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Synthesis of the *trans*-hydrindane core of dictyoxetane

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Supporting Information

Contents

Determination of stereochemistry of **1**, **3**, **7**, **8**.....S2

X-ray crystallography.....S5

NMR spectra..... S8

NMR analysis to determine the stereochemistry of alcohol 5, epoxides 7 and 8 and alcohol 1.

Alcohol **5**:

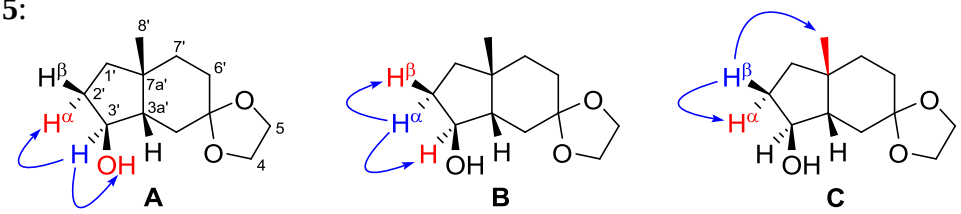


Figure 1: nOe studies of alcohol **5**. Observed nOe signals upon A) irradiation of H-3'; B) irradiation of H-2' (H^{α}); C) irradiation of H-2' (H^{β}).

Irradiation of H-3' gave rise to an nOe to the alcohol hydrogen and to a vicinal proton at H-2' (H^{α}). No nOe was observed to the methyl group (figure 1A). Irradiation of H^{α} gave an nOe to H-3' and to the geminal partner H^{β}. Again, no nOe to the methyl group was detected (figure 1B). Irradiation of H^{β} gave rise to nOe signals to H^{α} and to the methyl group (figure 1C). This data indicates that the methyl group is on the same face of the molecule as H^{β} and the OH group, and is consistent with H^{α} and H-3' being on the opposite face, indicating that the stereochemistry is as presented. From the *cis* ring junction an nOe would be expected between Me and H-3a'. However, due to the H-3a' signals overlapping with several other proton environments, the observed nOe cannot be unambiguously assigned as arising from any one particular proton.

Epoxide **7**:

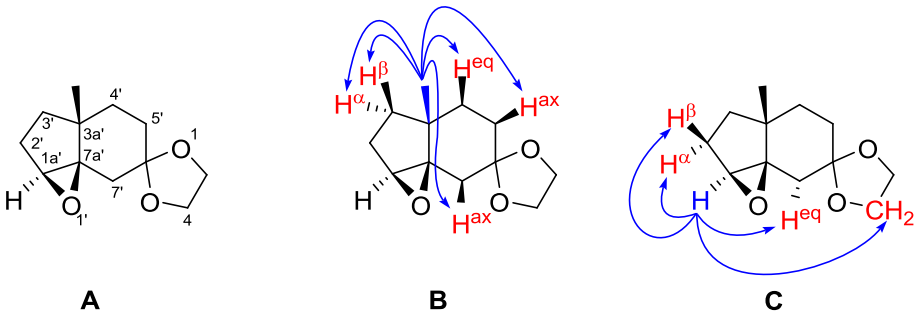


Figure 2: nOe studies of epoxide **7**. A) Numbering of **7**; B) observed nOe signals upon irradiation of the methyl group; C) observed irradiation of H-1a'.

Irradiation of the methyl protons gave rise to nOe signals to both H-3' protons, H-4'$_{eq}$, H-5'$_{ax}$ and H-7'$_{ax}$ (figure 2B). No nOe was observed to H-1a', suggesting that the epoxide is on the same face as the methyl group. Irradiation of H-1a' gave rise to a very weak signal to H-7'$_{ax}$ (not shown) and a strong nOe to H-7'$_{eq}$ (figure 2C). This indicates that the epoxide is on the

same face of the molecule as H-7'$_{ax}$, i.e. the same face as the methyl group. Also observed were signals to both H-2' protons and to protons on the acetal protecting group.

Epoxide **8**:

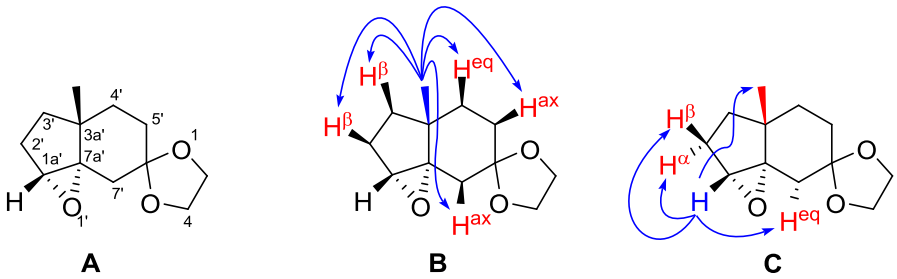


Figure 3: nOe studies of epoxide **8**. A) Numbering of **8**; B) observed nOe signals upon irradiation of the methyl group; C) observed irradiation of H-1a'.

Irradiation of the methyl protons gave rise to nOe signals to one H-3' proton, H-4'$_{eq}$, H-5'$_{ax}$, H-7'$_{ax}$ and H-2' (figure 3A). A very weak nOe was observed to H-1a' (not shown). Irradiation of H-1a' gave rise to a strong nOe signal to H-7'$_{eq}$, along with signals to both H-2' protons and to the methyl group (figure 3B and C). This confirms that the epoxide is on the opposite face of the molecule to the methyl group.

Alcohol **1**:

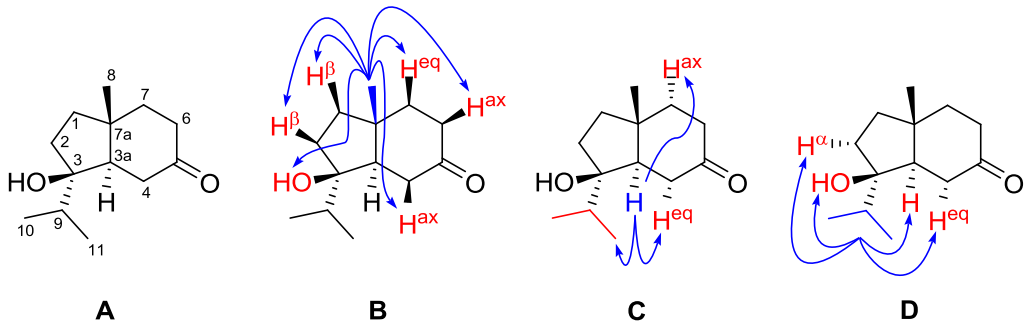


Figure 4: nOe studies of alcohol **1**. A) Numbering of **1**; B) observed nOe signals upon irradiation of the methyl group; C) observed nOe signals upon irradiation of H-3a; D) observed nOe signals upon irradiation of *iso*-propyl methyl groups.

Irradiation of the methyl group gave rise to nOe signals to OH, indicating that the OH and the methyl are probably on the same face (figure 4B). Also observed were nOe signals to H-1, H-2, H-7$_{eq}$, H-6$_{ax}$ and one H-4 proton (presumably the axial). No nOe signal was observed to H-3a, suggesting that the methyl group and H-3a are on opposite faces. Irradiation of H-3a gave rise to nOe signals to the *iso*-propyl methyl groups suggesting that they are on the same face of the molecule (figure 1C). Further signals to H-7$_{ax}$ and one of the H-4 protons (presumably

the equatorial) were also observed. Significantly, no signals arising from an nOe to the C-8 methyl group or to the OH were detected. Irradiation of the higher frequency ^{i}Pr methyl group gave the expected nOe signals to the other *iso*-propyl methyl group, H-9 and the OH group (figure 1D). Also observed were nOe signals to H-3a, H-2 and one of the H-4 protons (presumably equatorial). This indicates that H-3a is on the same face as the *iso*-propyl group. Irradiation of the lower frequency *iso*-propyl methyl group gave a similar set of signals, though that arising from an nOe to H-3a was stronger in this case.

N.B. The resolved H-1 proton at 0.81 ppm shows strong ^{4}J coupling to C-6 in the HMBC.

X-Ray Crystallography

Suitable crystals were selected and datasets were measured on a Bruker SMART 6000 diffractometer ($\lambda_{Cu\text{-}K\alpha}$ = 1.5418 Å) for **6** and **10**. The data collections were driven by SMART[1] and processed by SAINTPLUS[2] and absorption corrections were applied using SADABS[3]. The structures were solved using ShelXS-97[4] and refined by a full-matrix least-squares procedure on F^2 in ShelXL-97,[4] in the case of **6** within SHELXTL.[5] All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were added at calculated positions and refined by use of a riding model with isotropic displacement parameters based on the equivalent isotropic displacement parameter (U_{eq}) of the parent atom. Figures were produced using OLEX2.[6]

1 *SMART, program for instrument control and data acquisition*, 1997, Bruker AXS, Inc. 5465 East Cheryl Parkway, Madison, Wisconsin 53711-5373, USA.

2 *SAINTPLUS, program suite for data processing*, 1997, Bruker AXS, Inc. 5465 East Cheryl Parkway, Madison, Wisconsin 53711-5373, USA.

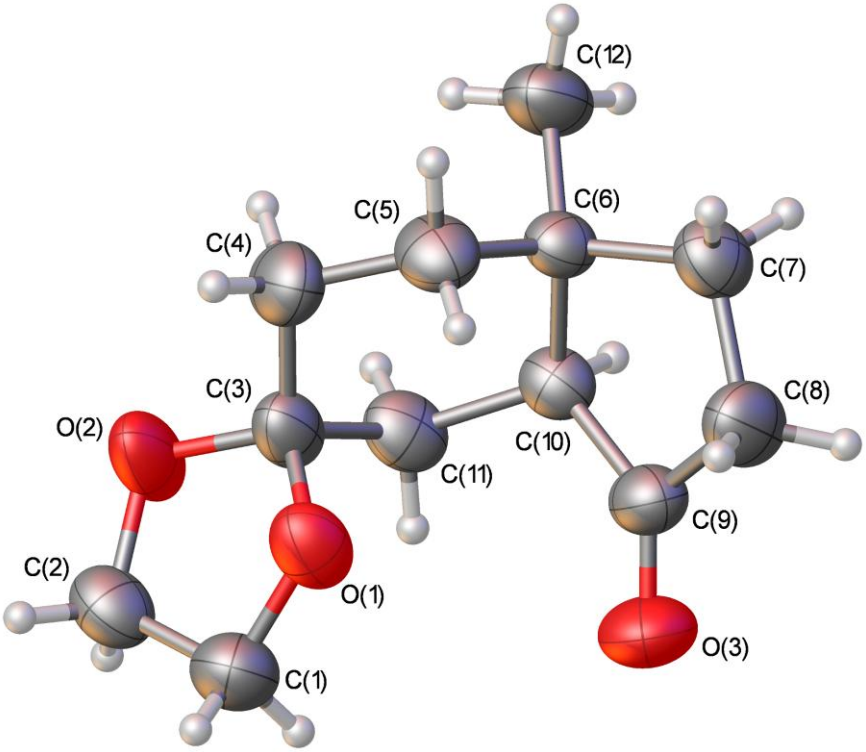
3 Sheldrick, G. M. 2007, *SADABS*, Bruker AXS Inc., Madison, Wisconsin, USA.

4 Sheldrick, G. M. *Acta Cryst.*, 2008, **A64**, 112-122.

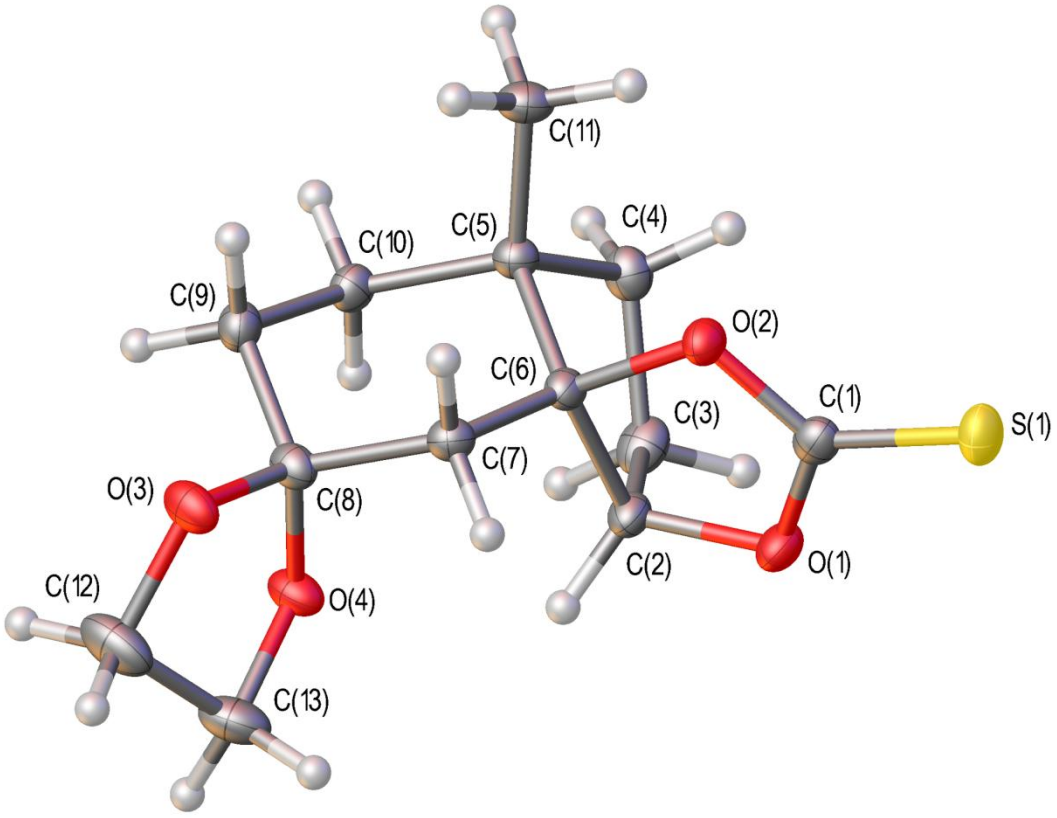
5 *SHELXTL, program suite for structure solution and refinement*, 1997, Bruker AXS, Inc. 5465 East Cheryl Parkway, Madison, Wisconsin 53711-5373, USA.

6 Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. *J. Appl. Crystallogr.*, 2009, **42**, 339-341.

