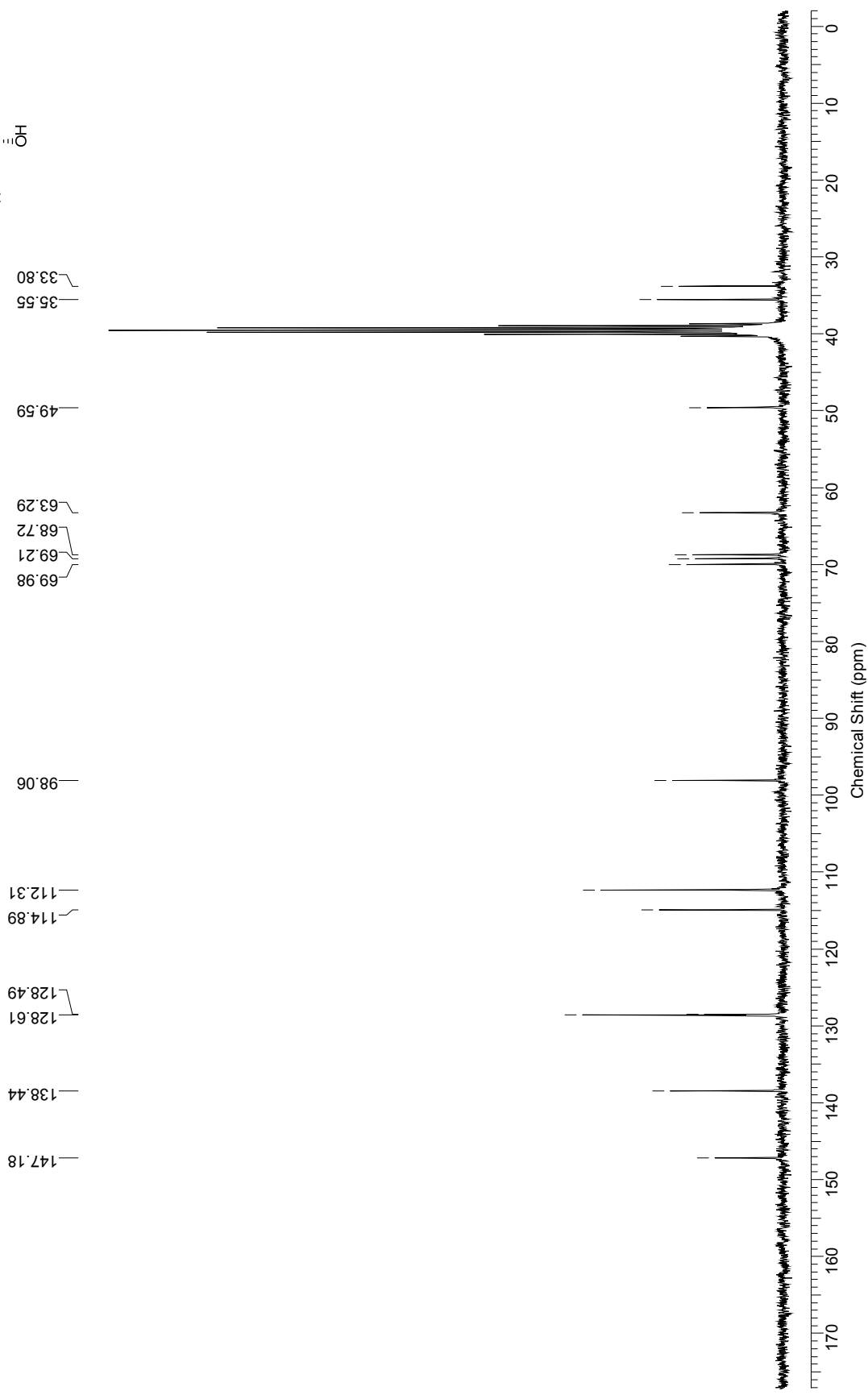
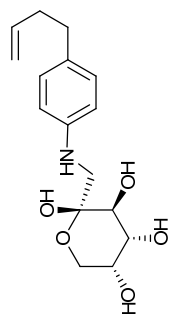
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6.91
6.88
6.56
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5.78
5.77
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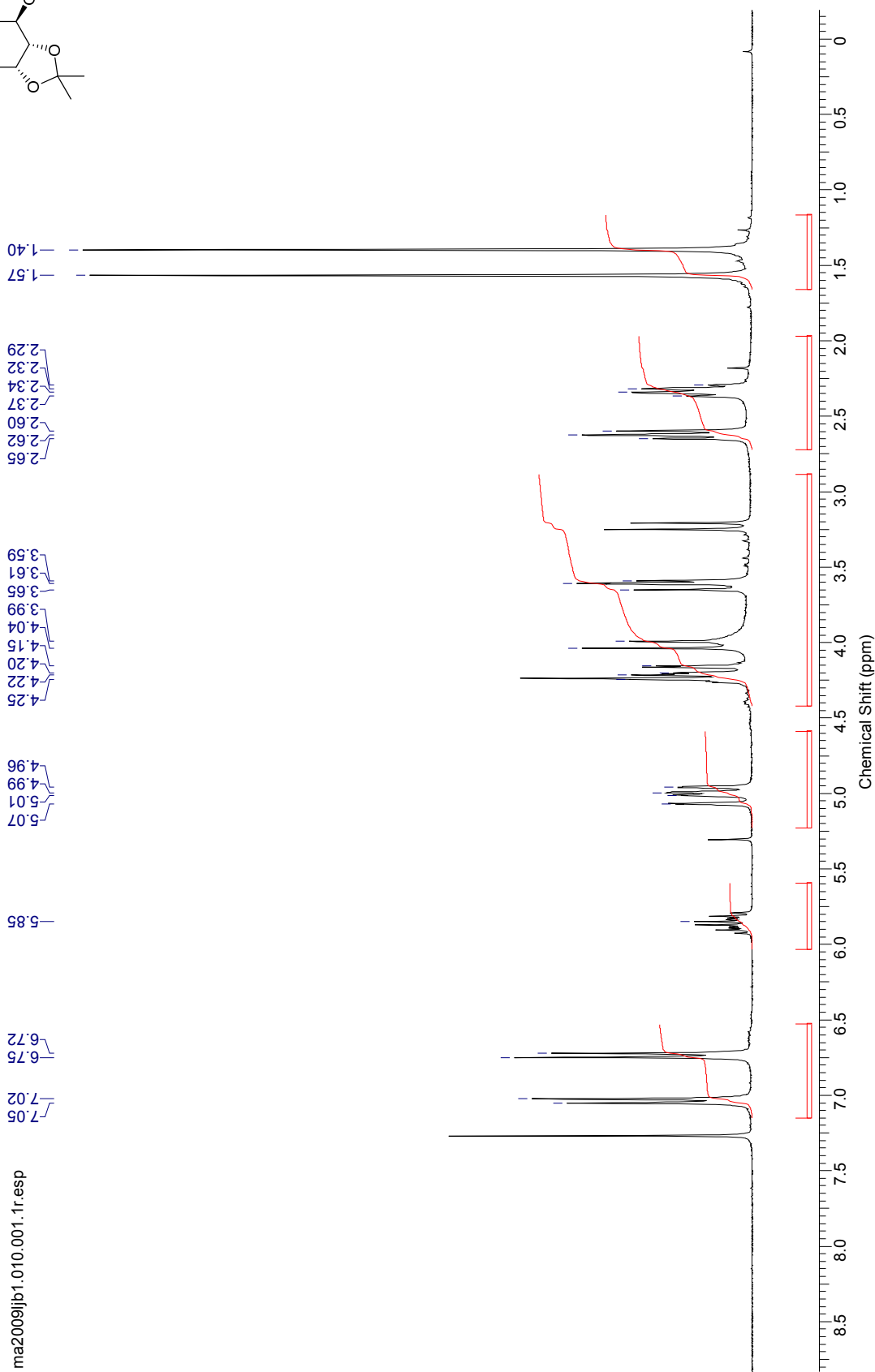
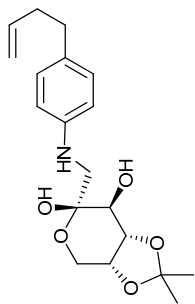
^{13}C NMR for (2*R*,3*S*,4*R*,5*R*)-2-(((4-(*But-3-en-1-yl*)phenyl)amino)methyl) tetrahydro-2*H*-pyran-2,3,4,5-tetraol (**2**).

[75 MHz, DMSO]



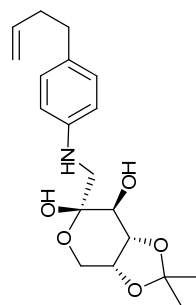
¹H NMR for (3aR,6R,7S,7aS)-6-(((4-(But-3-en-1-yl)phenyl)amino)methyl)-2,2-dimethyltetrahydro-3aH-[1,3]dioxolo[4,5-c]pyran-6,7-diol (**3**).

[300 MHz, CDCl₃]

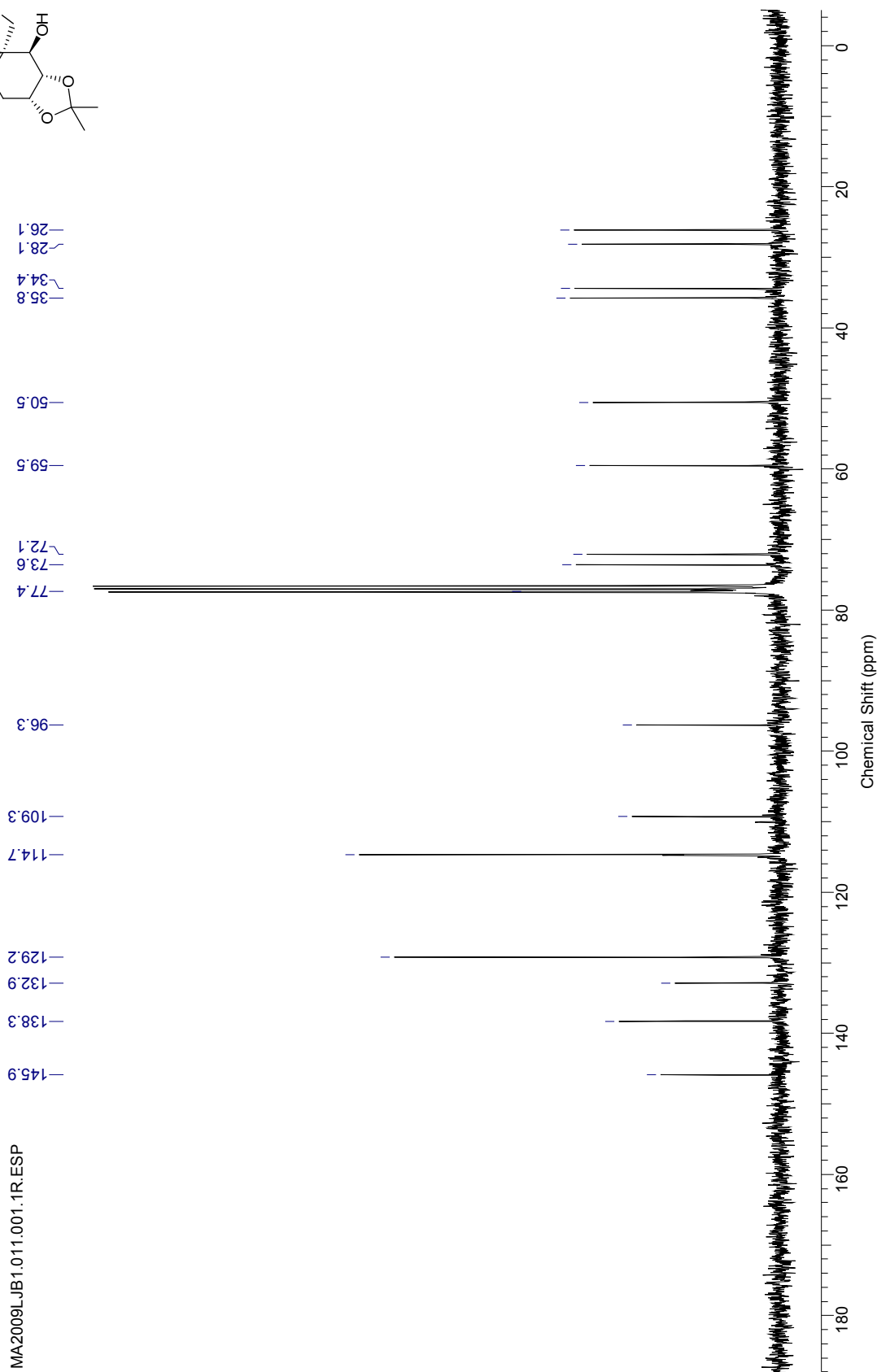


^{13}C NMR for (3*aR*,6*R*,7*S*,7*aS*)-6-(((4-(But-3-en-1-yl)phenyl)amino)methyl)-2,2-dimethyltetrahydro-3*aH*-[1,3]dioxolo[4,5-*c*]pyran-6,7-diol (**3**).