Crystal structure of **6** with ellipsoids drawn at the 50 % probability level (arbitrary atom numbering):

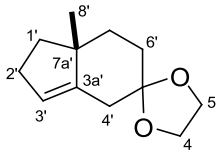


Crystal structure of **10** with ellipsoids drawn at the 50 % probability level (arbitrary atom numbering):

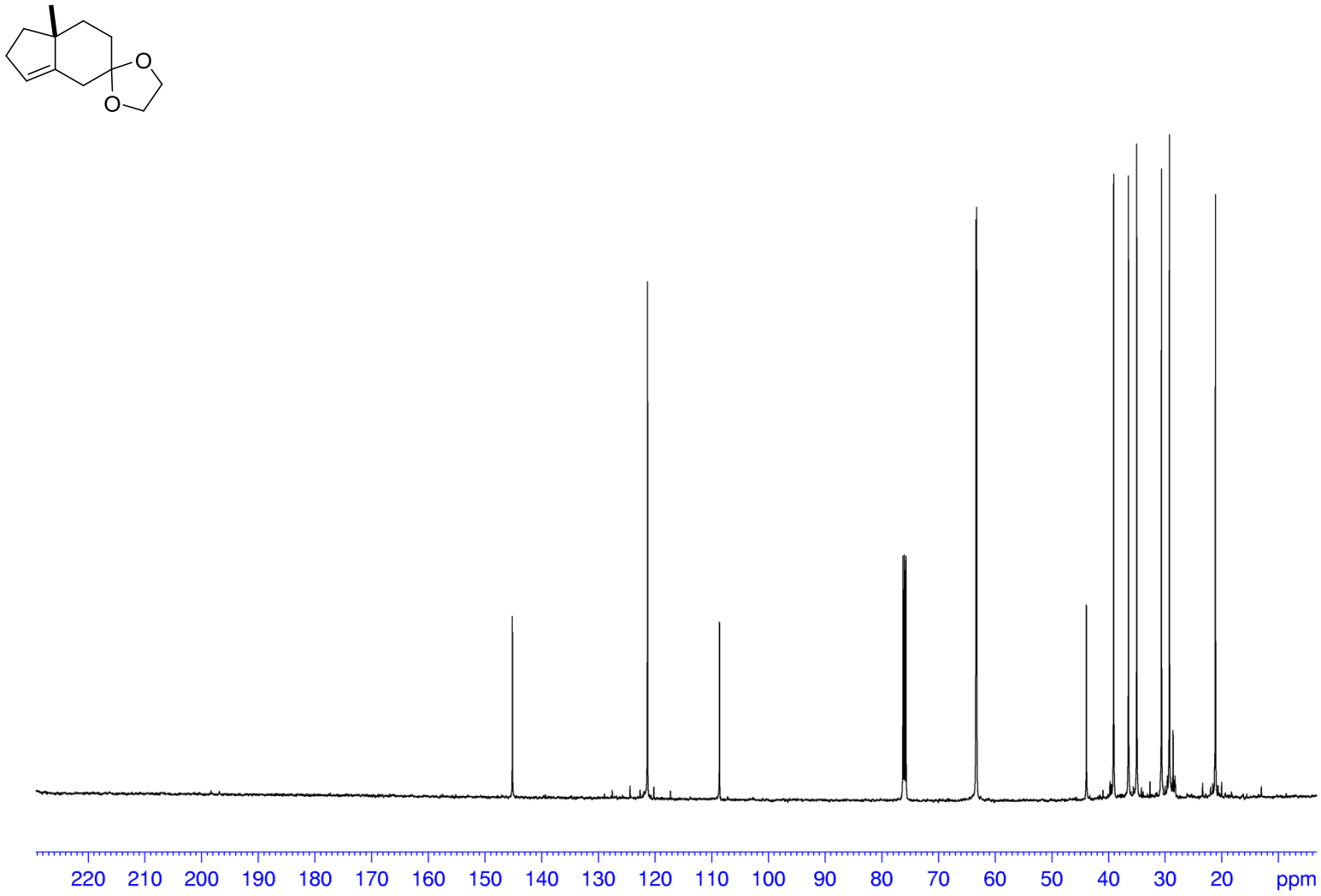


NMR spectra

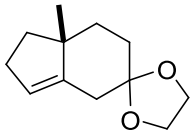
(±)-(*R**)-7a'-Methyl-1',2',4',6',7',7a'-hexahydrospiro[[1,3]dioxolane-2,5'-indene] **3**; $CDCl_3$, 500 MHz.

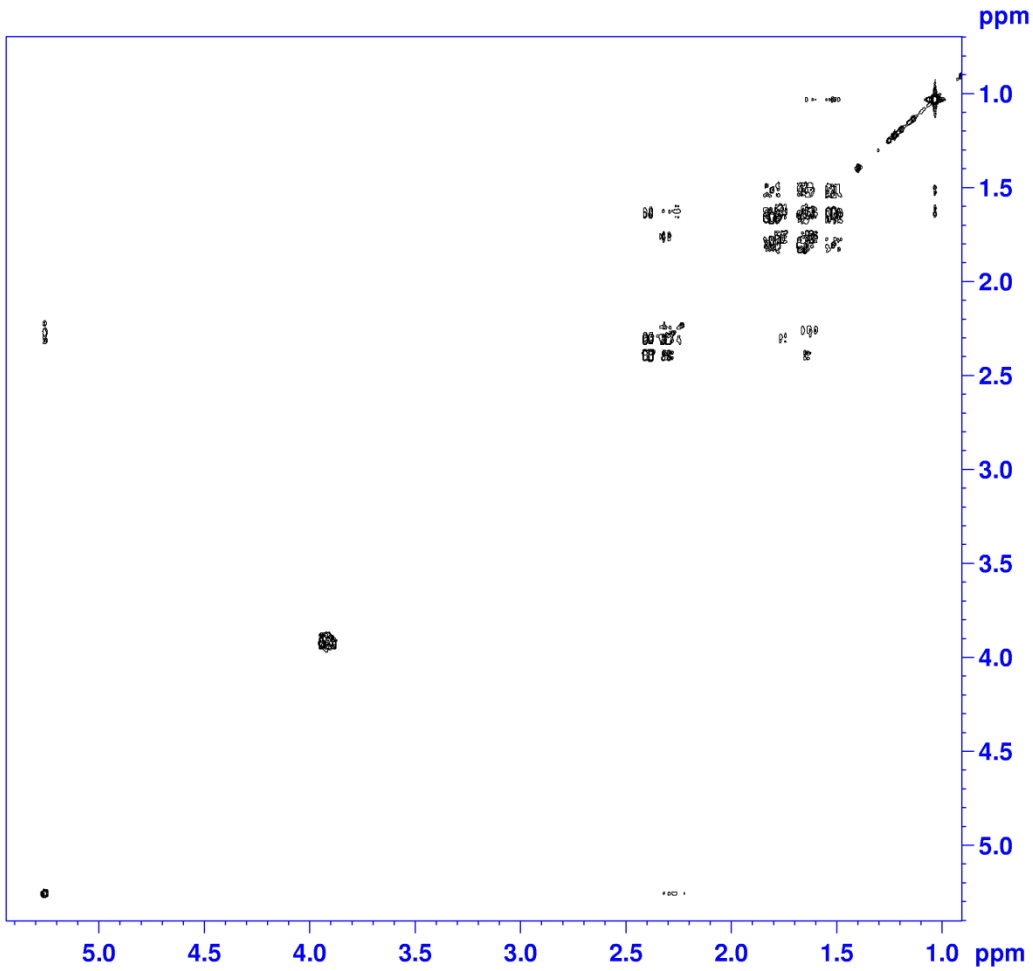


(±)-(*R**)-7a'-Methyl-1',2',4',6',7',7a'-hexahydrospiro[[1,3]dioxolane-2,5'-indene] **3**; $CDCl_3$, 125 MHz.

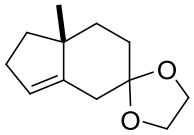


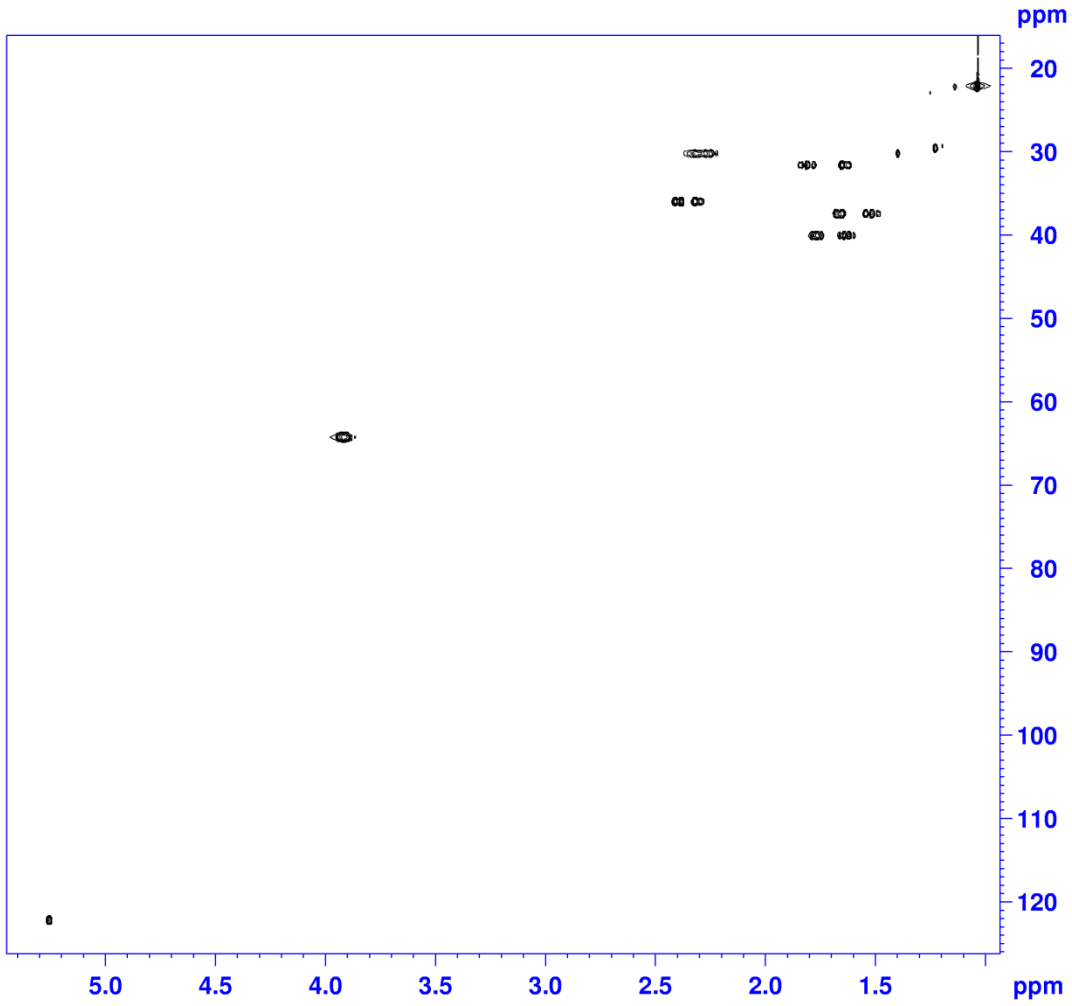
(±)-(*R**)-7a'-Methyl-1',2',4',6',7',7a'-hexahydrospiro[[1,3]dioxolane-2,5'-indene] **3**; $CDCl_3$, 500 MHz.



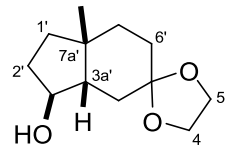


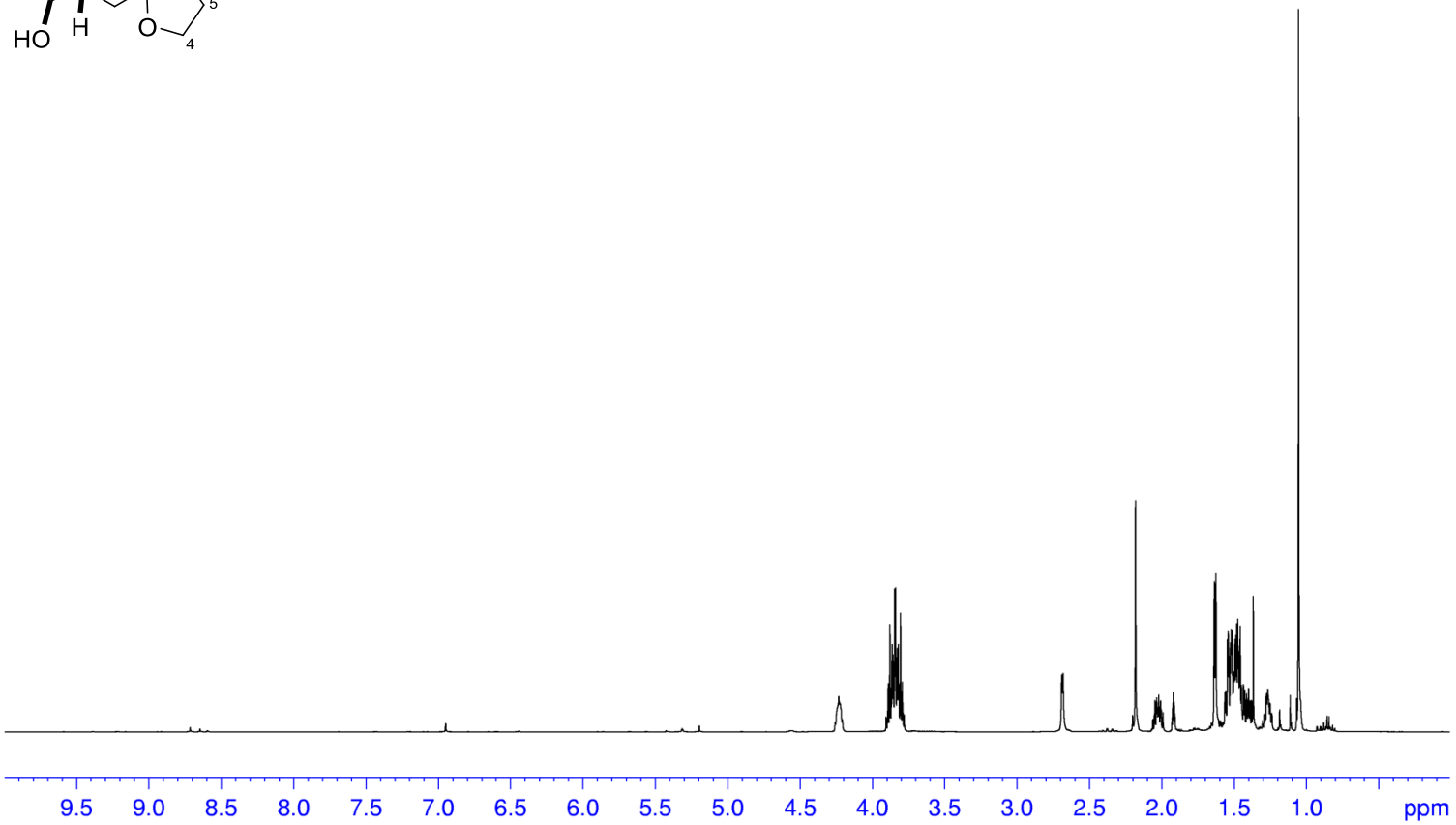
(±)-(*R**)-7a'-Methyl-1',2',4',6',7',7a'-hexahydrospiro[[1,3]dioxolane-2,5'-indene] **3**; $CDCl_3$, 500 MHz/125 MHz.



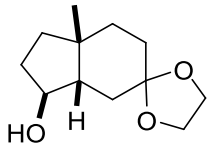


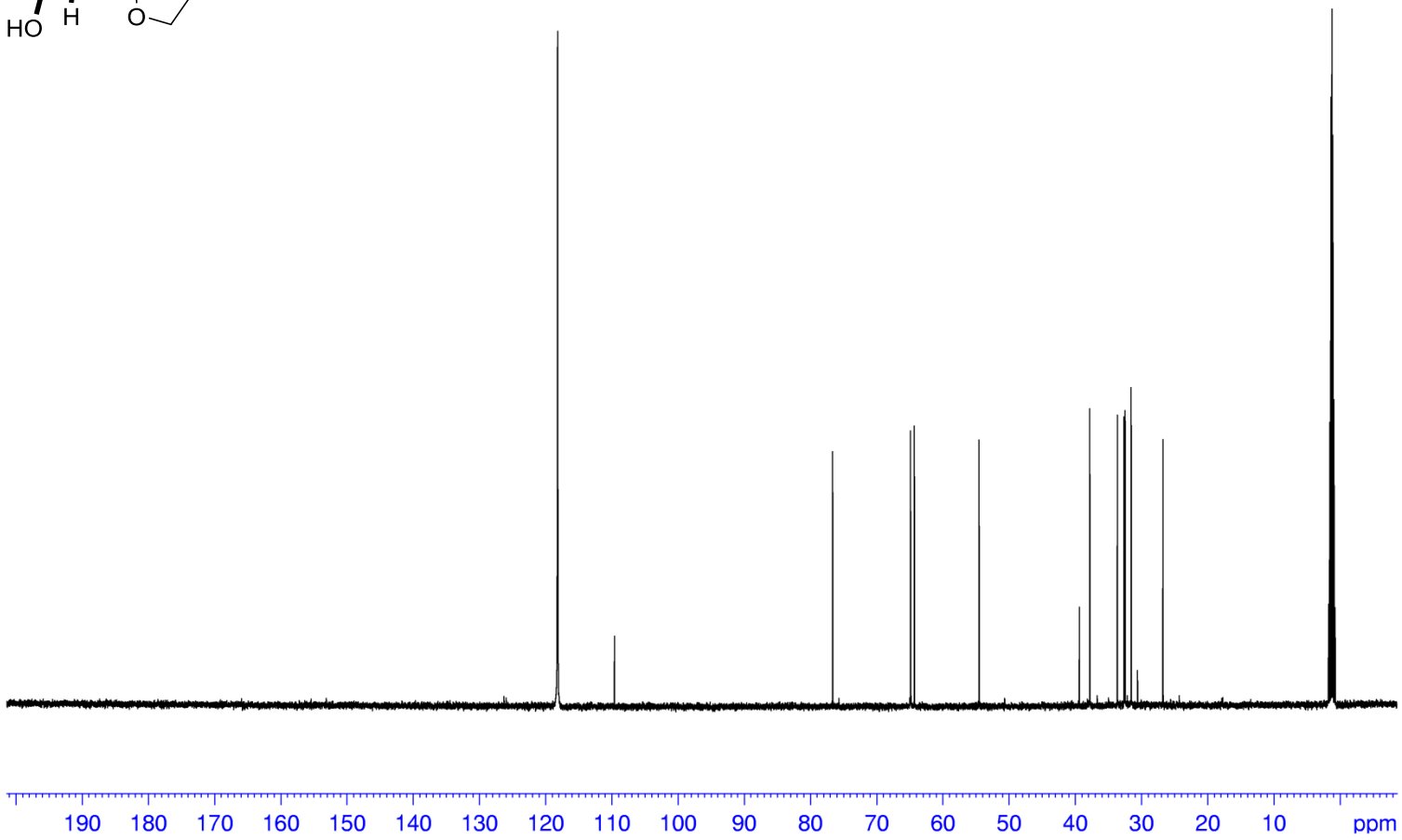
(±)-(3'*S**,3a'*S**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **5**; CD_3CN, 500 MHz.



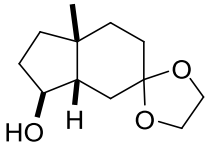


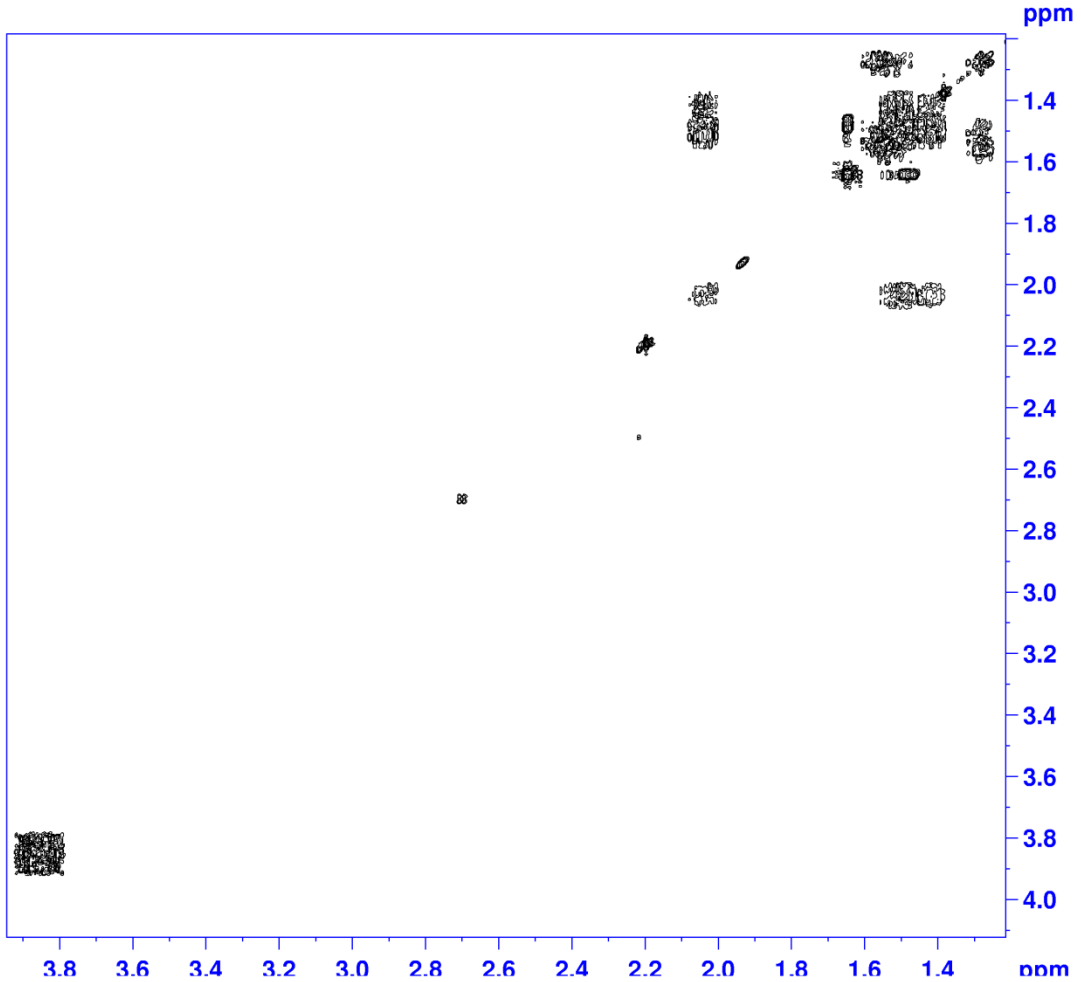
(±)-(3'*S**,3a'*S**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **5**; CD_3CN, 125 MHz.



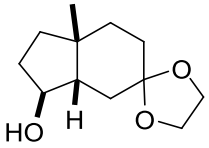


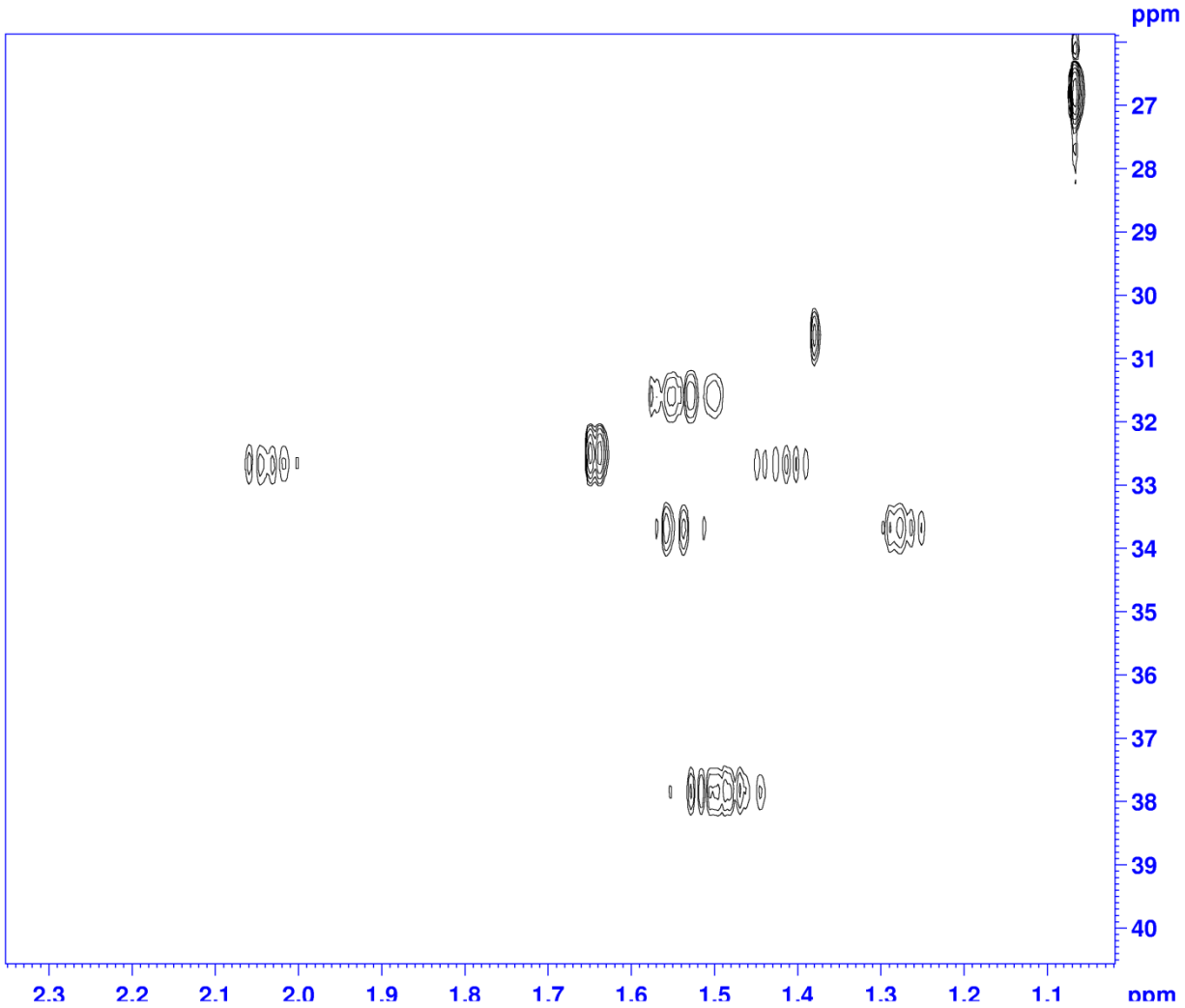
(±)-(3'*S**,3a'*S**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **5**; CD_3CN, 500 MHz.



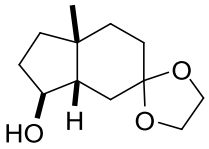


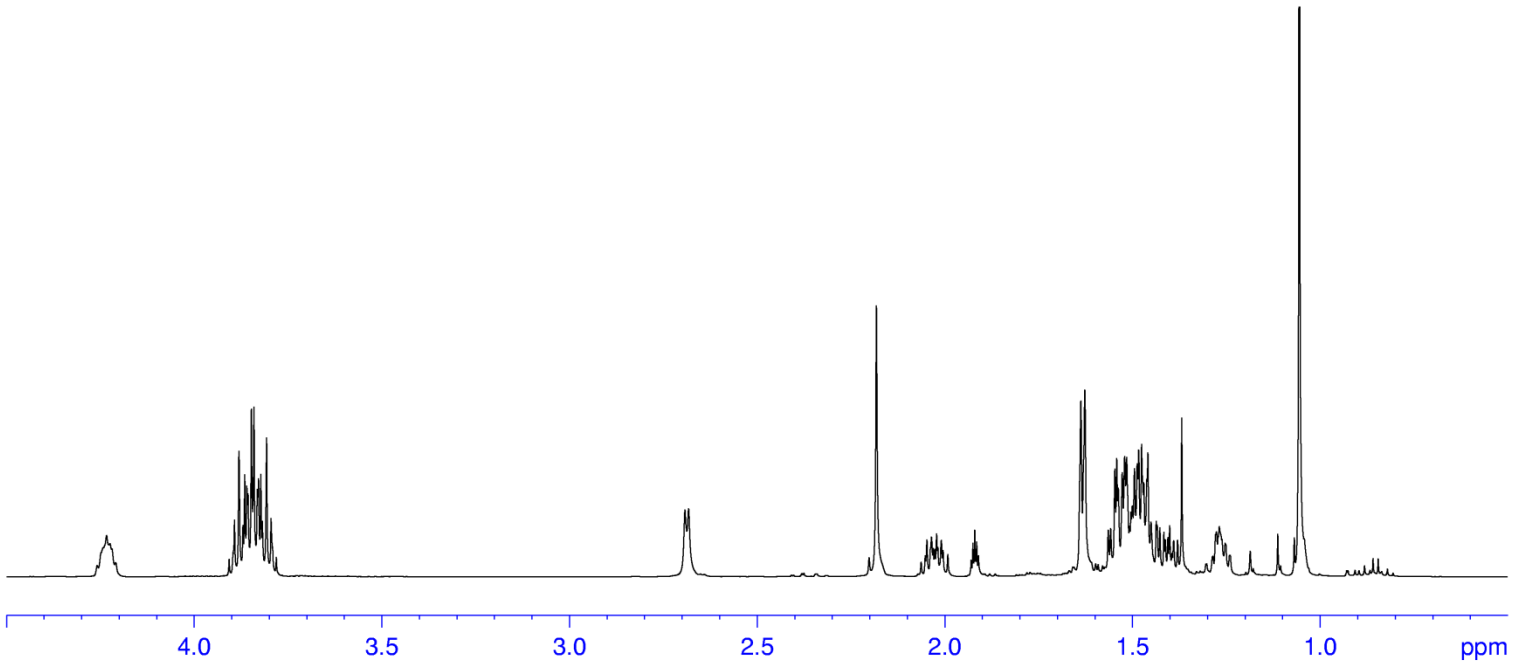
(±)-(3'*S**,3a'*S**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **5**; CD_3CN, 500 MHz/125 MHz.



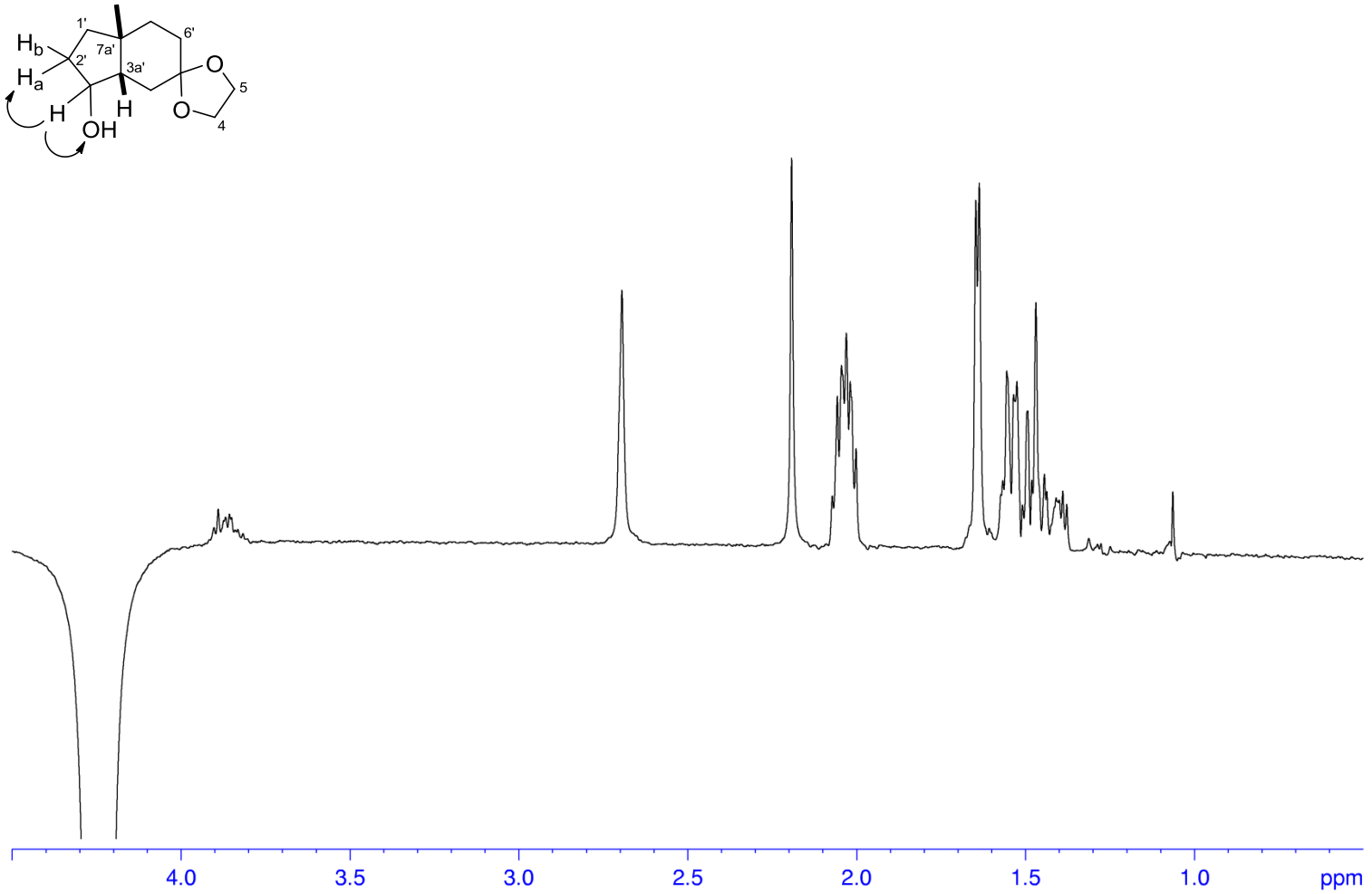


(±)-(3'*S**,3a'*S**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **5**; CD_3CN, 500 MHz.