[75 MHz, CDCl_3]

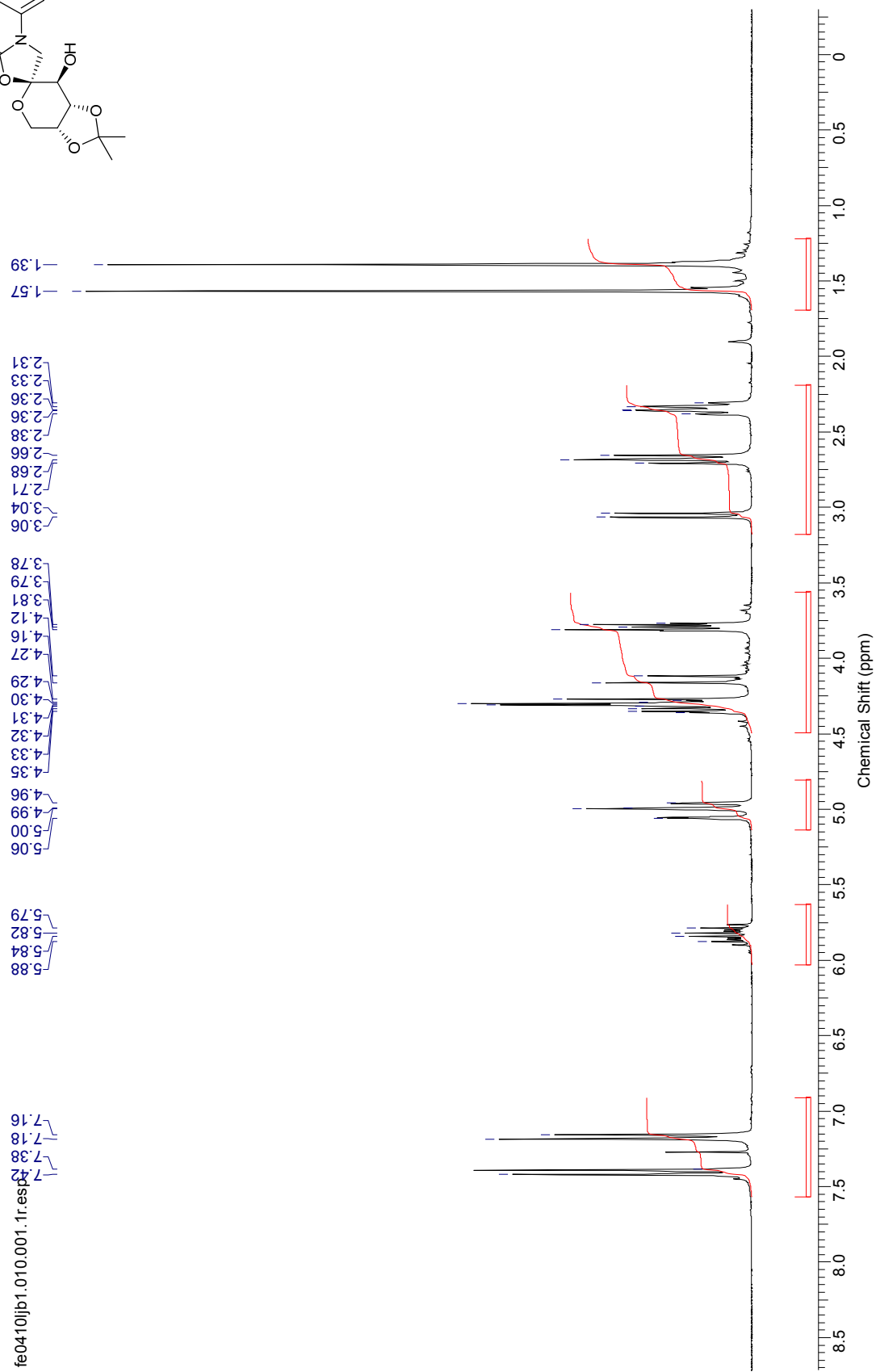
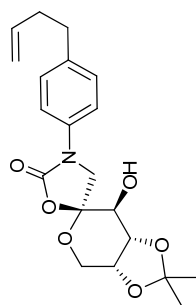


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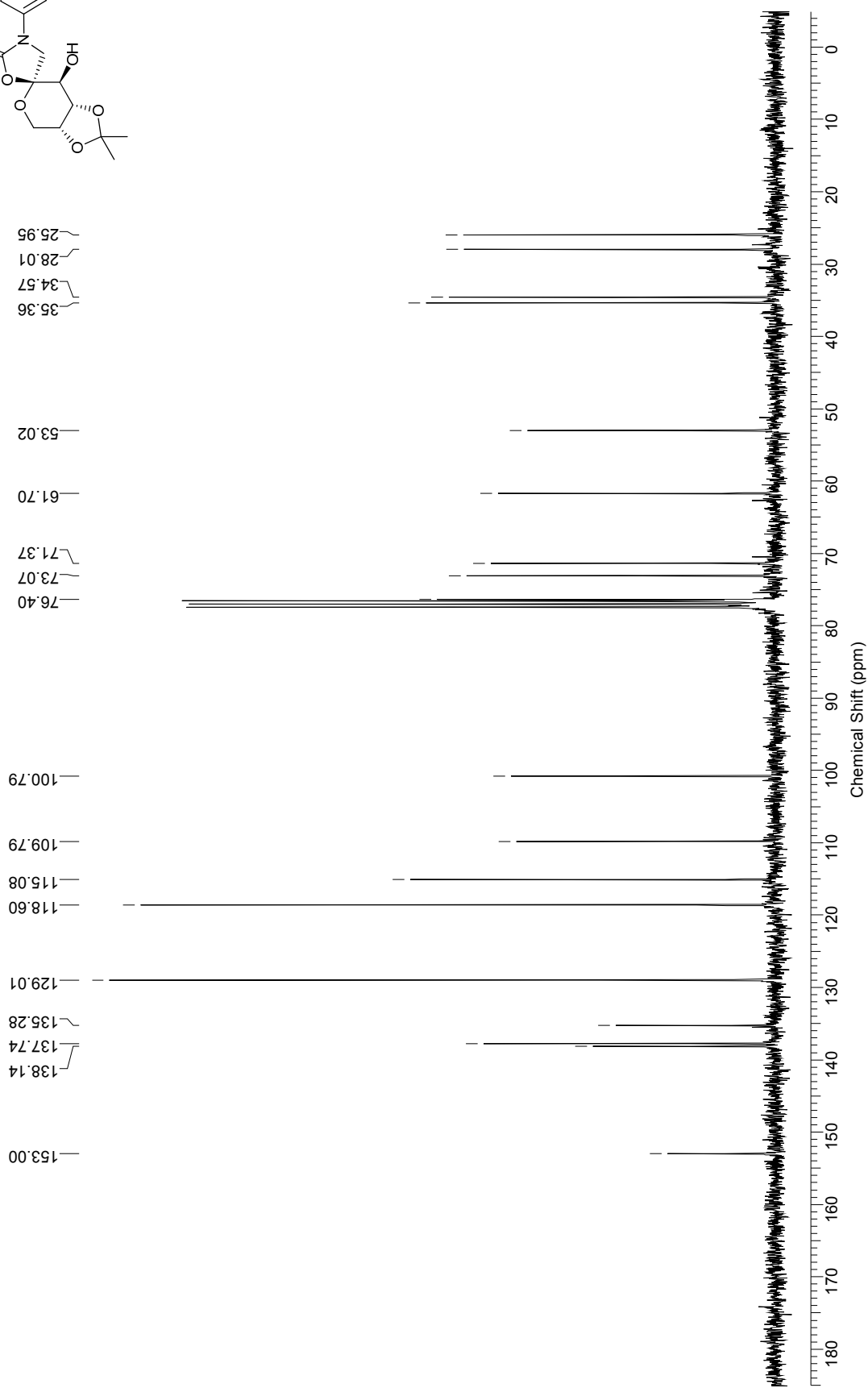
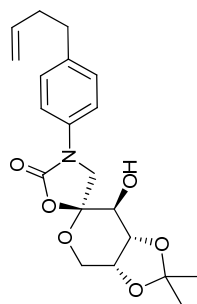
¹H NMR for (3aR,5'S,7S,7aS)-3'-(4-(But-3-en-1-yl)phenyl)-7-hydroxy-2,2-dimethyltetrahydrospiro[[1,3]dioxolo[4,5-c]pyran-6,5'-oxazolidin]-2'-one (**4**).

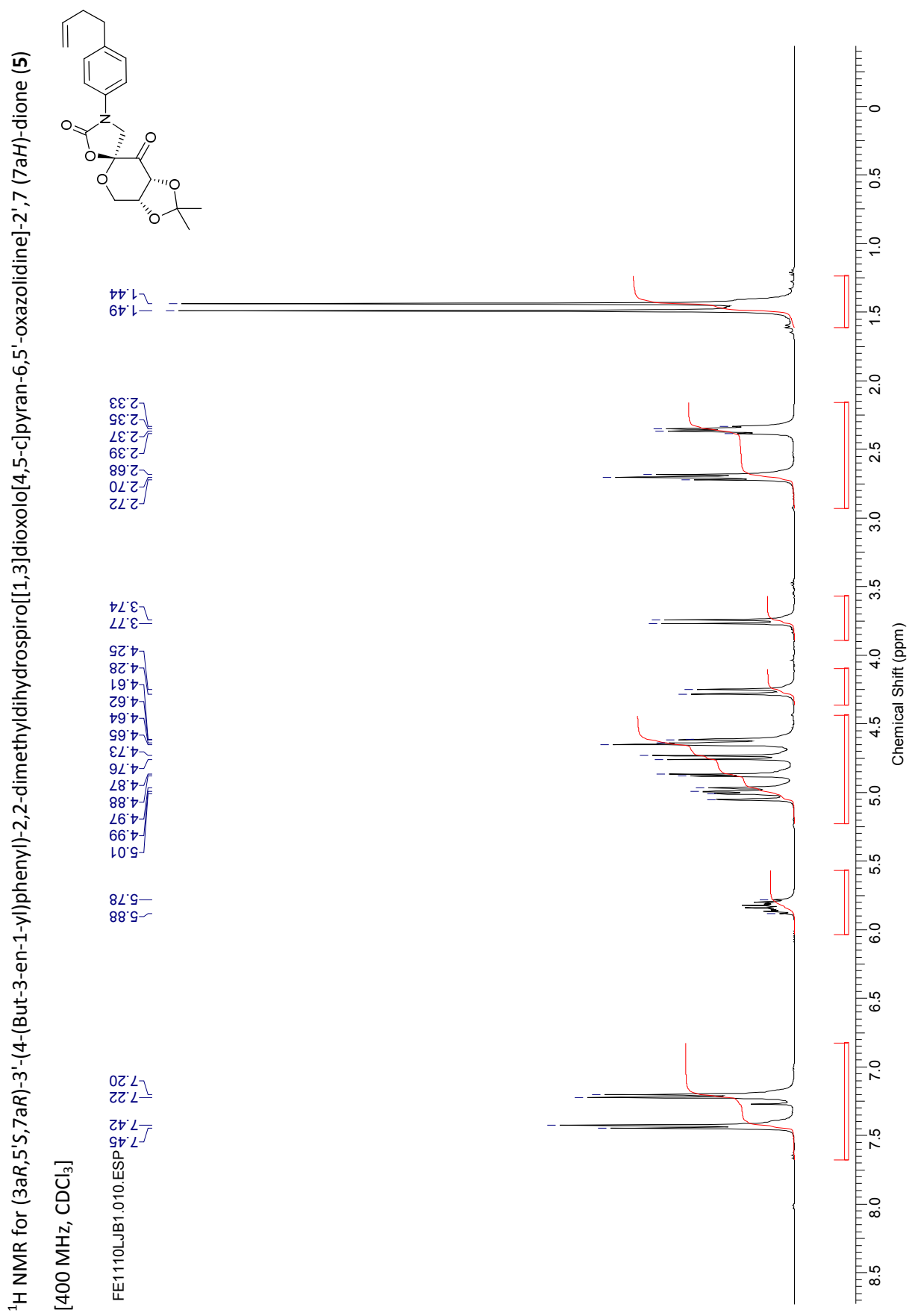
[300 MHz, CDCl₃]



^{13}C NMR for (3*R*,5*S*,7*S*,7*aS*)-3'-(4-(*n*-But-3-en-1-yl)phenyl)-7-hydroxy-2,2-dimethyltetrahydrospiro[[1,3]dioxolo[4,5-*c*]pyran-6,5'-oxazo lidin]-2'-one (**4**).

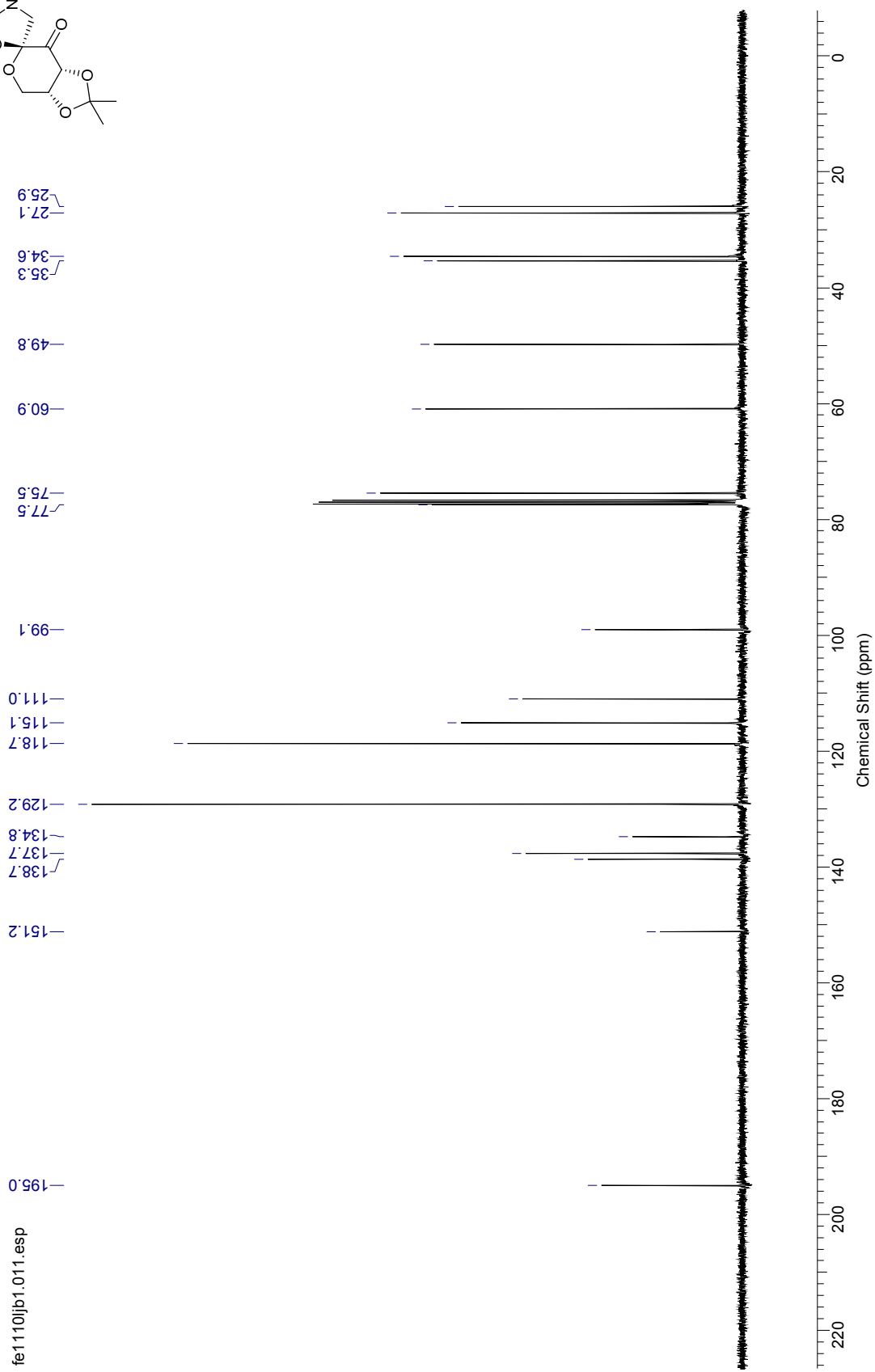
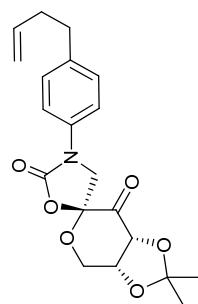
[75 MHz, CDCl_3]





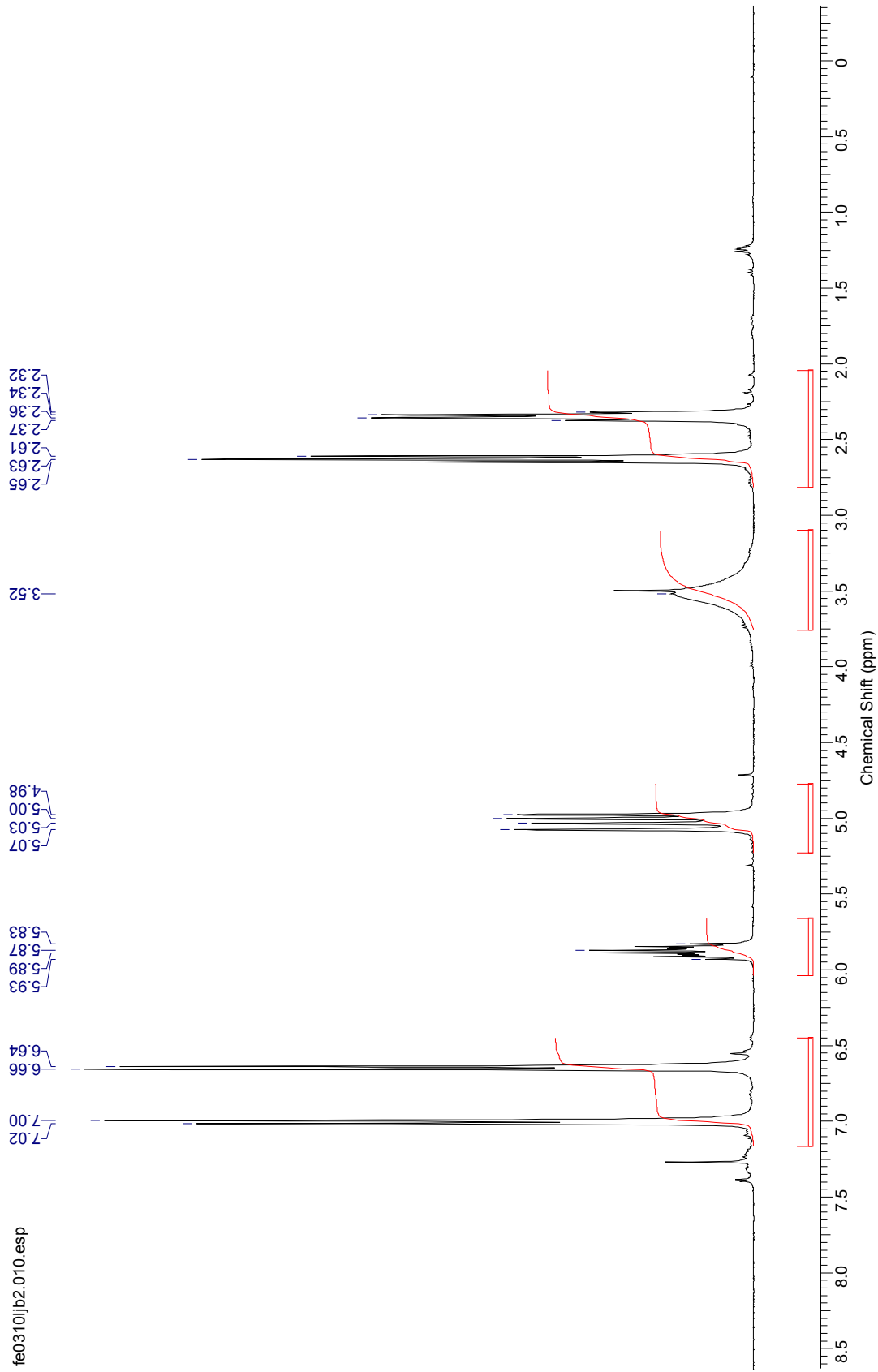
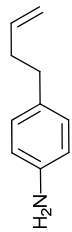
^{13}C NMR for (3*aR*,5*S*,7*aR*)-3'-(4-(But-3-en-1-yl)phenyl)-2,2-dimethylhydrospiro[[1,3]dioxolo[4,5-*c*]pyran-6,5'-oxazolidine]-2',7 (7*aH*)-dione (**5**)

[100 MHz, CDCl_3]

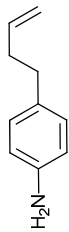


¹H NMR for 4-(But-3-en-1-yl) aniline (7)

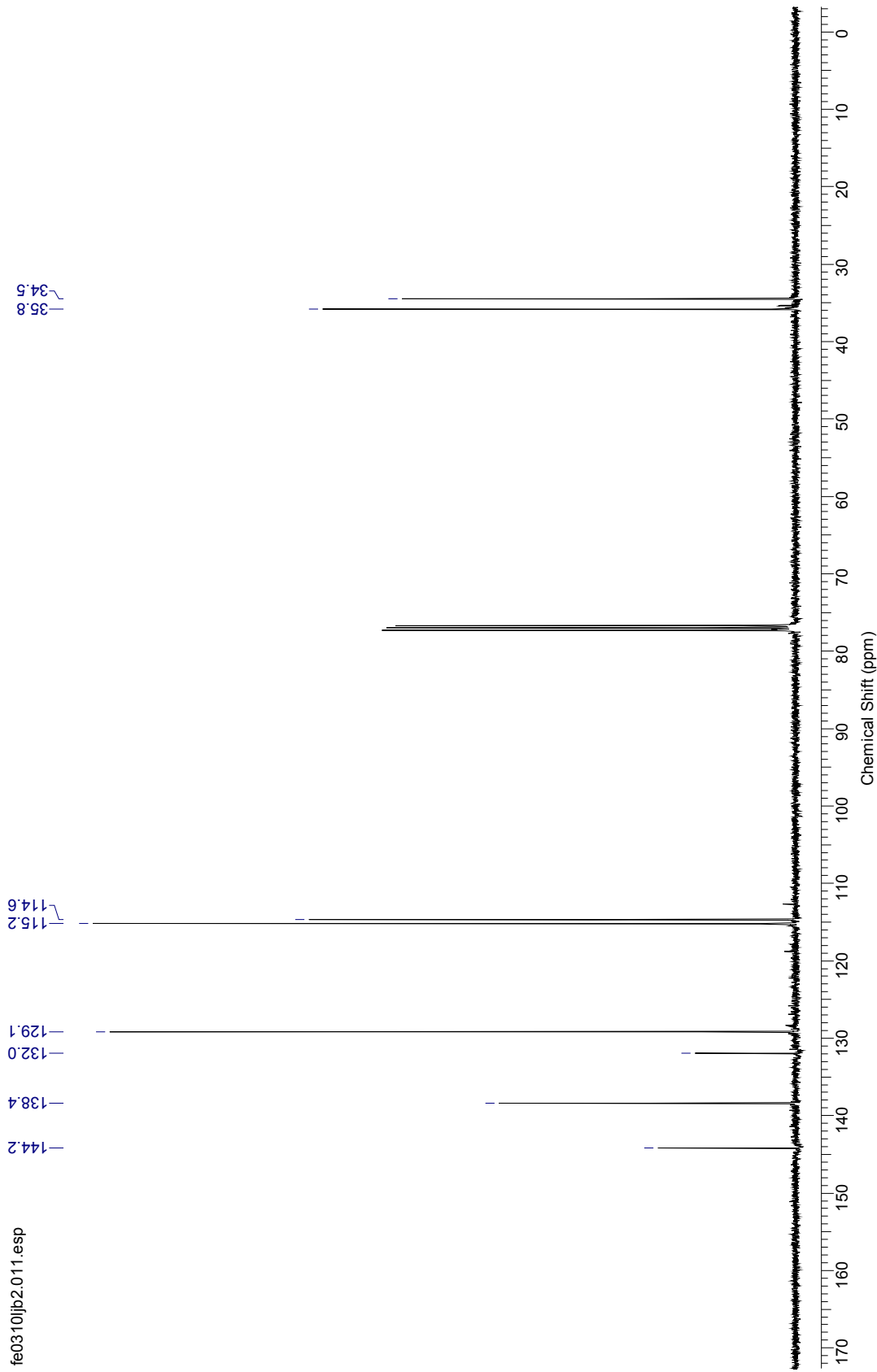
[400 MHz, CDCl₃]



¹³C NMR for 4-(But-3-en-1-yl) aniline (**7**)

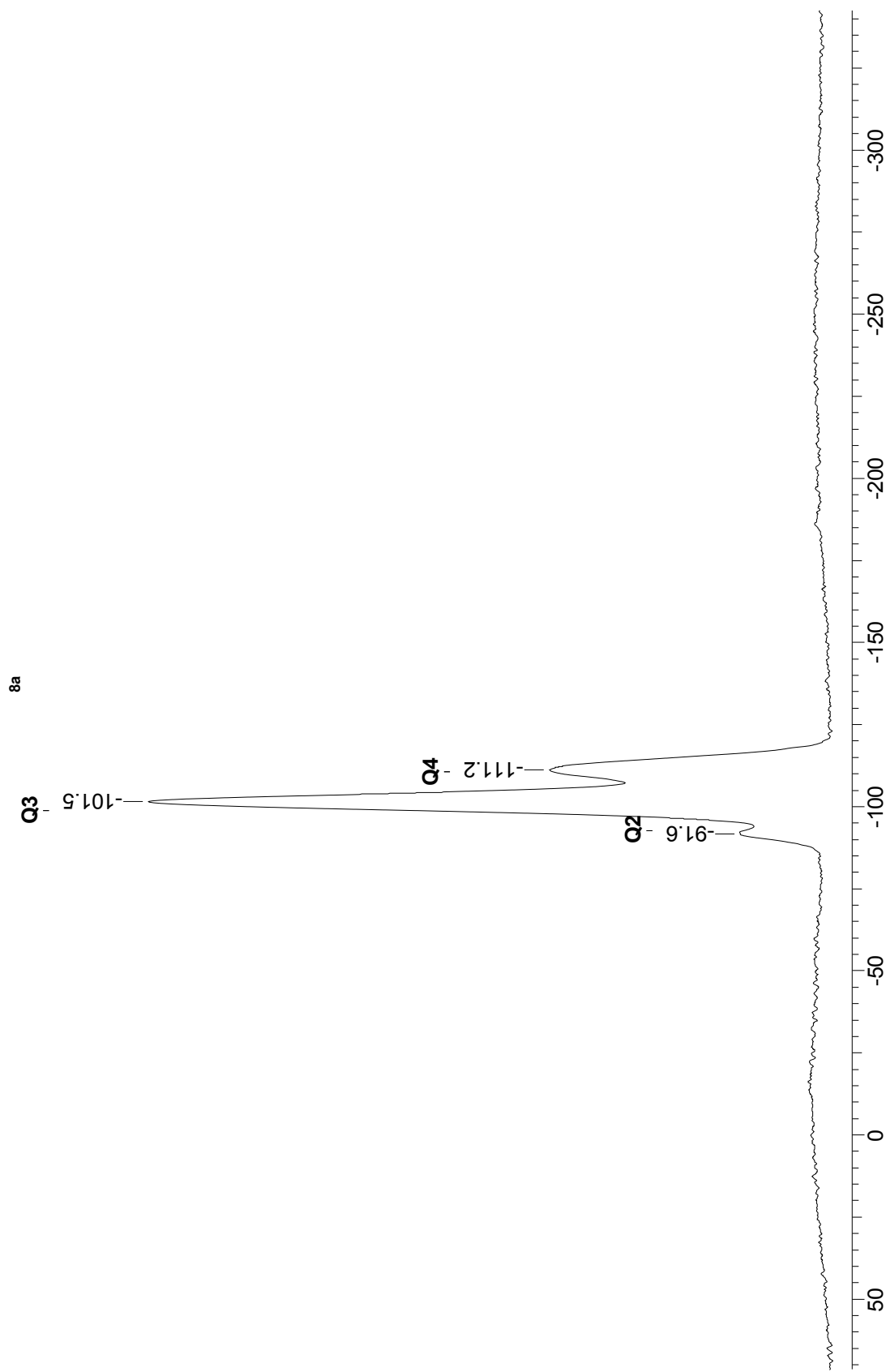
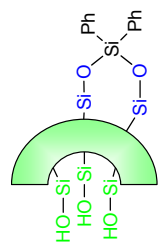