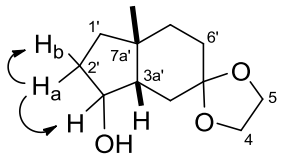


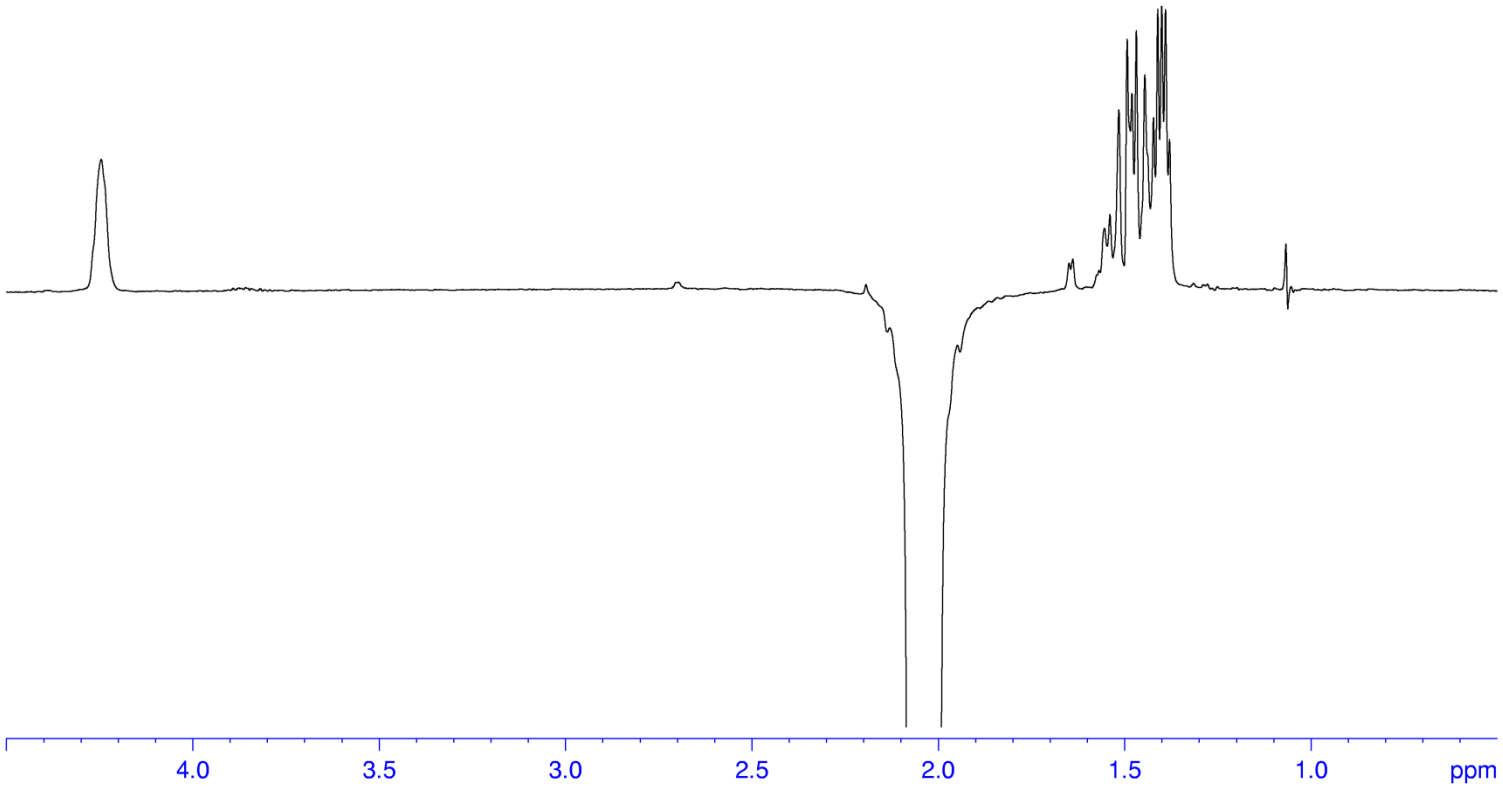


(±)-(3'*S**,3a'*S**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **5**; CD_3CN, 500 MHz; nOe irradiation of H-3'.

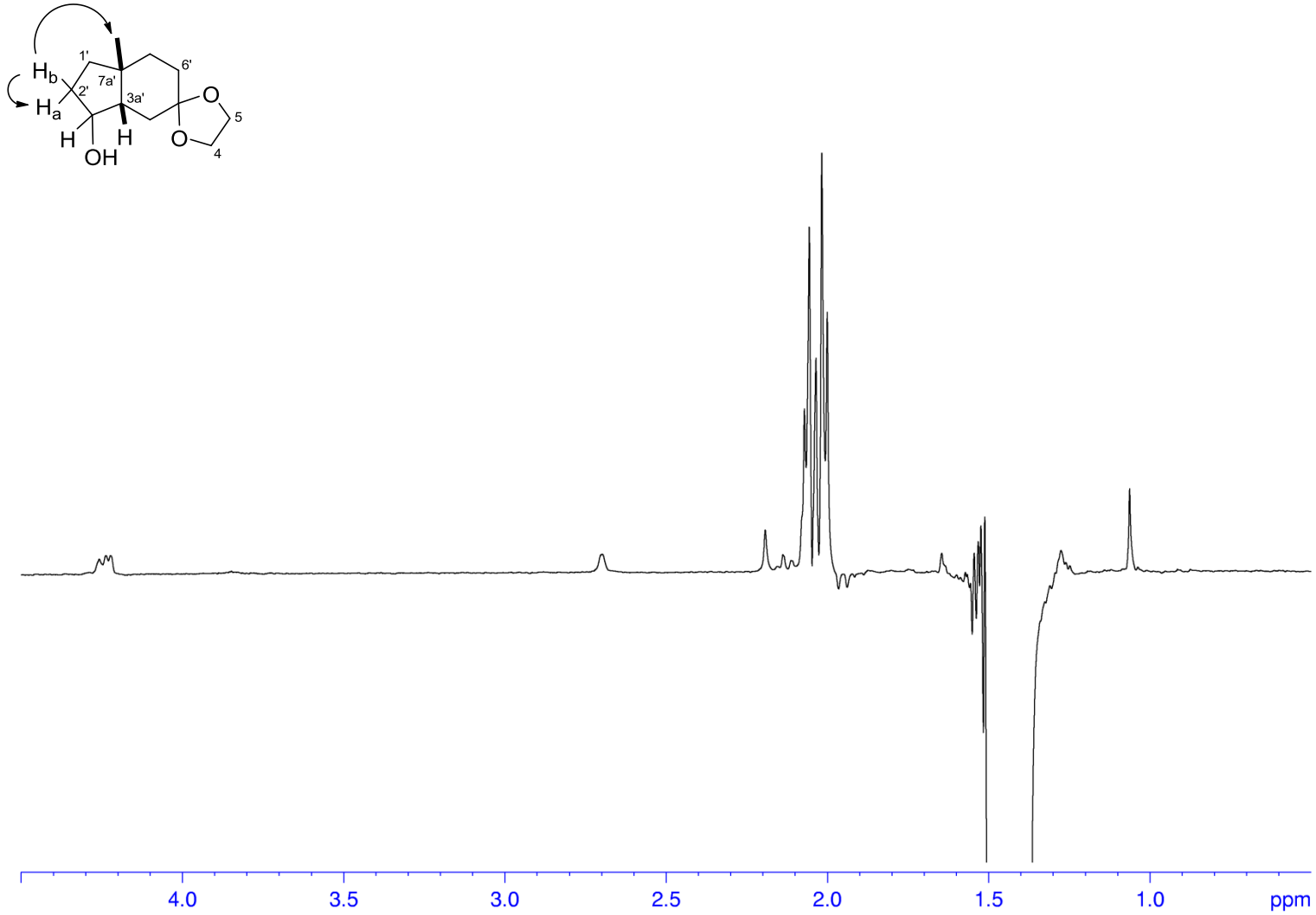


(±)-(3'*S**,3a'*S**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **5**; CD_3CN, 500 MHz; nOe irradiation of H-2_a.





(±)-(3'*S**,3a'*S**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **5**; CD_3CN, 500 MHz; nOe irradiation of H-2_b.



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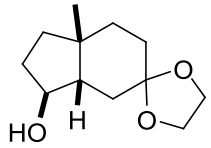
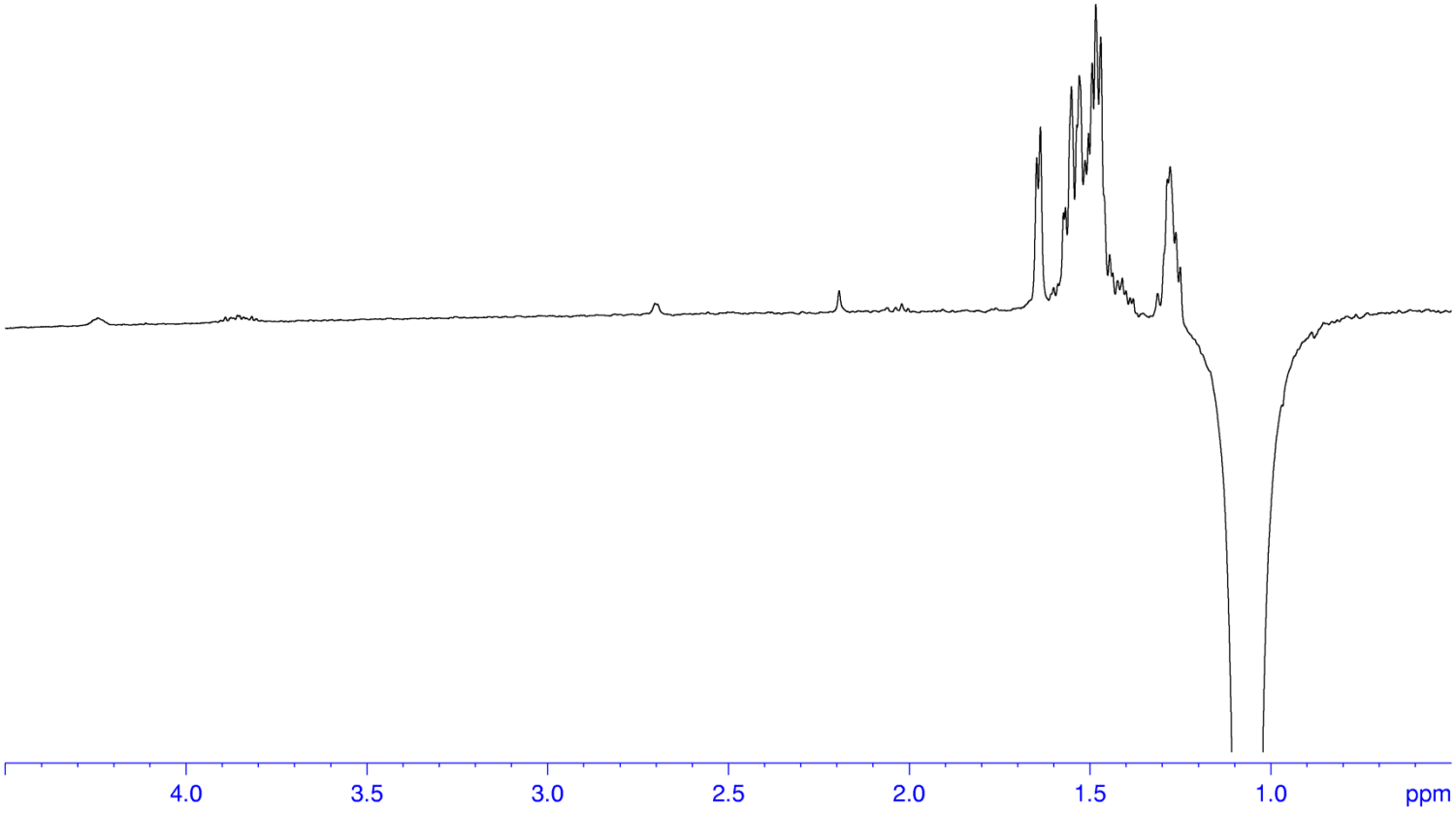
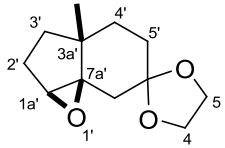




(±)-(1a'*S**,3a'*R**,7a'*R**)-3a'-Methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **7**; $CDCl_3$, 500 MHz.



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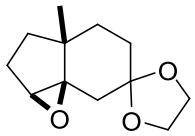
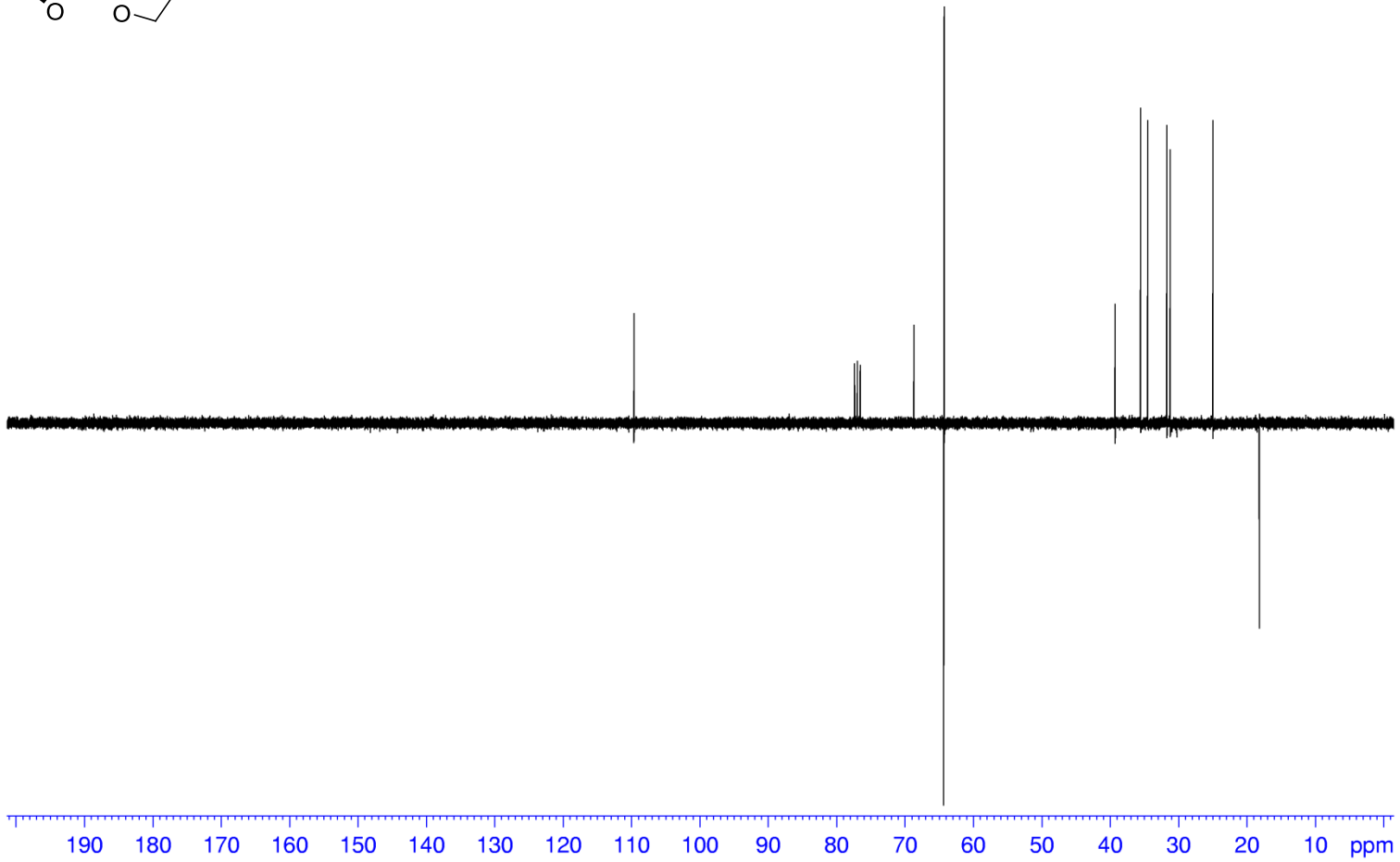
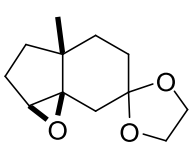
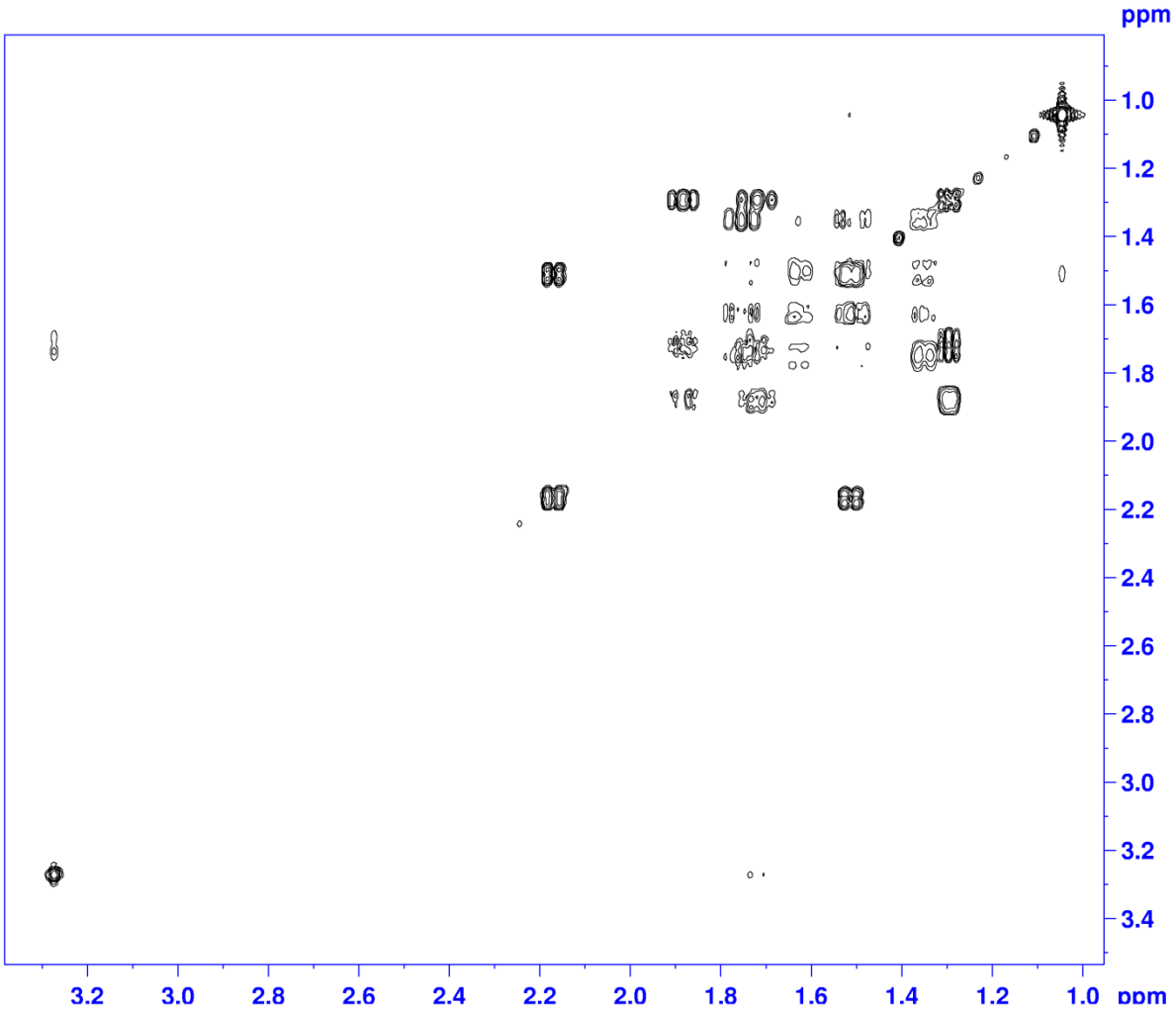


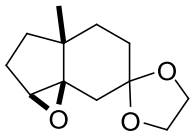


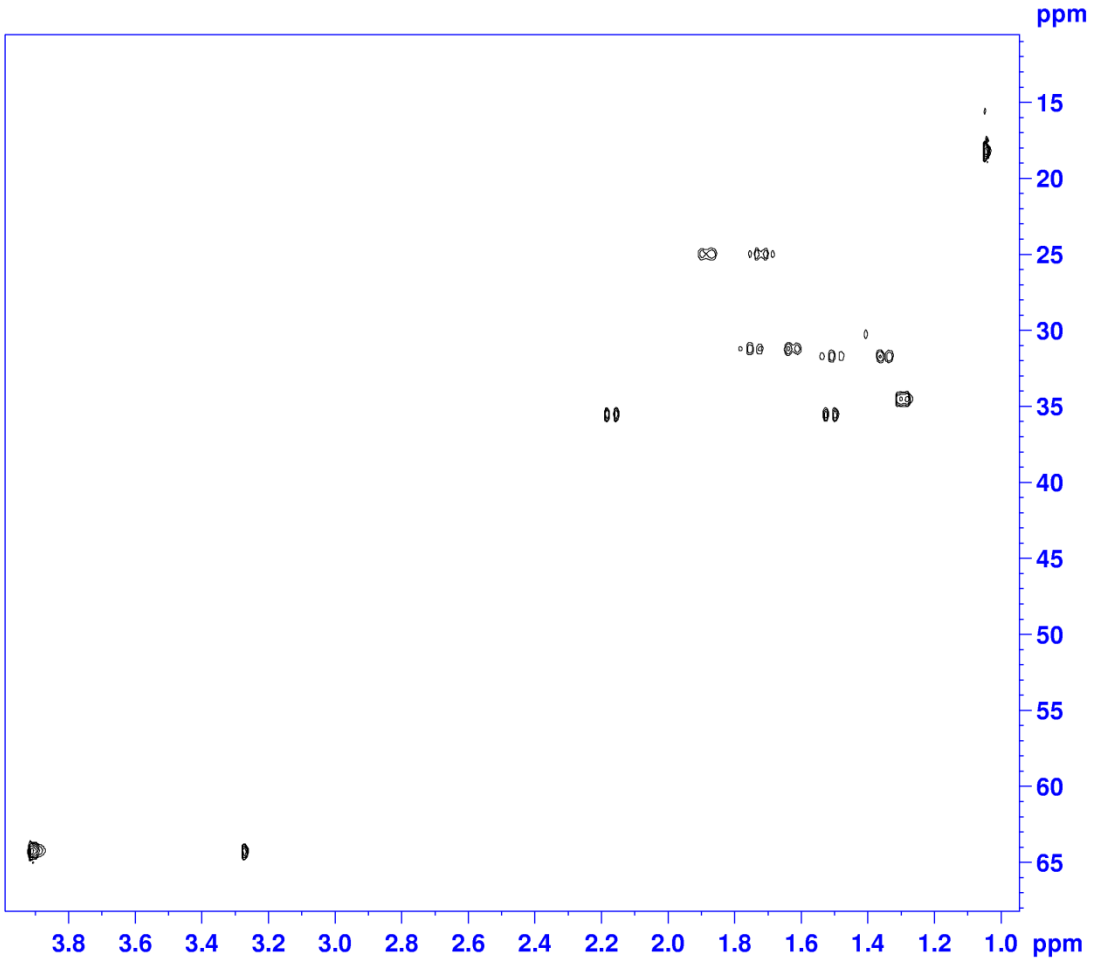
(±)-(1a'*S**,3a'*R**,7a'*R**)-3a'-Methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **7**; $CDCl_3$, 500 MHz.



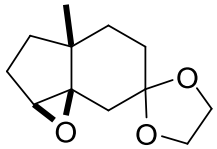


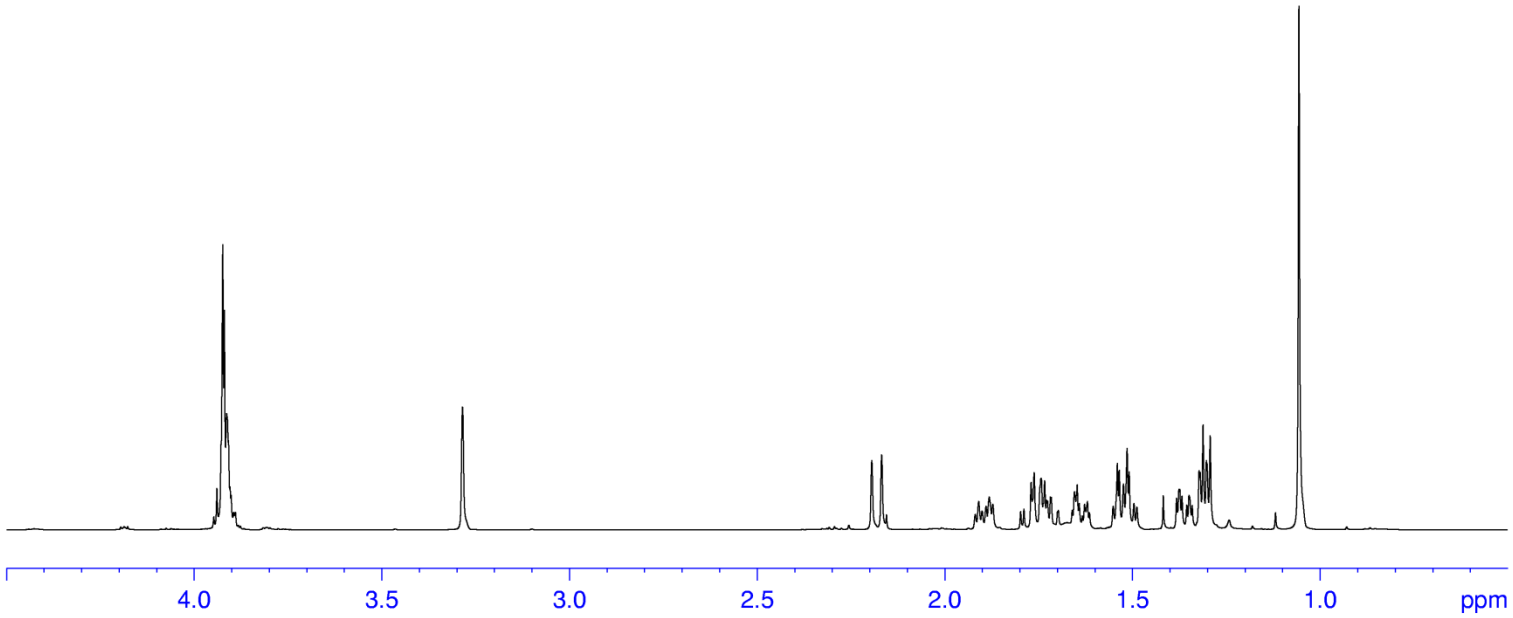
(±)-(1a'*S**,3a'*R**,7a'*R**)-3a'-Methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **7**; $CDCl_3$, 500 MHz/125 MHz.



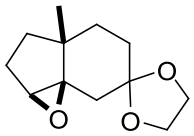


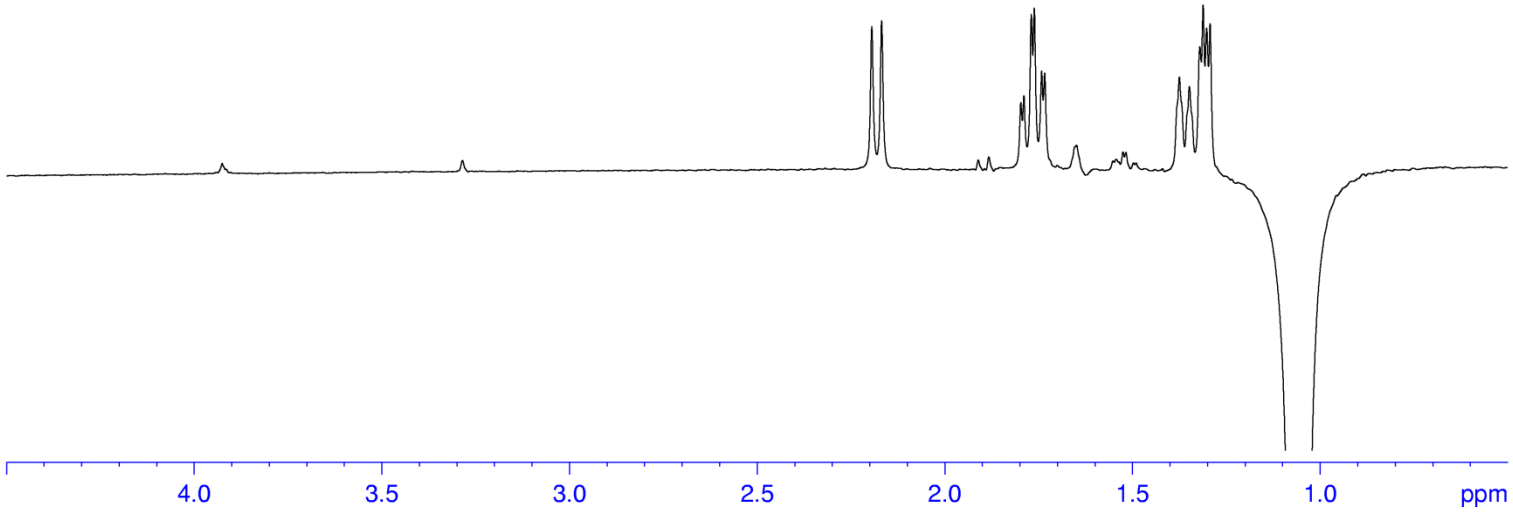
(±)-(1a'*S**,3a'*R**,7a'*R**)-3a'-Methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **7**; $CDCl_3$, 500 MHz.