[100 MHz, CDCl₃]



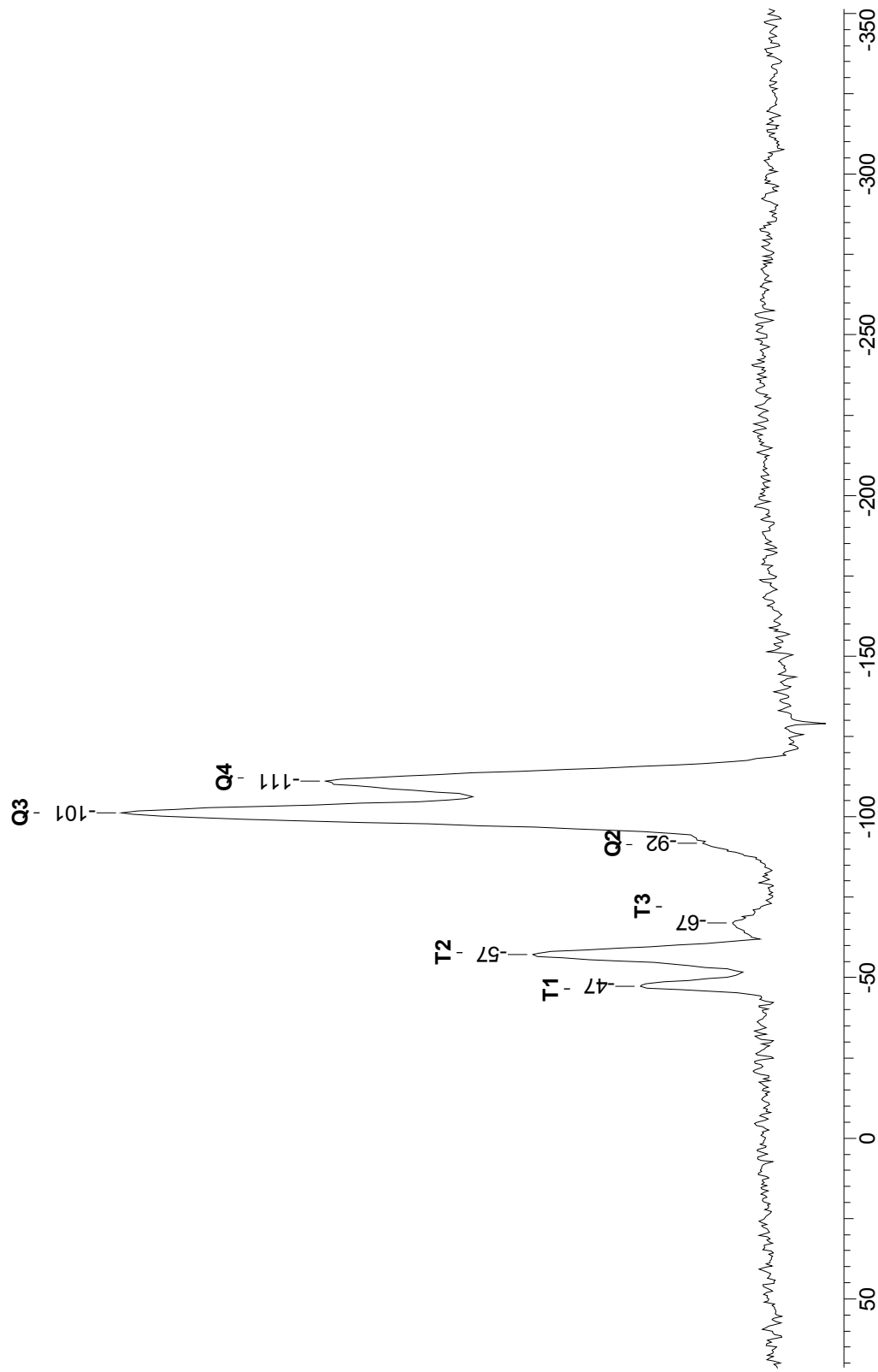
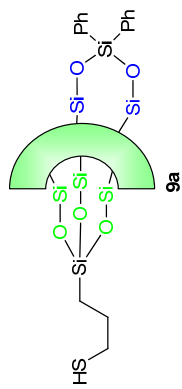
²⁹Si NMR Sílica **8a**

[79.4 MHz, CP: linear ramp on H, spin rate 6800 Hz]



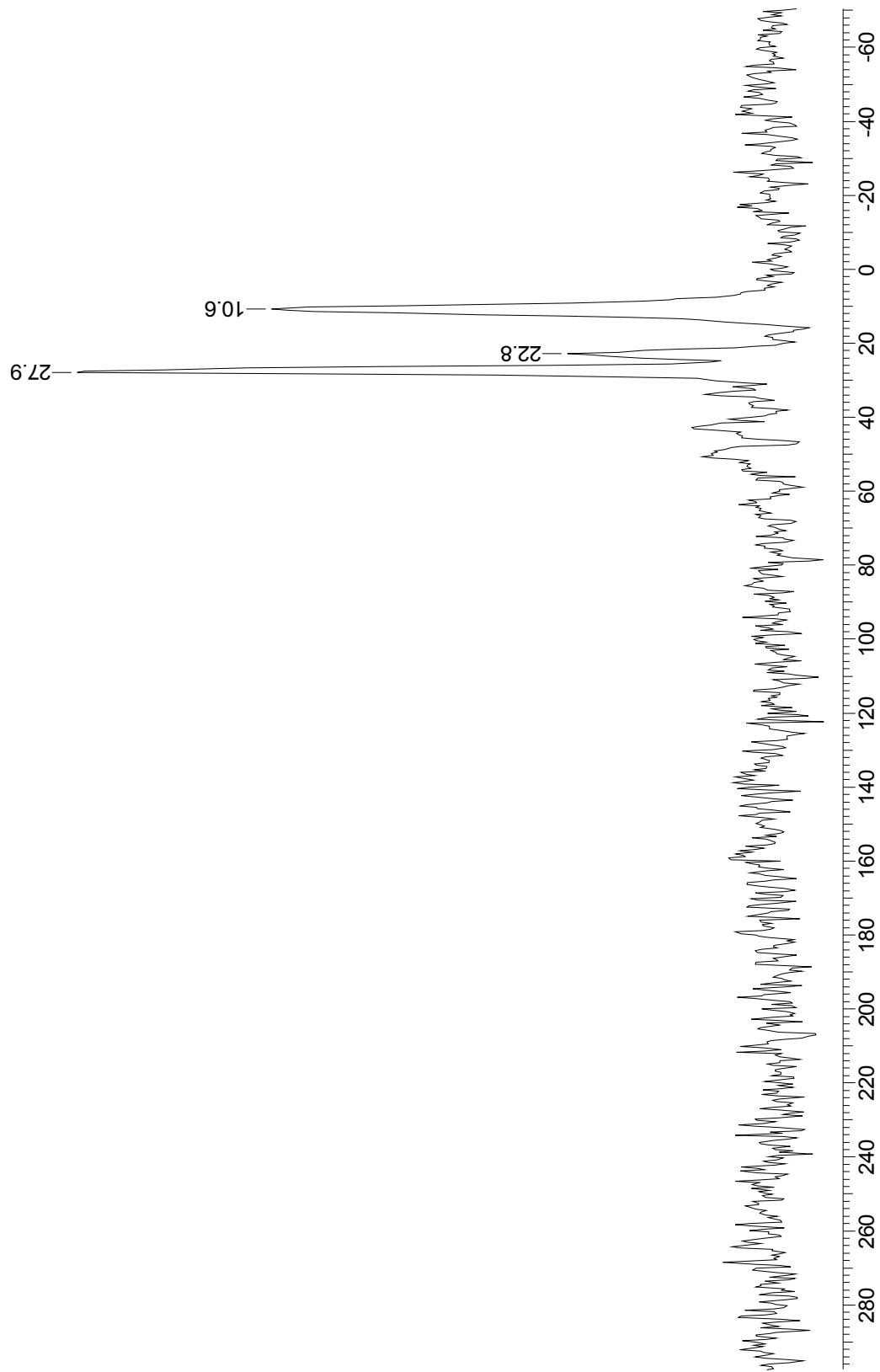
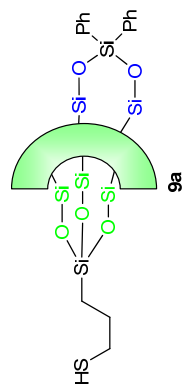
²⁹Si NMR Sílica **9a**

[79.4 MHz, CP: linear ramp on H, spin rate 6800 Hz]



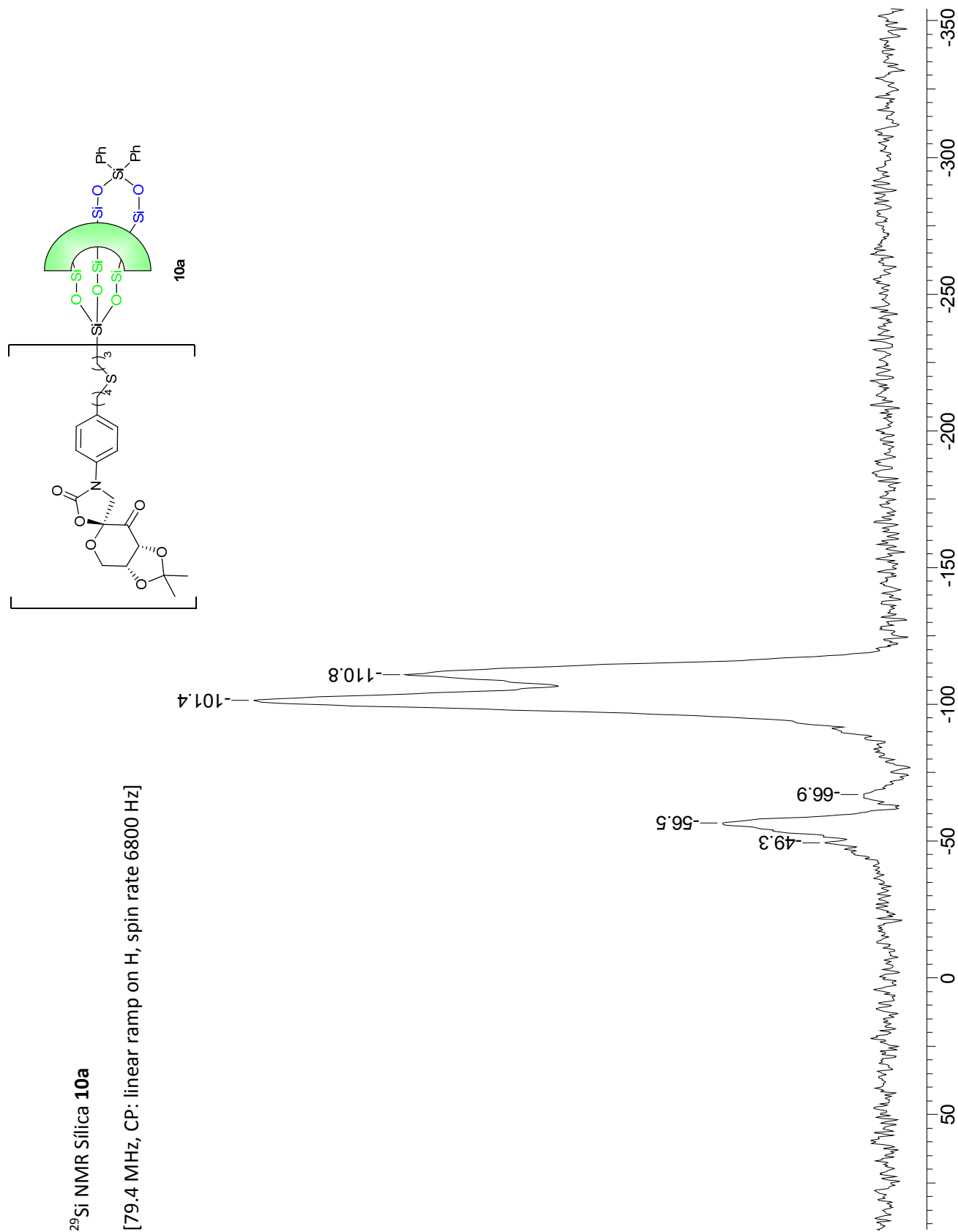
¹³C NMR Sílica **9a**

[100 MHz, CP: linear ramp on H, spin rate 6800 Hz]