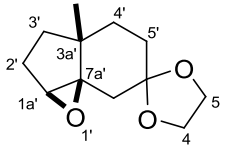


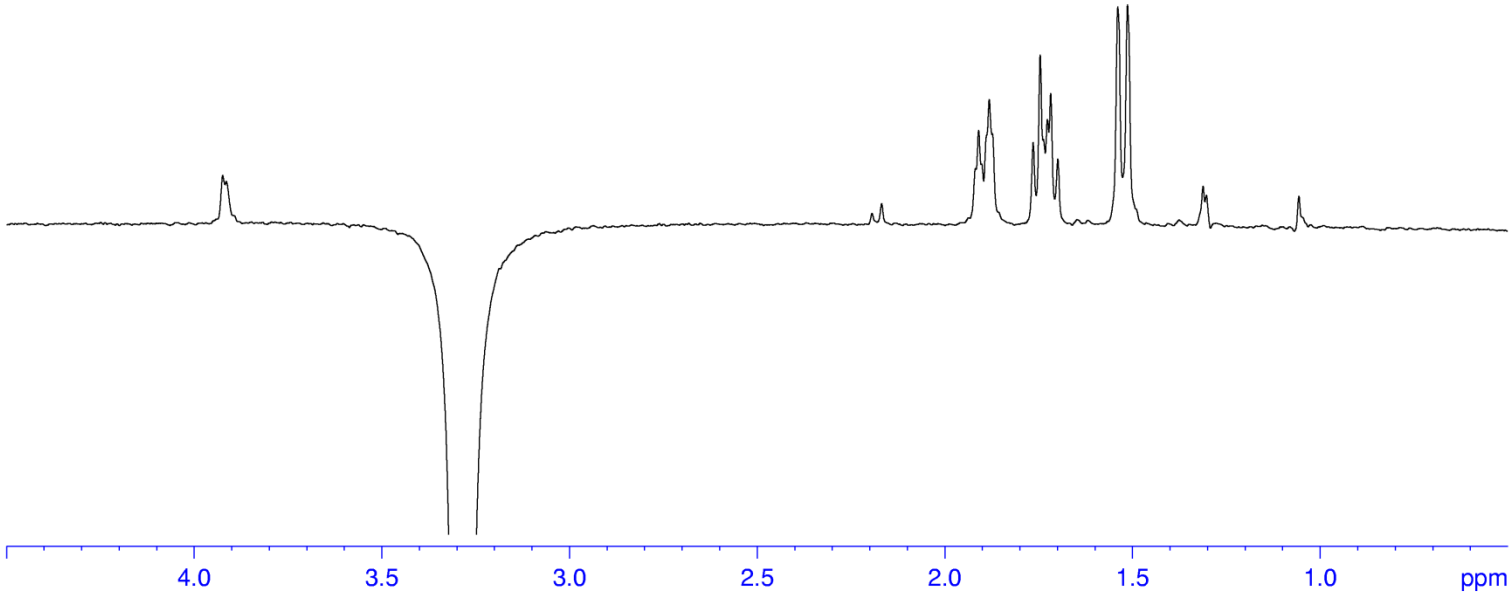
(±)-(1a'*S**,3a'*R**,7a'*R**)-3a'-Methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **7**; $CDCl_3$, 500 MHz; nOe irradiation of methyl group protons.



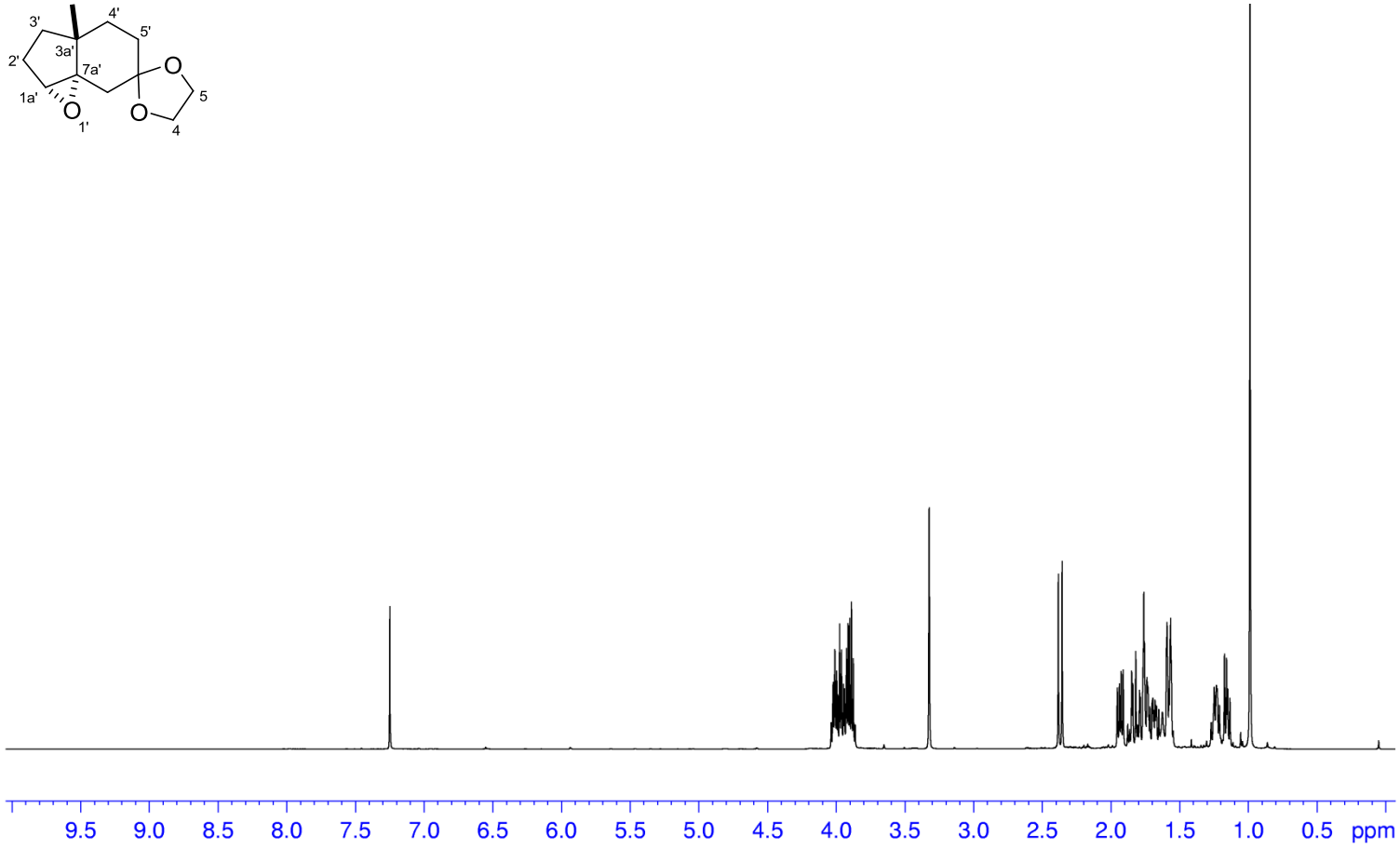


(±)-(1a'*S**,3a'*R**,7a'*R**)-3a'-Methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **7**; $CDCl_3$, 500 MHz; nOe irradiation of H-1a'.

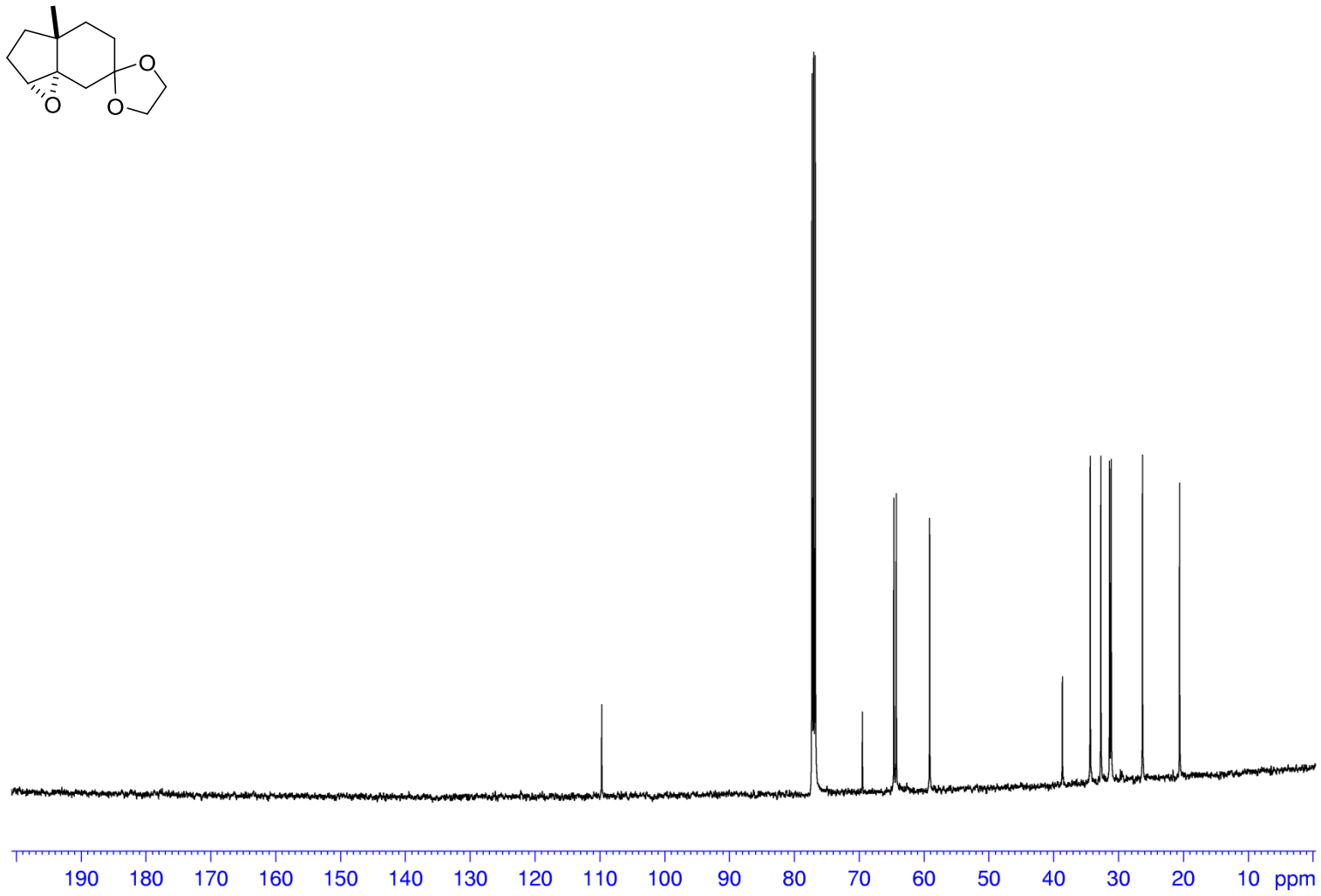




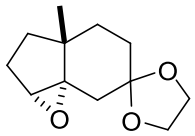
(±)-(1a'*R**,3a'*R**,7a'*S**)-3a'-methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **8**; $CDCl_3$, 500 MHz.

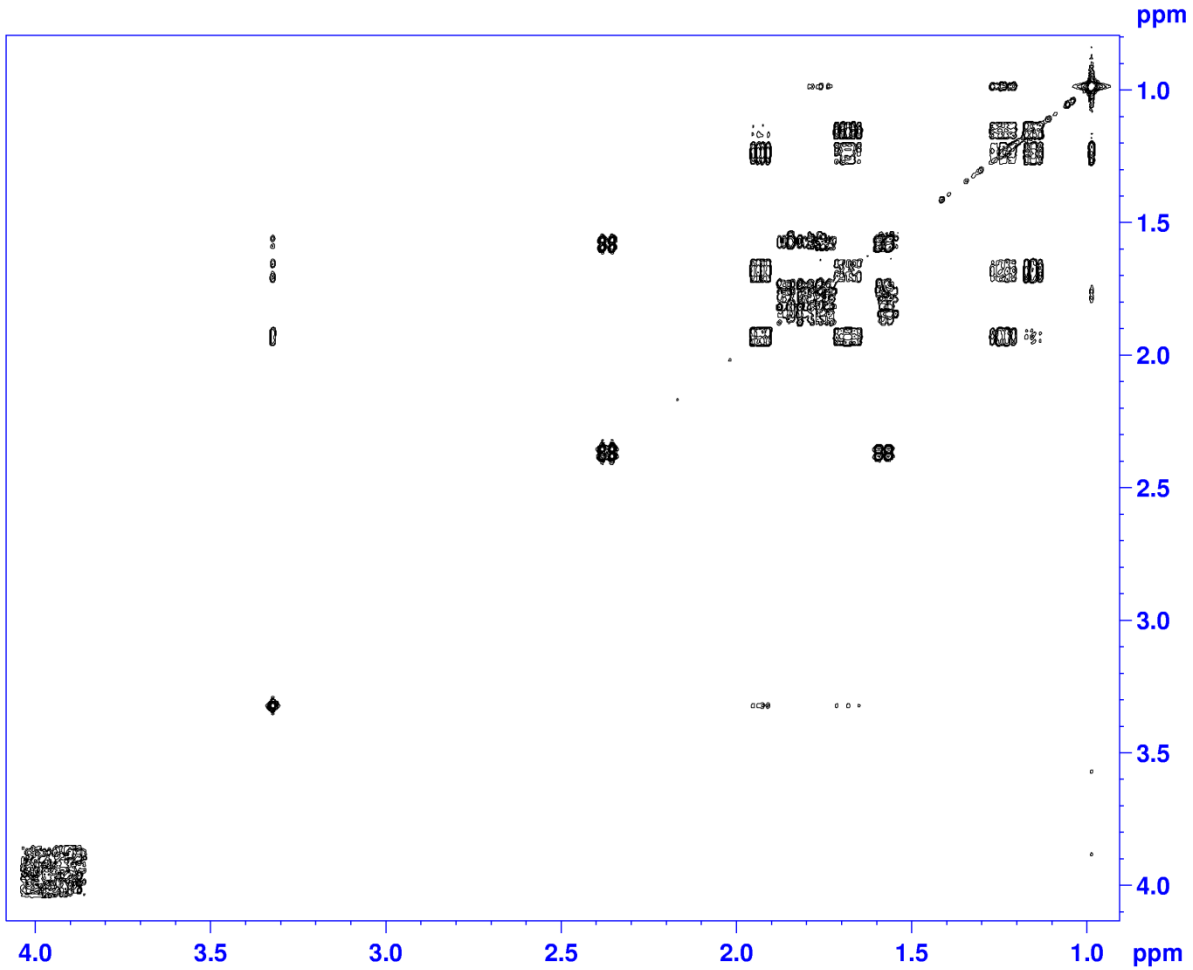


(±)-(1a'*R**,3a'*R**,7a'*S**)-3a'-methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **8**; $CDCl_3$, 125 MHz.

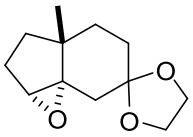


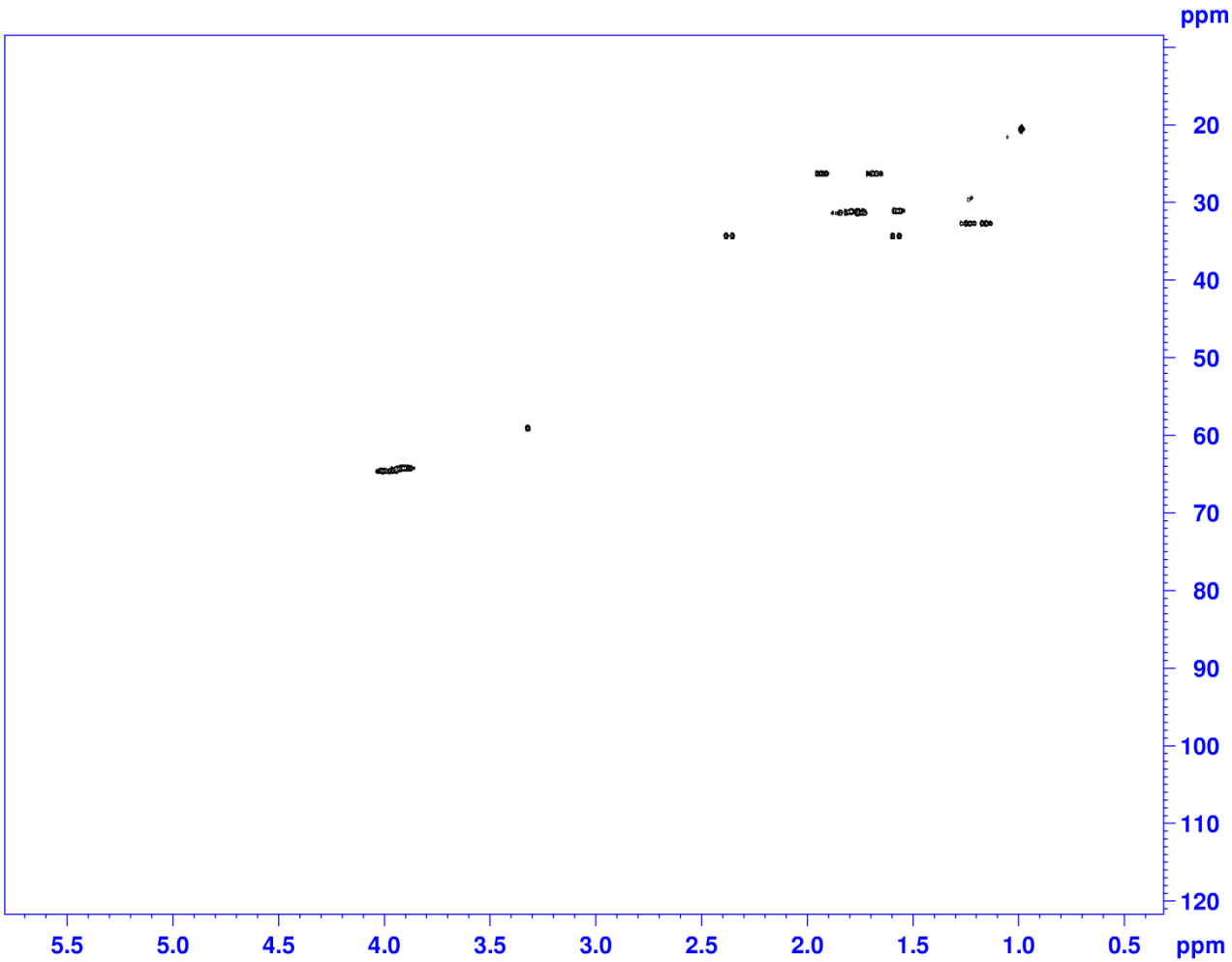
(±)-(1a'*R**,3a'*R**,7a'*S**)-3a'-methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **8**; $CDCl_3$, 500 MHz.



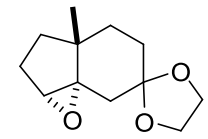


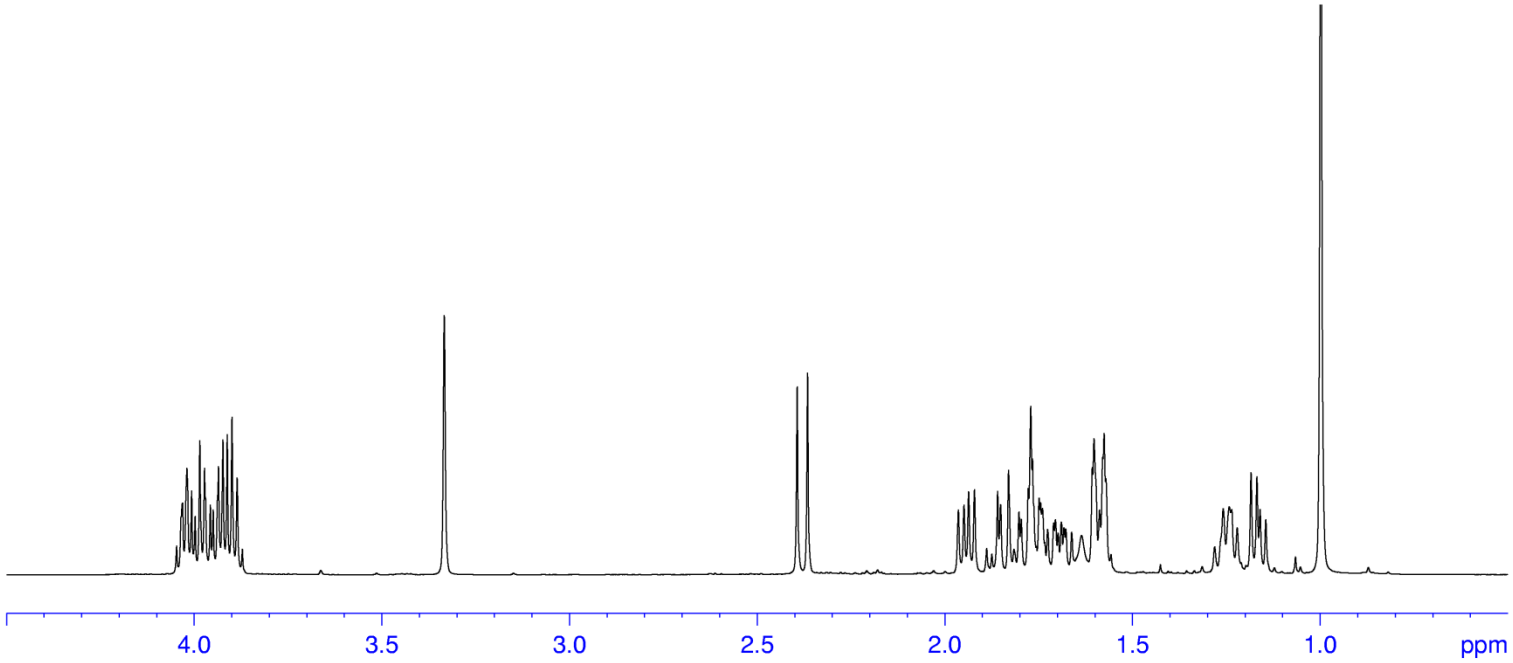
(±)-(1a'*R**,3a'*R**,7a'*S**)-3a'-methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **8**; $CDCl_3$, 500 MHz/125 MHz.



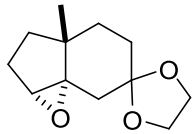


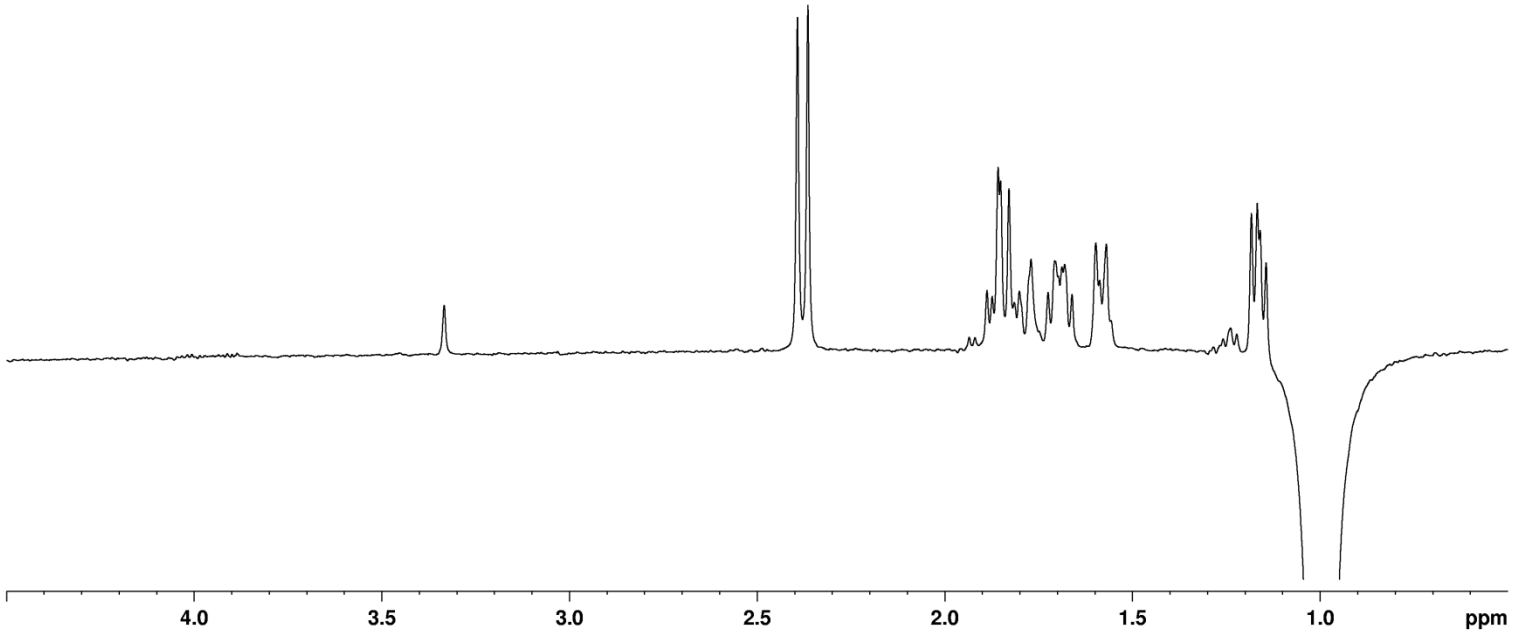
(±)-(1a'*R**,3a'*R**,7a'*S**)-3a'-methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **8**; $CDCl_3$, 500 MHz.



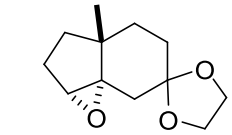


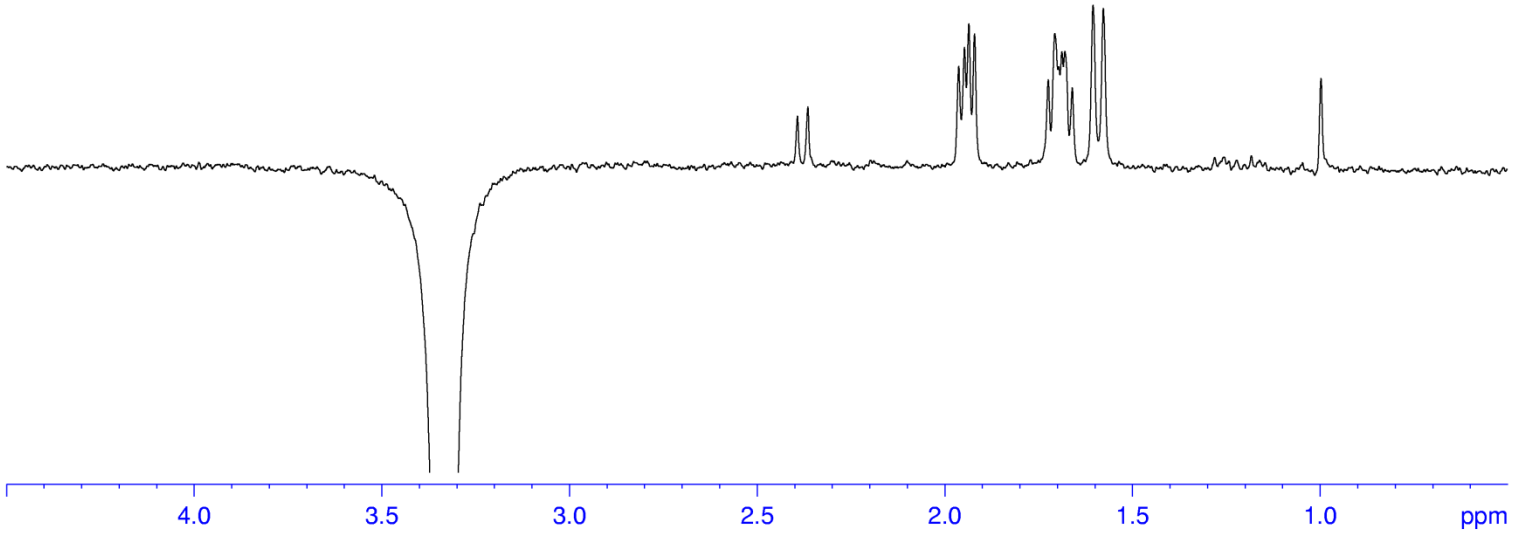
(±)-(1a'*R**,3a'*R**,7a'*S**)-3a'-methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **8**; $CDCl_3$, 500 MHz; nOe irradiation of methyl group protons.



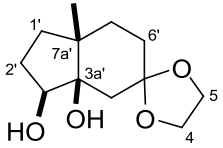


(±)-(1a'*R**,3a'*R**,7a'*S**)-3a'-methylhexahydro-1a'*H*-spiro[[1,3]dioxolane-2,6'-indeno[1,7a-*b*]oxirene] **8**; $CDCl_3$, 500 MHz; nOe irradiation of H-1a'.

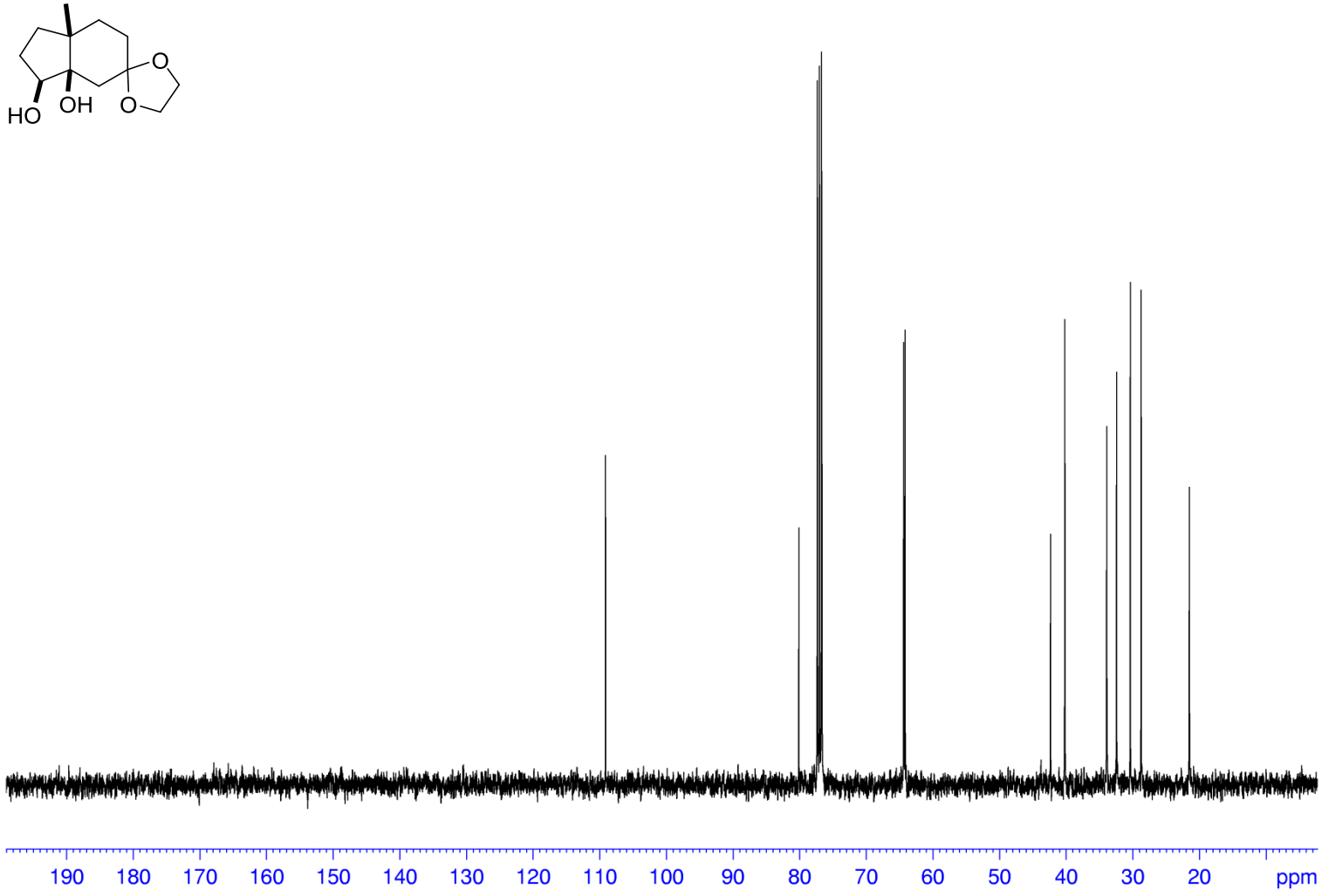




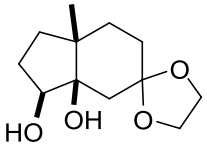
(±)-(3'*S**,3a'*R**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-indene]-3',3a'-diol **9**; $CDCl_3$, 400 MHz.