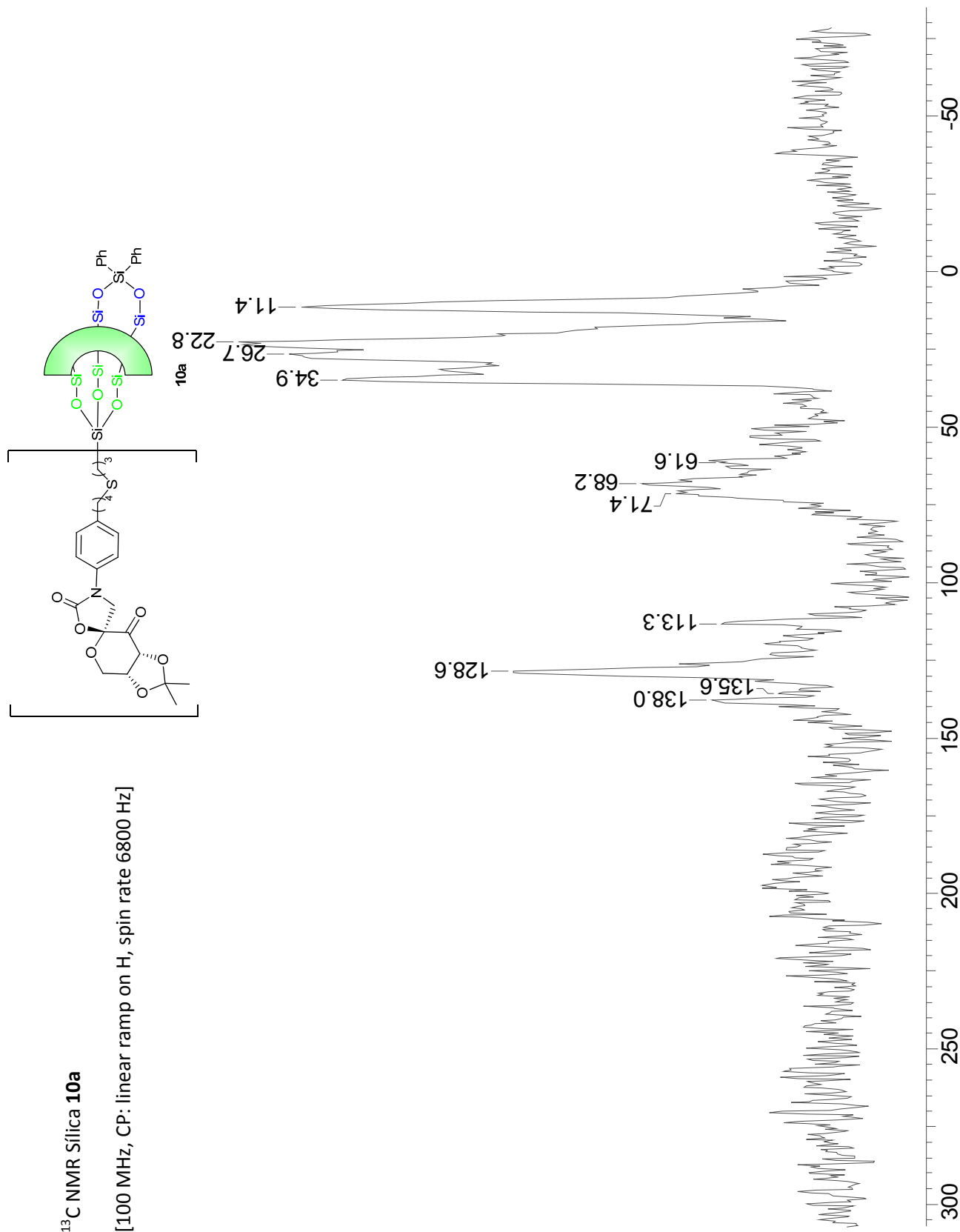
²⁹Si NMR Silica **10a**

[79.4 MHz, CP: linear ramp on H, spin rate 6800 Hz]

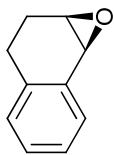


¹³C NMR Sílica **10a**

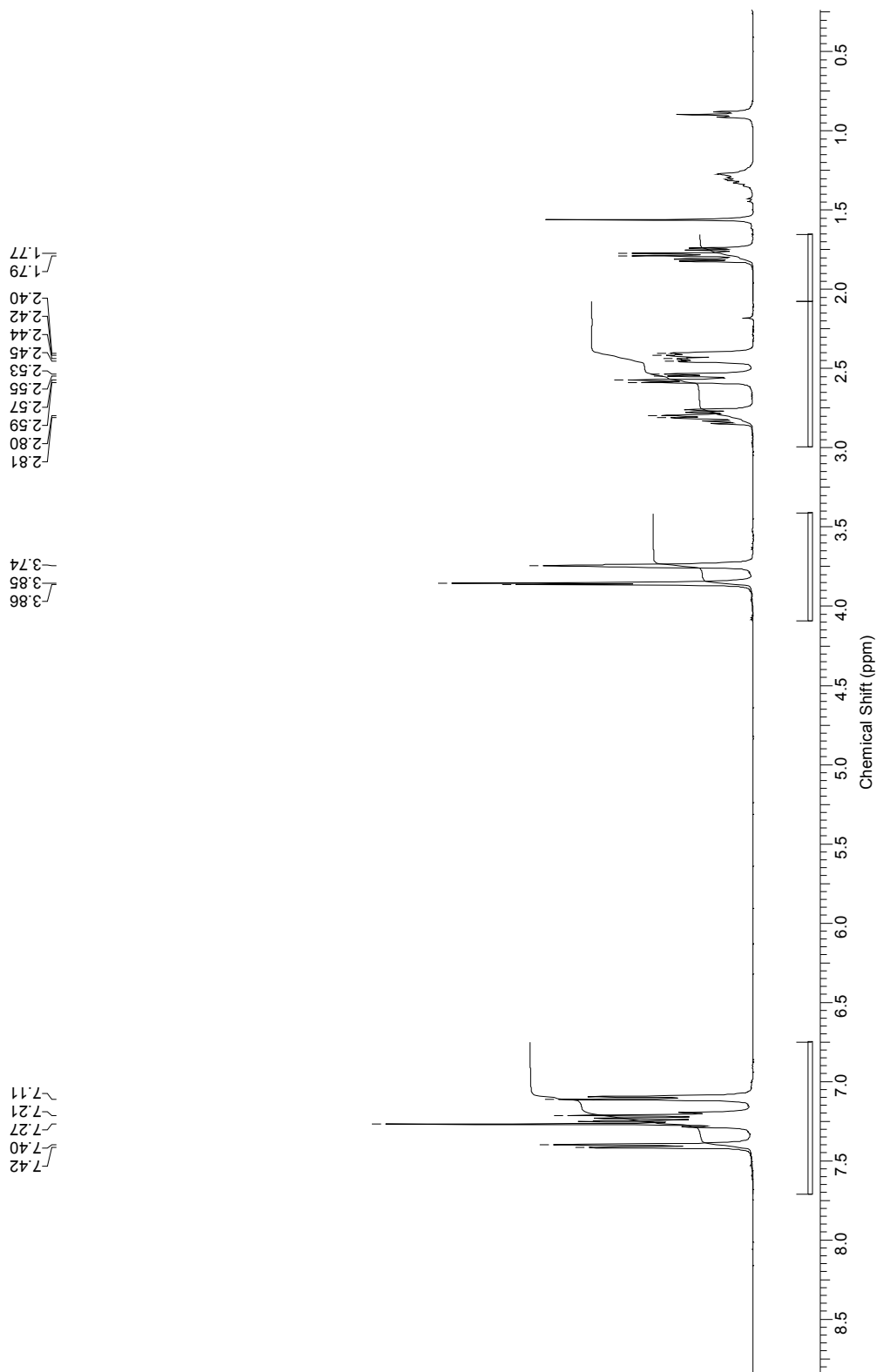
[100 MHz, CP: linear ramp on H, spin rate 6800 Hz]

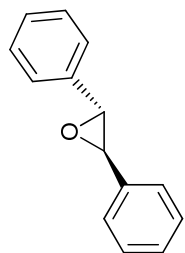


NMR spectroscopic data (epoxides)

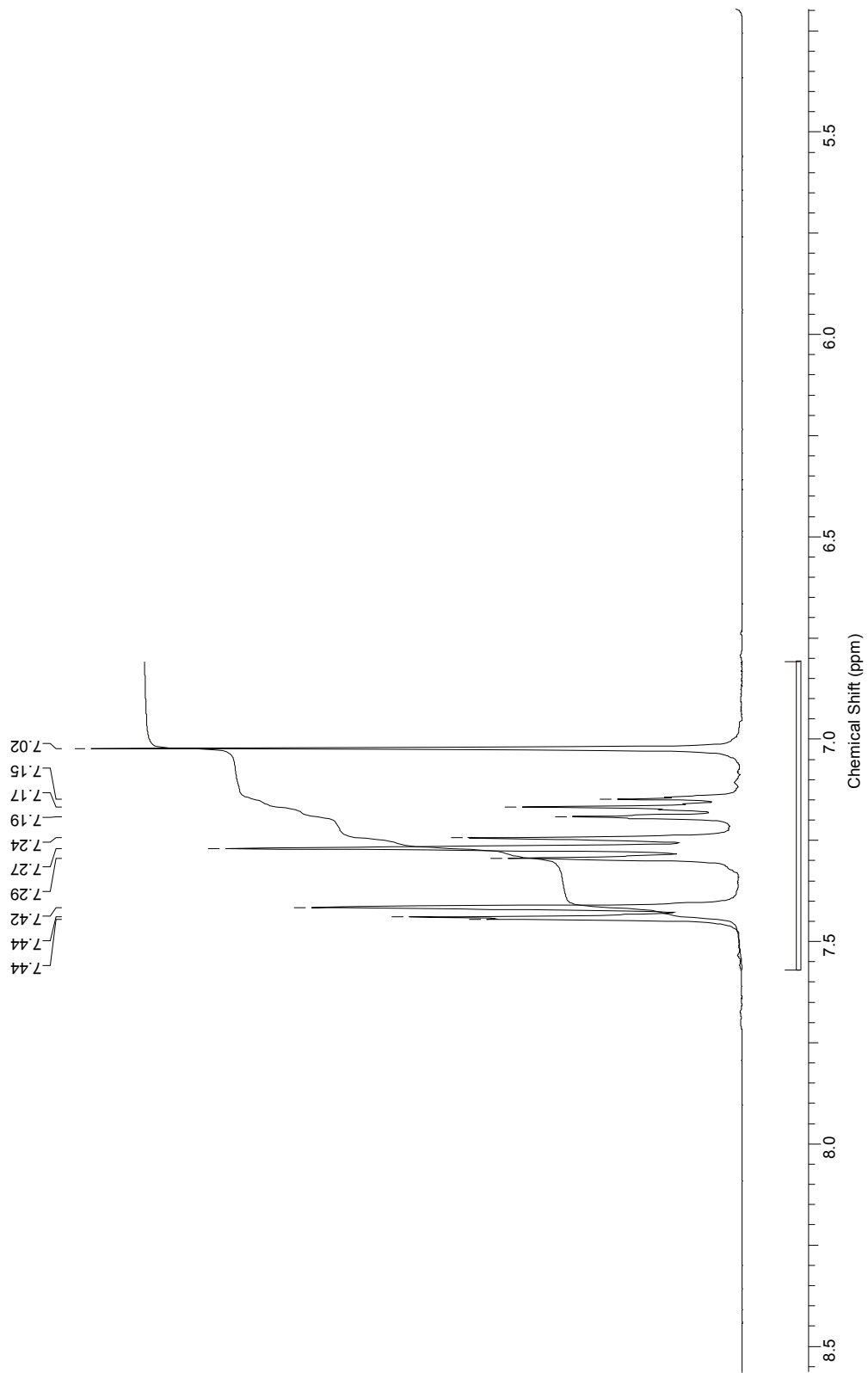


¹H NMR (1*S*,2*R*)- 1,2-dihydronaphthalene oxide (table 4, entry 1)
[400 MHz, CDCl₃]

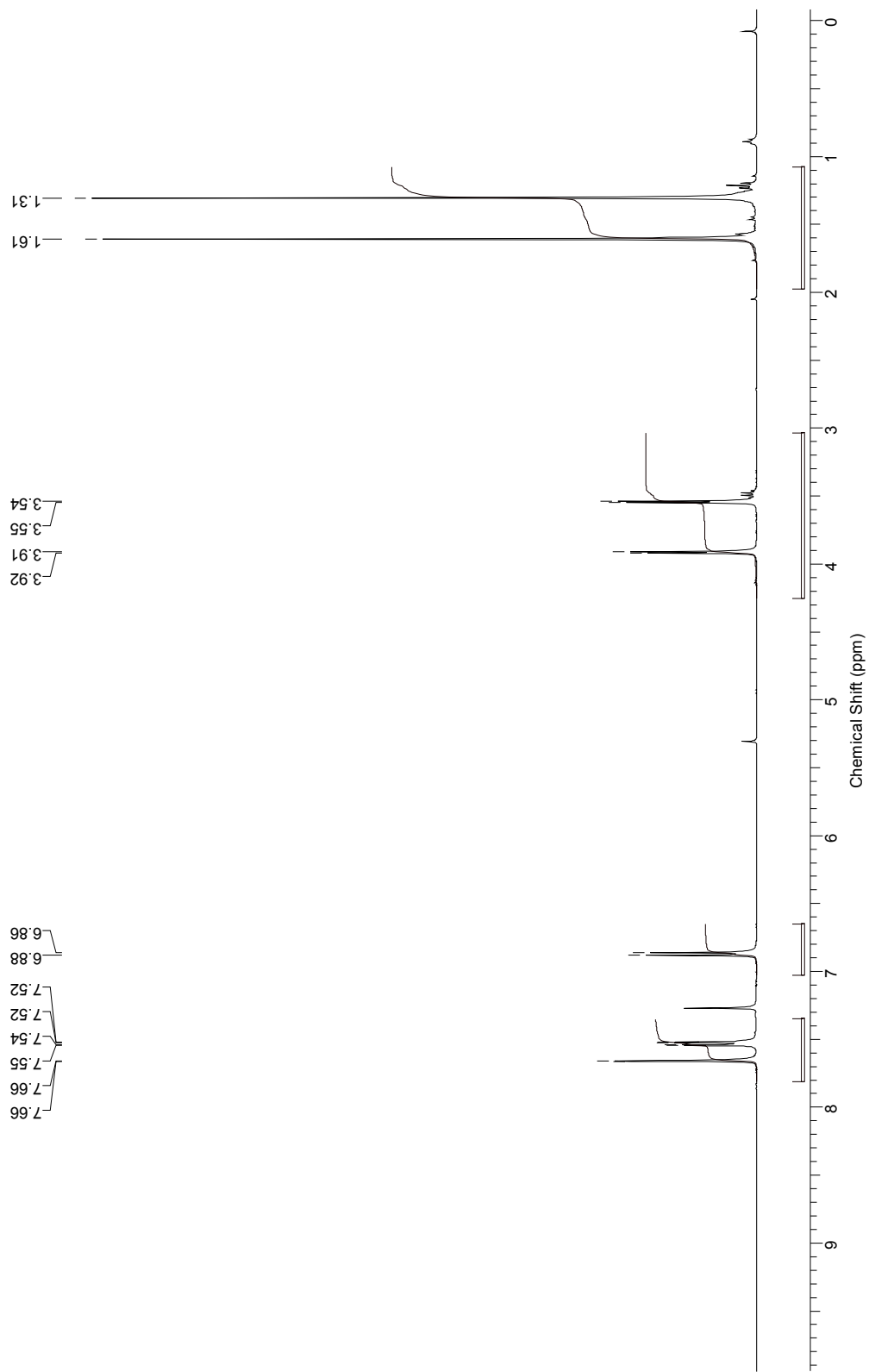
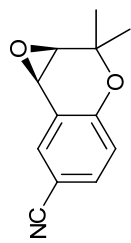




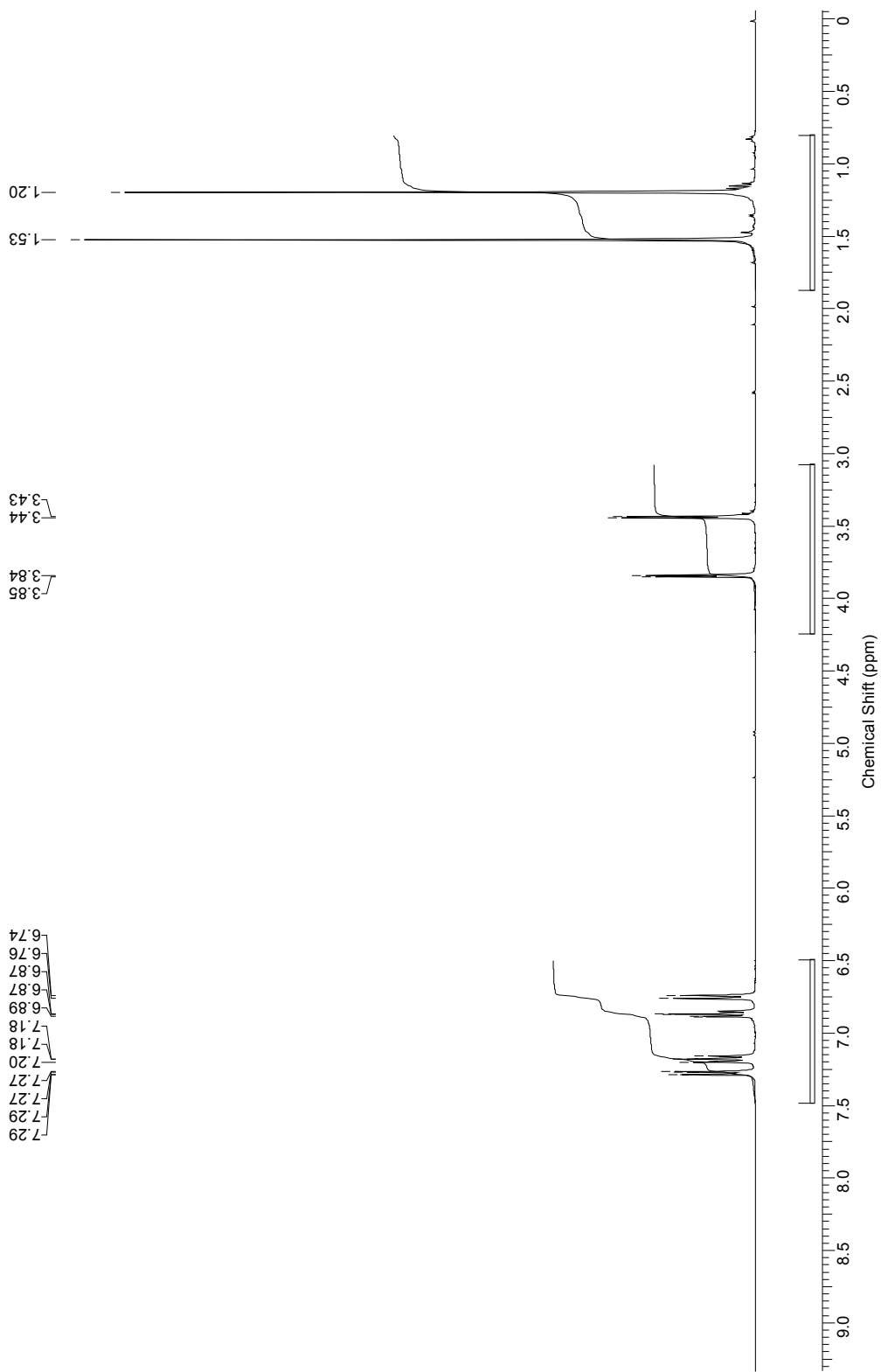
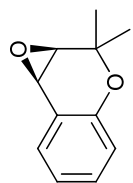
^1H NMR (*R,R*)-*trans*-stilbene oxide (table 4, entry 2)
[300 MHz, CDCl_3]



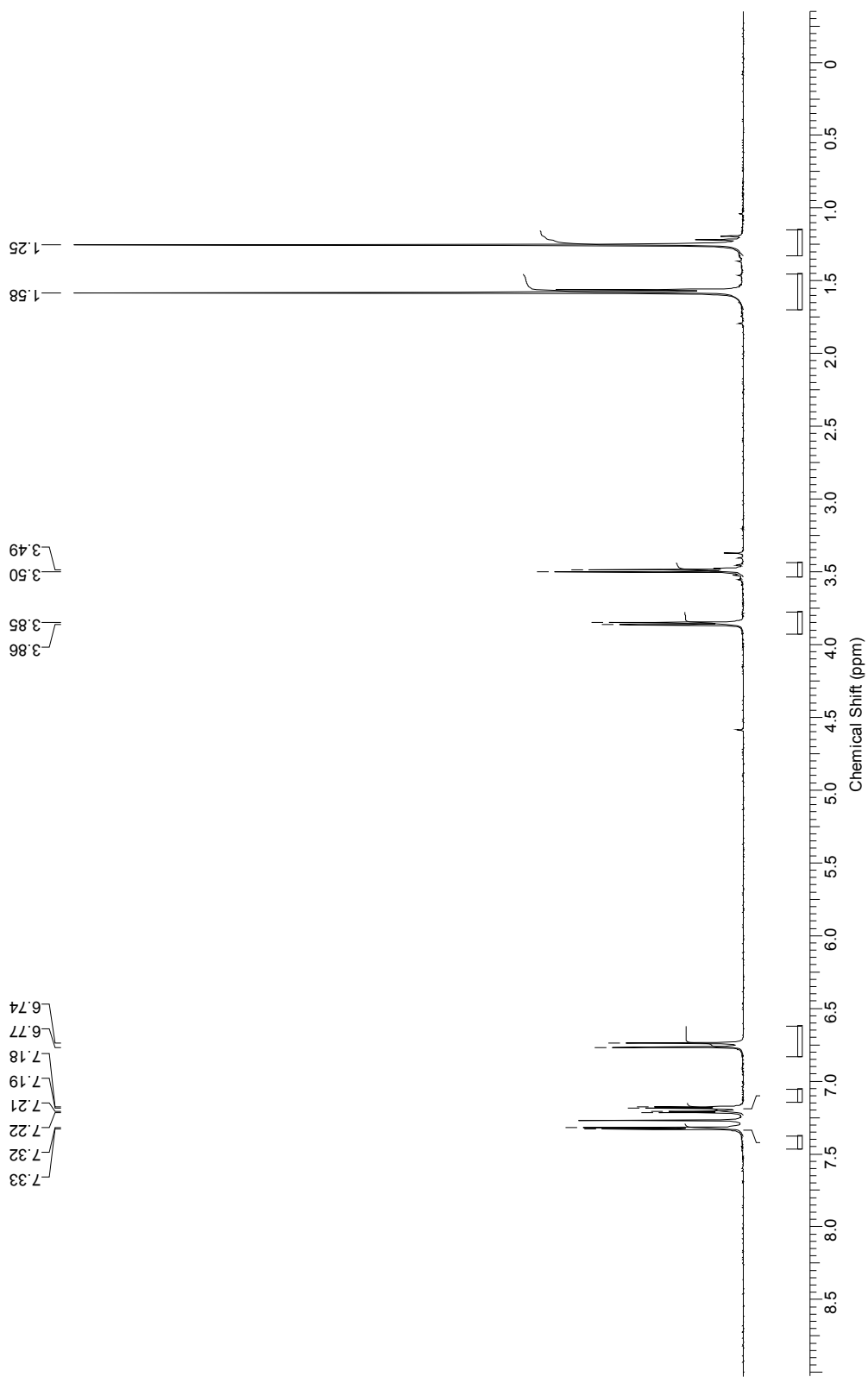
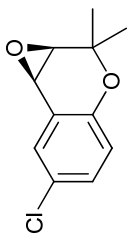
¹H NMR (3R,4R)-2,2-dimethyl-6-cyano-chromene oxide (table 4, entry 3)
[300 MHz, CDCl₃]

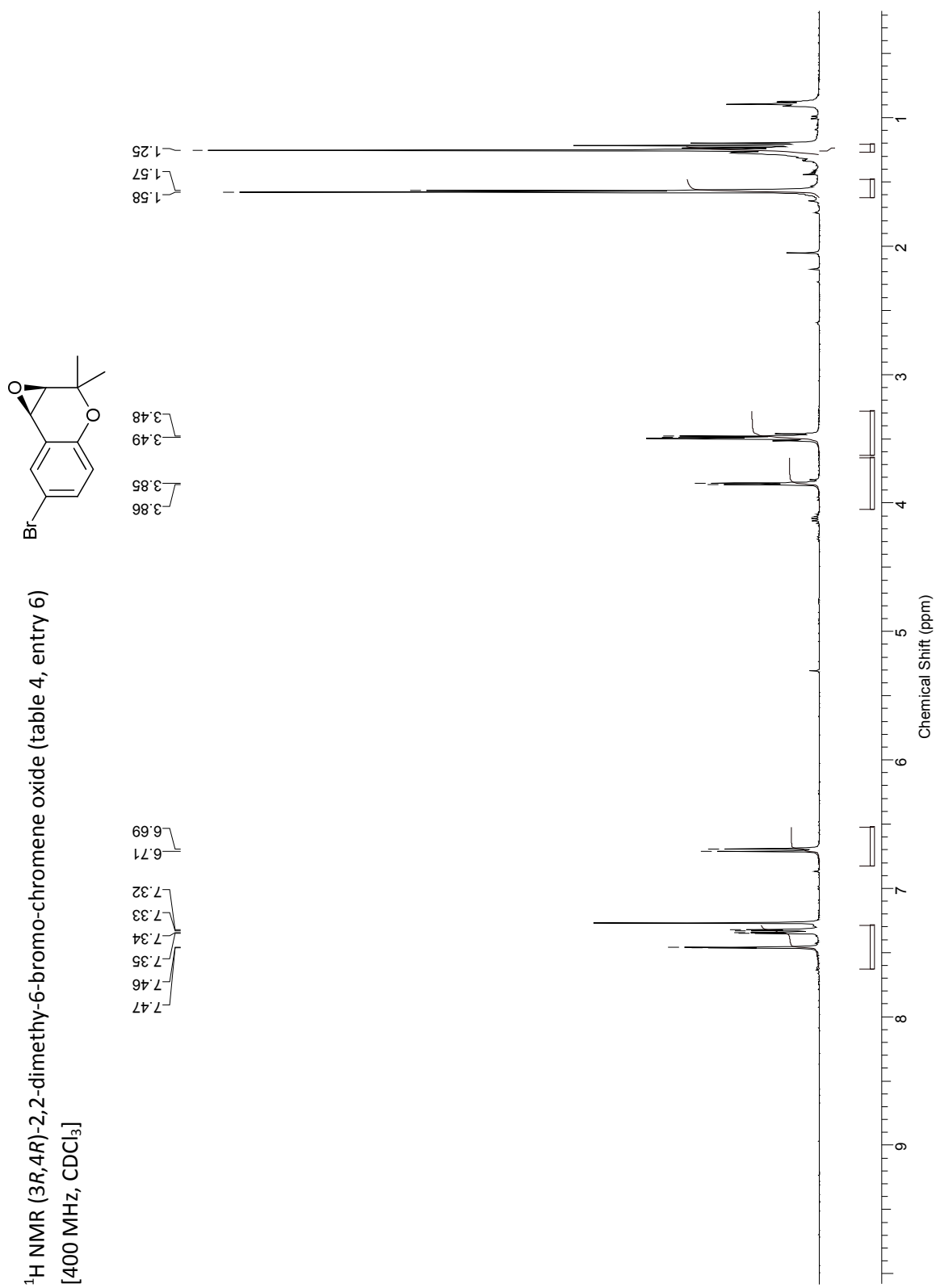


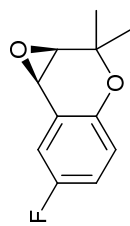
¹H NMR (3*R*,4*R*)-2,2-dimethylchromene oxide (table 4, entry 4)
[400 MHz, CDCl₃]



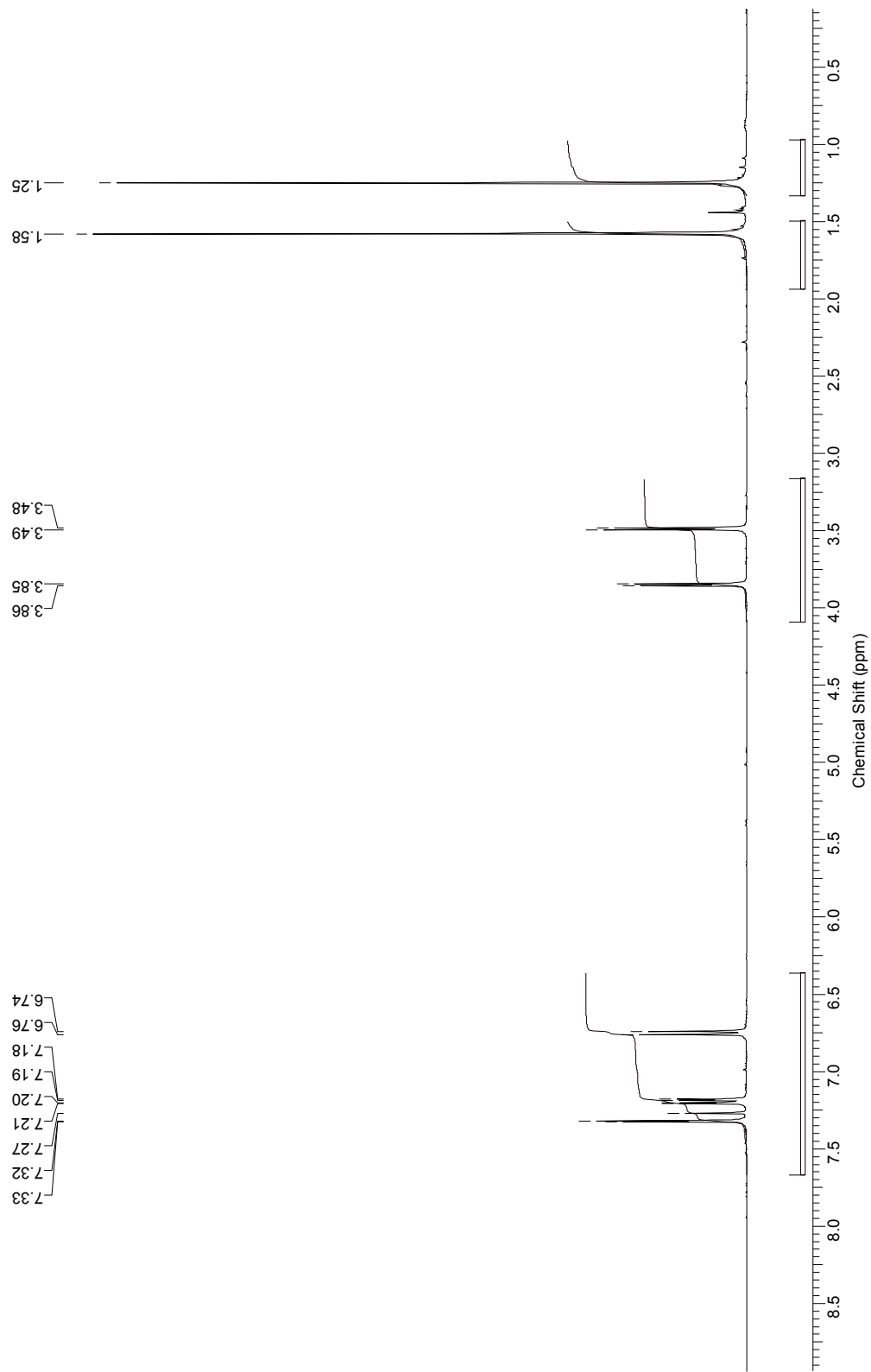
¹H NMR (3*R*,4*R*)-2,2-dimethyl-6-chloro-chromene oxide (table 4, entry 5)
[300 MHz, CDCl₃]



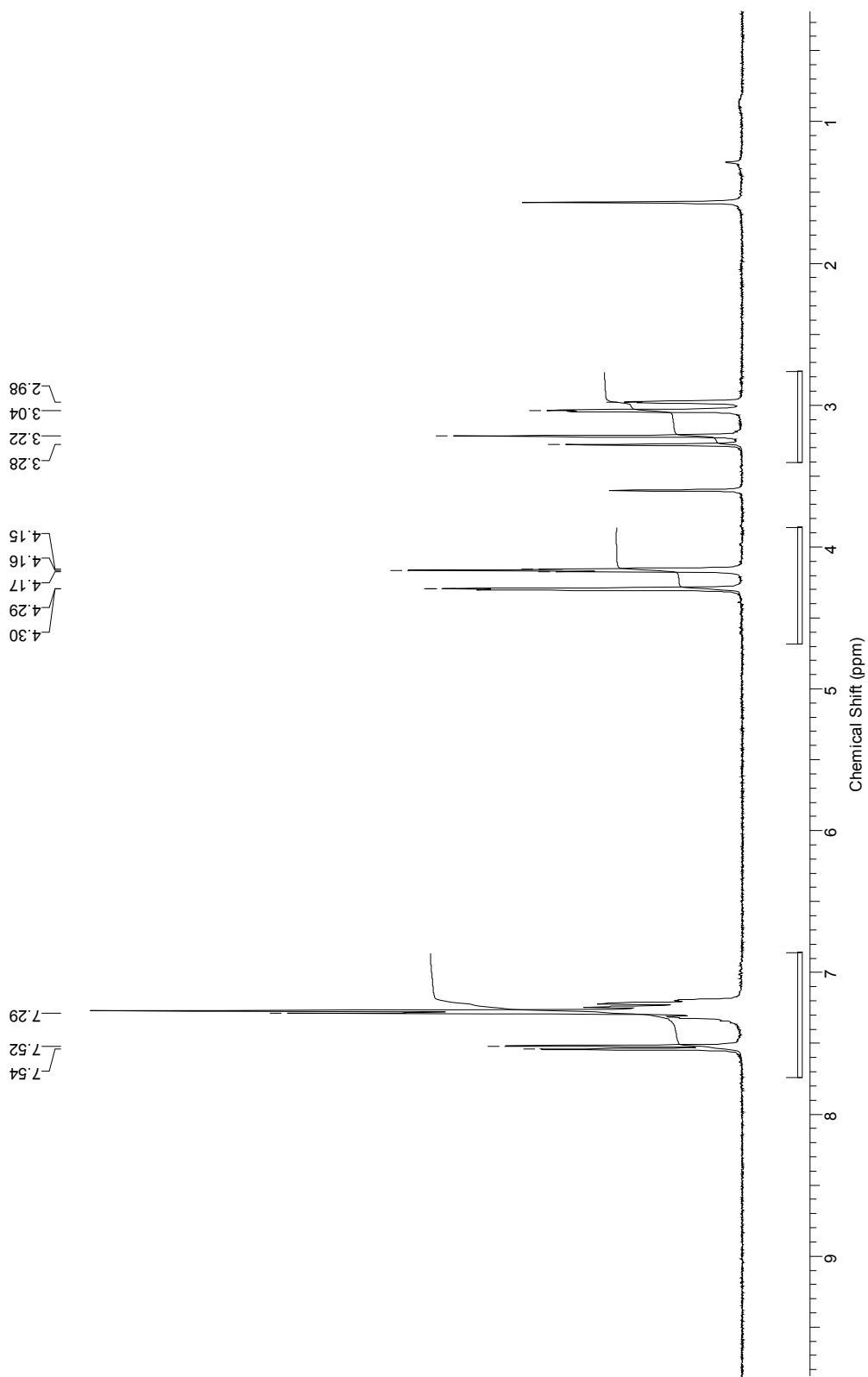




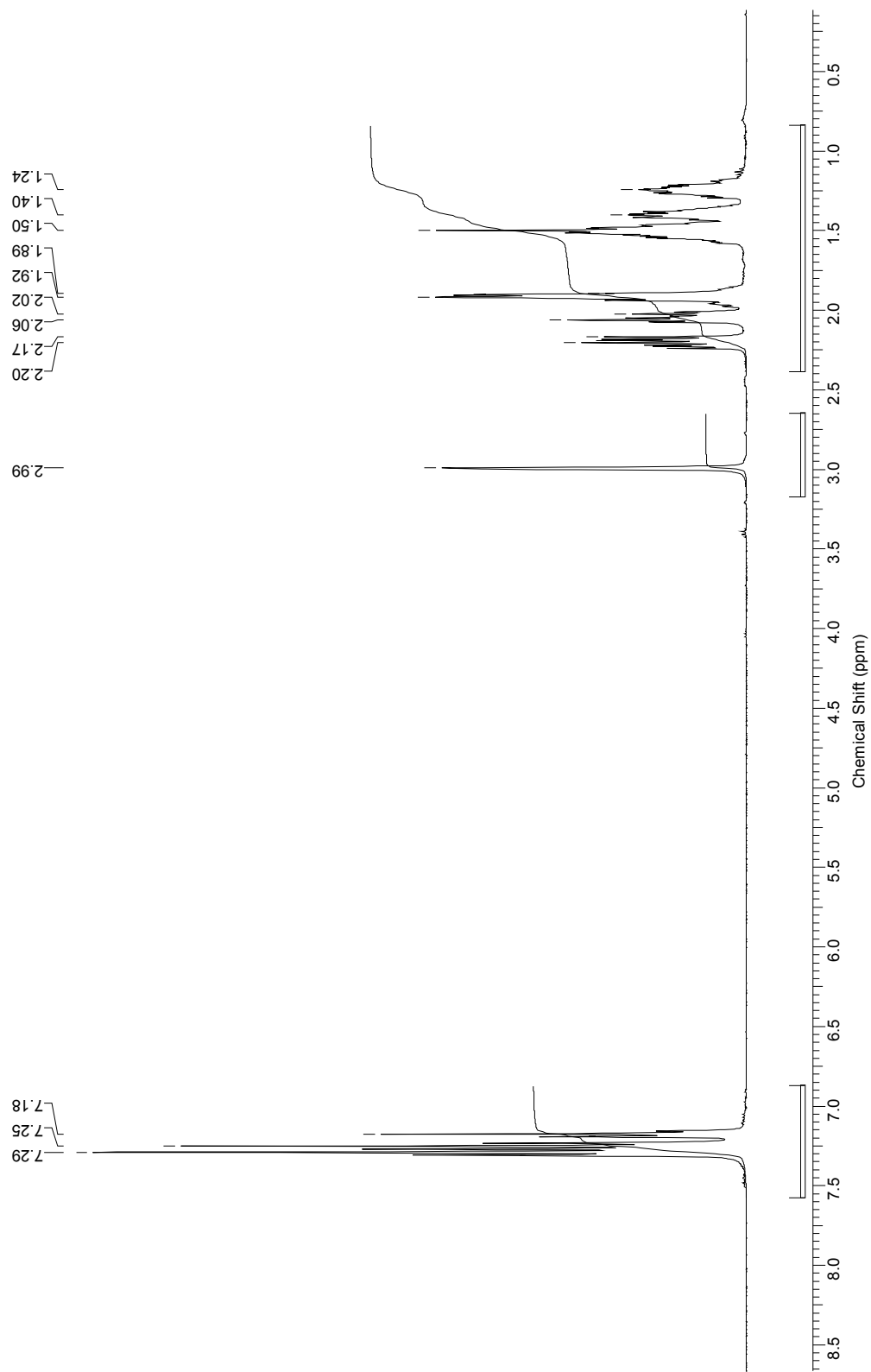
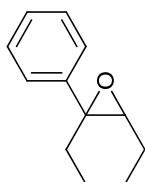
^1H NMR ($3R,4R$)-2,2-dimethyl-6-fluoro-chromene oxide (table 4, entry 7)
[400 MHz, CDCl_3]



¹H NMR (1*R*,2*S*)-indene oxide (table 4, entry 4)
[300 MHz, CDCl₃]



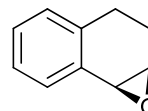
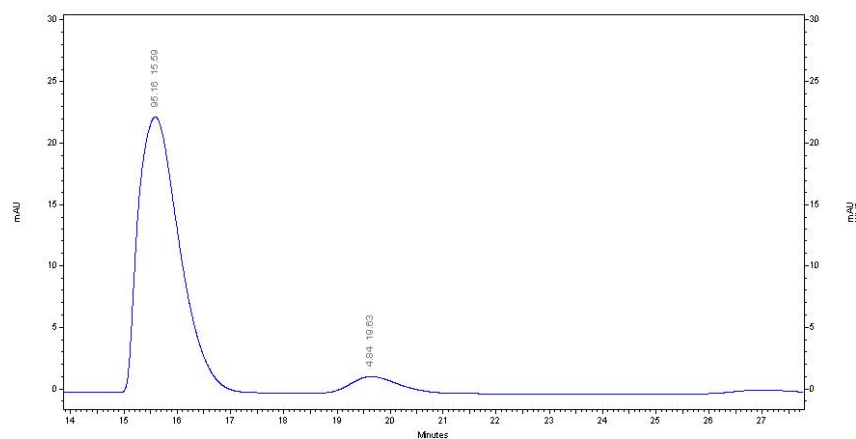
¹H NMR 1-Phenylcyclohexene oxide (Table 4, entry 5)
[300 MHz, CDCl₃]



Chiral HPLC chromatograms for epoxides

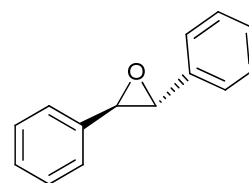
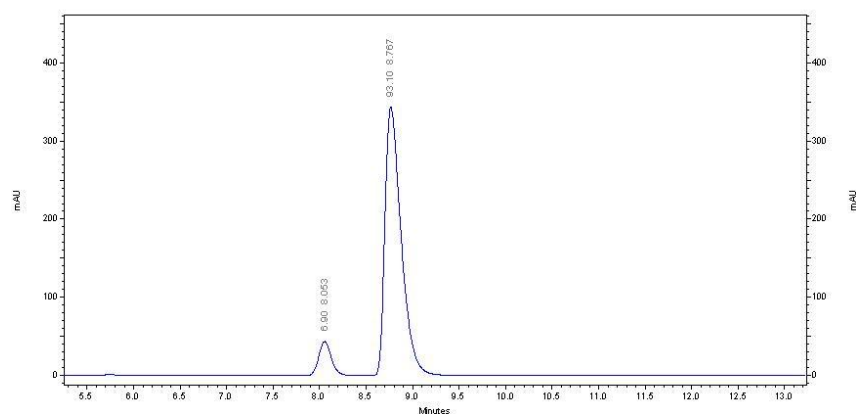
HPLC chromatogram of (1*S*,2*R*)-1,2-dihydronaphthalene oxide (table 4, entry 1)

[Column: Chiralcel OBH column; Eluent: 1 % IPA / hexanes; Flowrate: 1.0 mL min⁻¹; Detection: UV254nm] (e.e. 90 %)



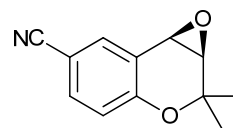
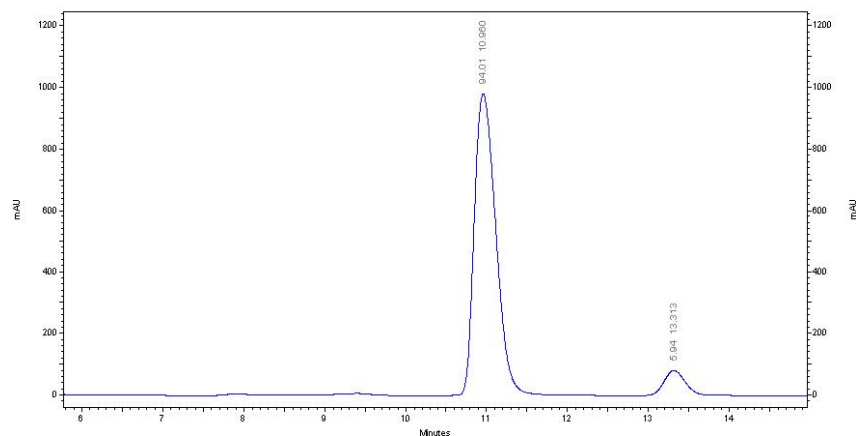
HPLC chromatogram of (*R,R*)-*trans*-stilbene oxide (table 4, entry 2)

[Column: Chiralcel ODH column; Eluent: 2 % IPA / hexanes; Flowrate: 1.0 mL min⁻¹; Detection: UV254nm] (e.e. 86 %)



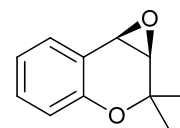
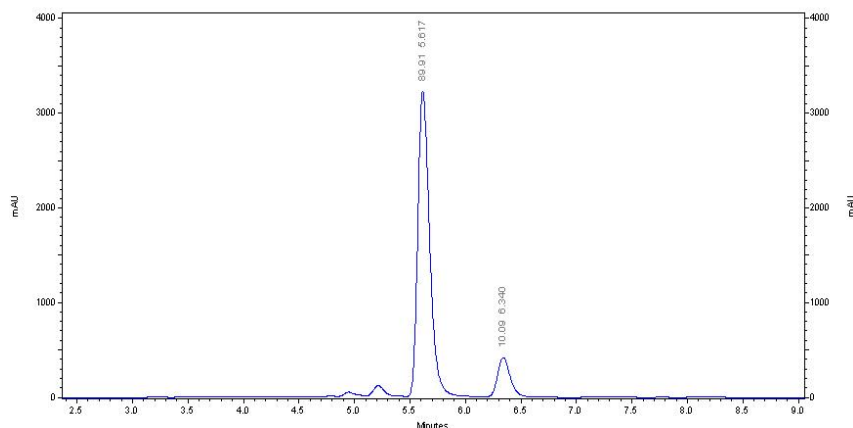
HPLC chromatogram of (3*R*,4*R*)-2,2-dimethyl-6-cyano-chromene oxide (table 4, entry 3)

[Column: Chiralcel ODH column; Eluent: 10 % IPA / hexanes; Flowrate: 1.0 mL min⁻¹; Detection: UV220nm] (e.e. 88 %)



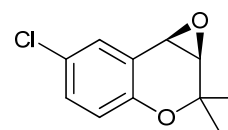
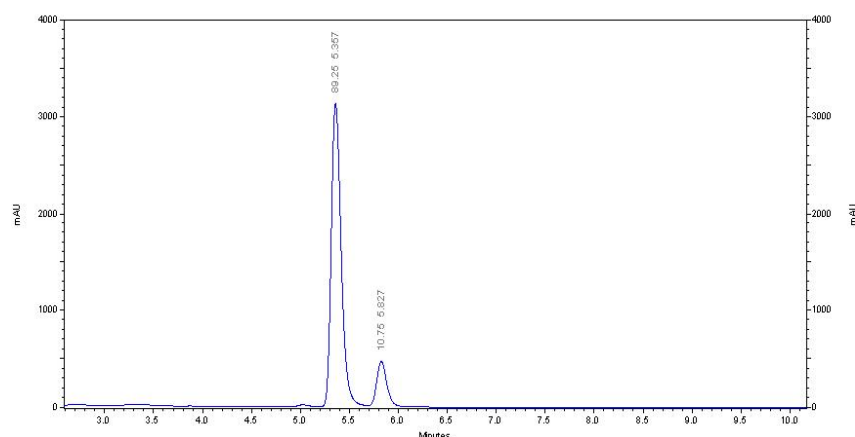
HPLC chromatogram of (3*R*,4*R*)-2,2-dimethylchromene oxide (table 4, entry 4)

[Column: Chiralcel ODH column; Eluent: 10 % IPA / hexanes; Flowrate: 1.0 mL min⁻¹; Detection: UV220nm] (e.e. 80 %)



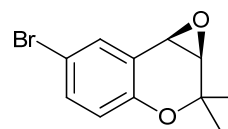
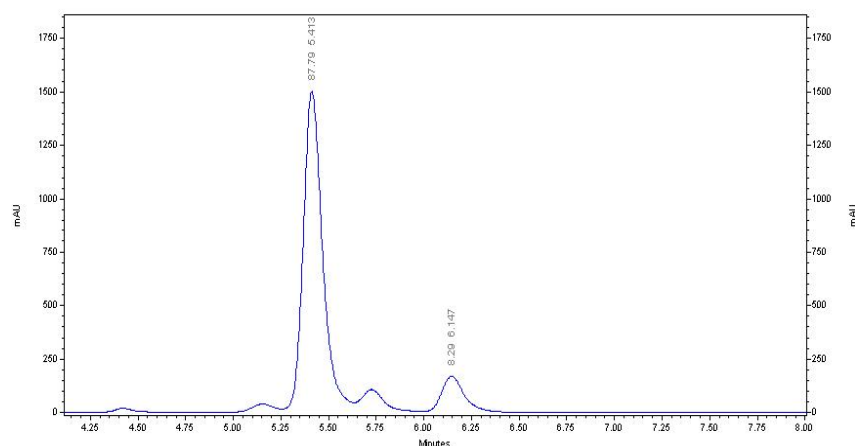
HPLC chromatogram of (3*R*,4*R*)-2,2-dimethyl-6-chloro-chromene oxide (table 4, entry 5)

[Column: Chiralcel ODH column; Eluent: 10 % IPA / hexanes; Flowrate: 1.0 mL min⁻¹; Detection: UV220nm] (e.e. 79 %)



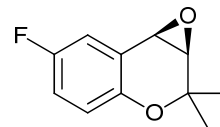
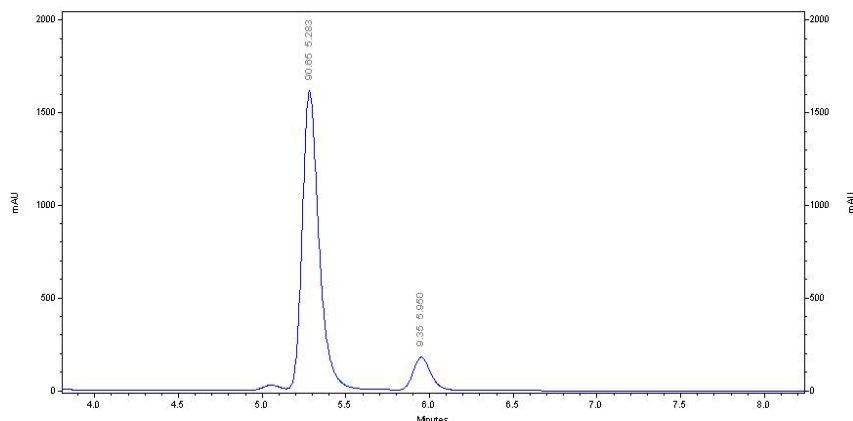
HPLC chromatogram of (3*R*,4*R*)-2,2-dimethyl-6-bromo-chromene oxide (table 4, entry 6)

[Column: Chiralcel ODH column; Eluent: 10 % IPA / hexanes; Flowrate: 1.0 mL min⁻¹; Detection: UV220nm] (e.e. 83 %)



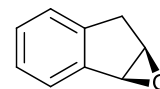
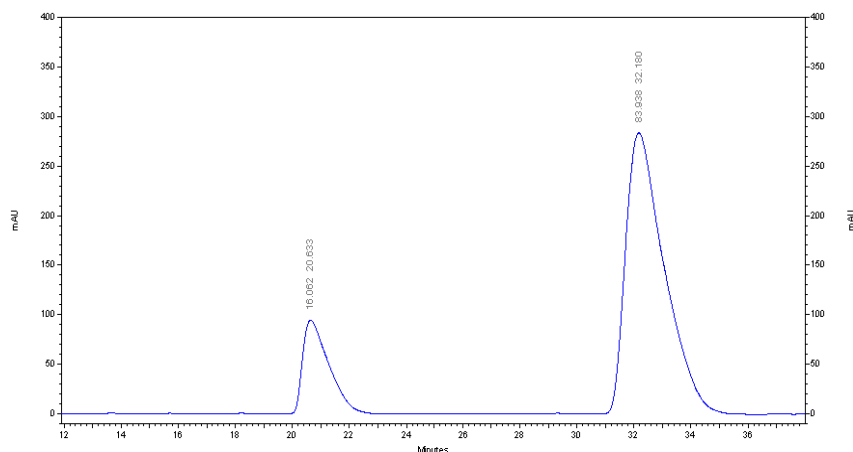
HPLC chromatogram of (3*R*,4*R*)-2,2-dimethyl-6-fluoro-chromene oxide (table 4, entry 7)

[Column: Chiralcel ODH column; Eluent: 10 % IPA / hexanes; Flowrate: 1.0 mL min⁻¹; Detection: UV220nm] (e.e. 81 %)



HPLC chromatogram of (1*R*,2*S*)-indene oxide (table 4, entry 8)

[Column: Chiralcel OB column; Eluent: 0.5 % IPA / hexanes; Flowrate: 1.0 mL min⁻¹; Detection: UV220nm] (e.e. 68 %)



HPLC chromatogram of phenylcyclohexene oxide (table 4, entry 9)

[Column: Chiralcel ODH column; Eluent: 1 % IPA / hexanes; Flowrate: 0.5 mL min⁻¹; Detection: UV220nm] (e.e. 18 %)