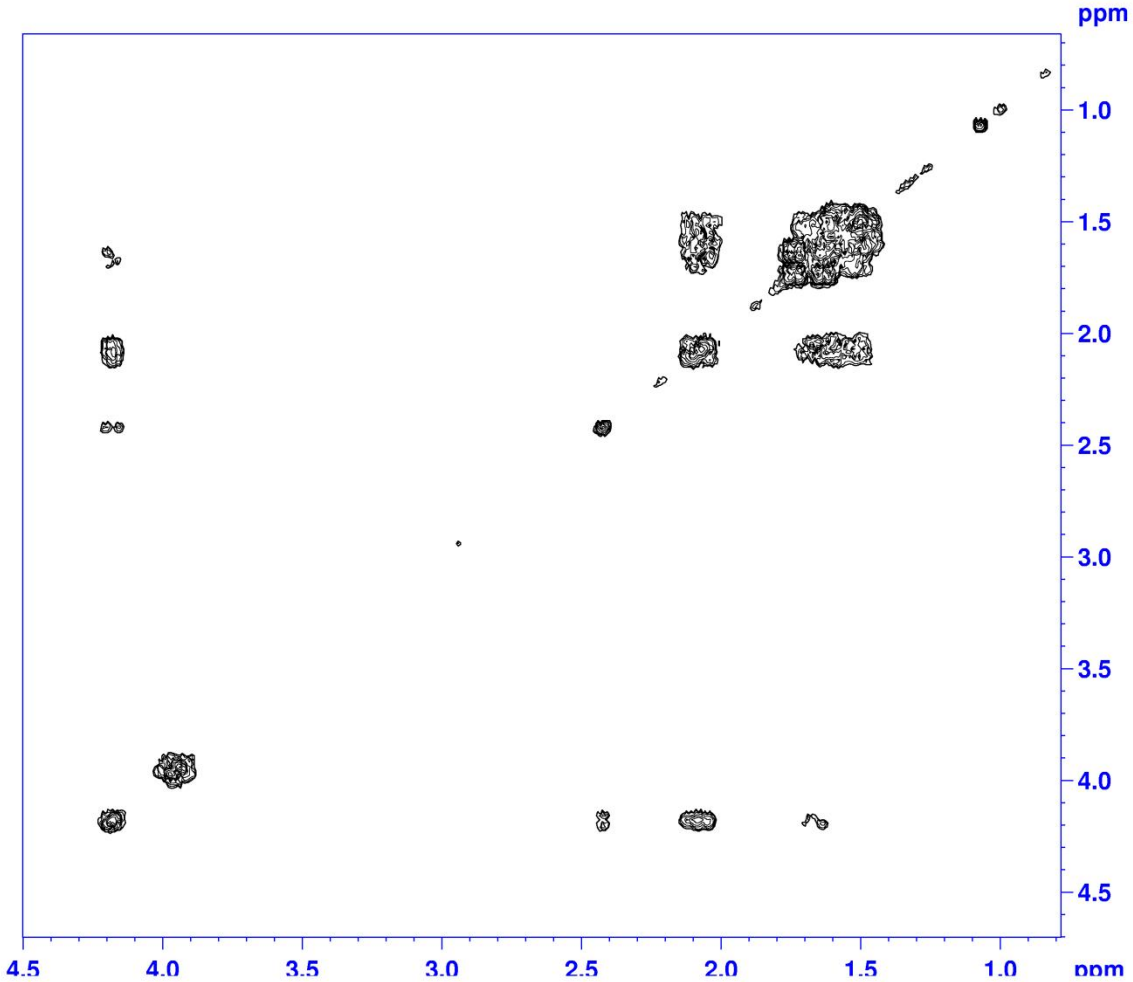


(±)-(3'*S**,3a'*R**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-indene]-3',3a'-diol **9**; $CDCl_3$, 100 MHz.

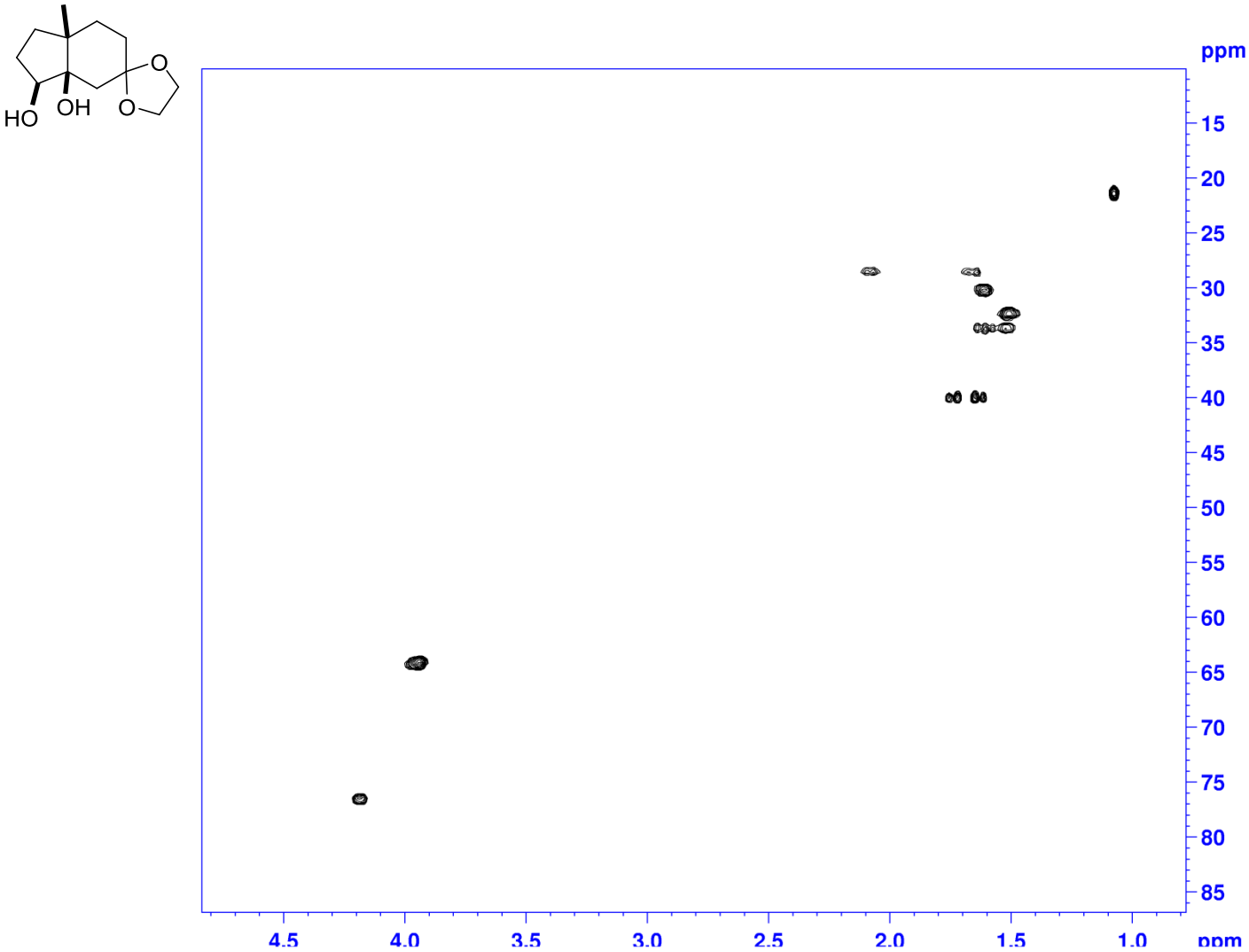


(±)-(3'*S**,3a'*R**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-indene]-3',3a'-diol **9**; $CDCl_3$, 400 MHz.

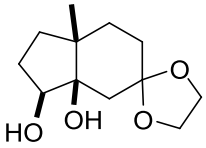


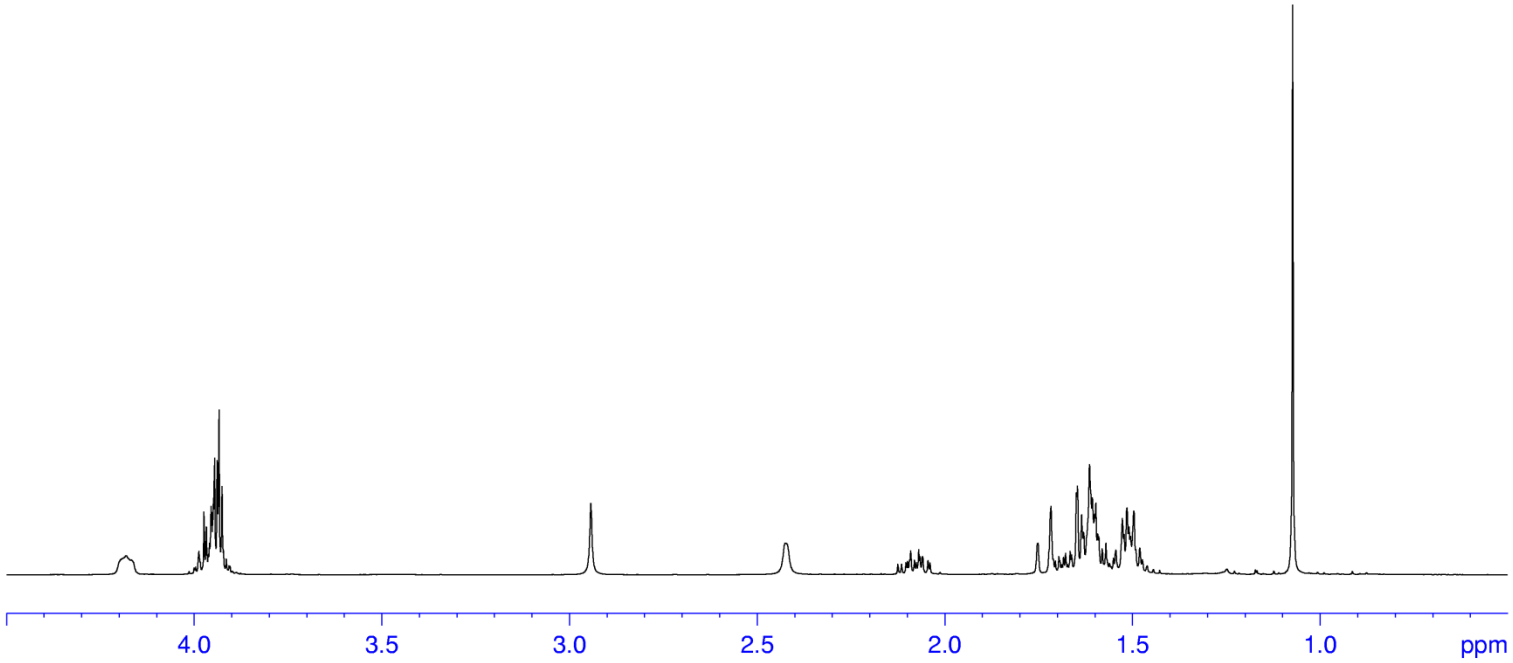


(±)-(3'*S**,3a'*R**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-indene]-3',3a'-diol **9**; $CDCl_3$, 400 MHz/100 MHz.

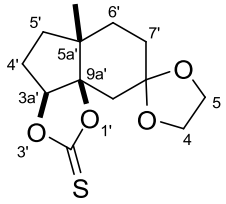


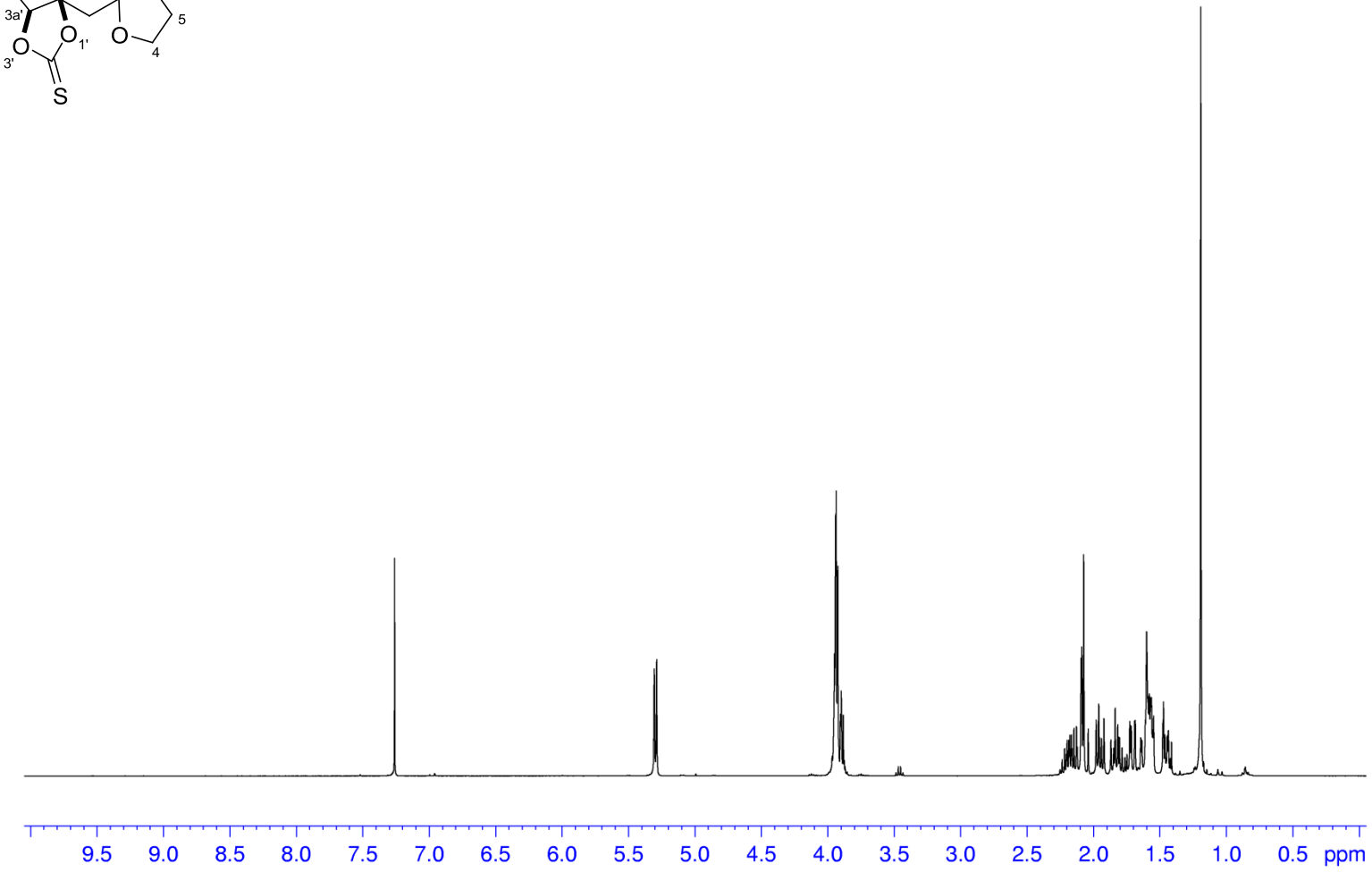
(±)-(3'*S**,3a'*R**,7a'*R**)-7a'-Methyloctahydrospiro[[1,3]dioxolane-2,5'-indene]-3',3a'-diol **9**; $CDCl_3$, 400 MHz.



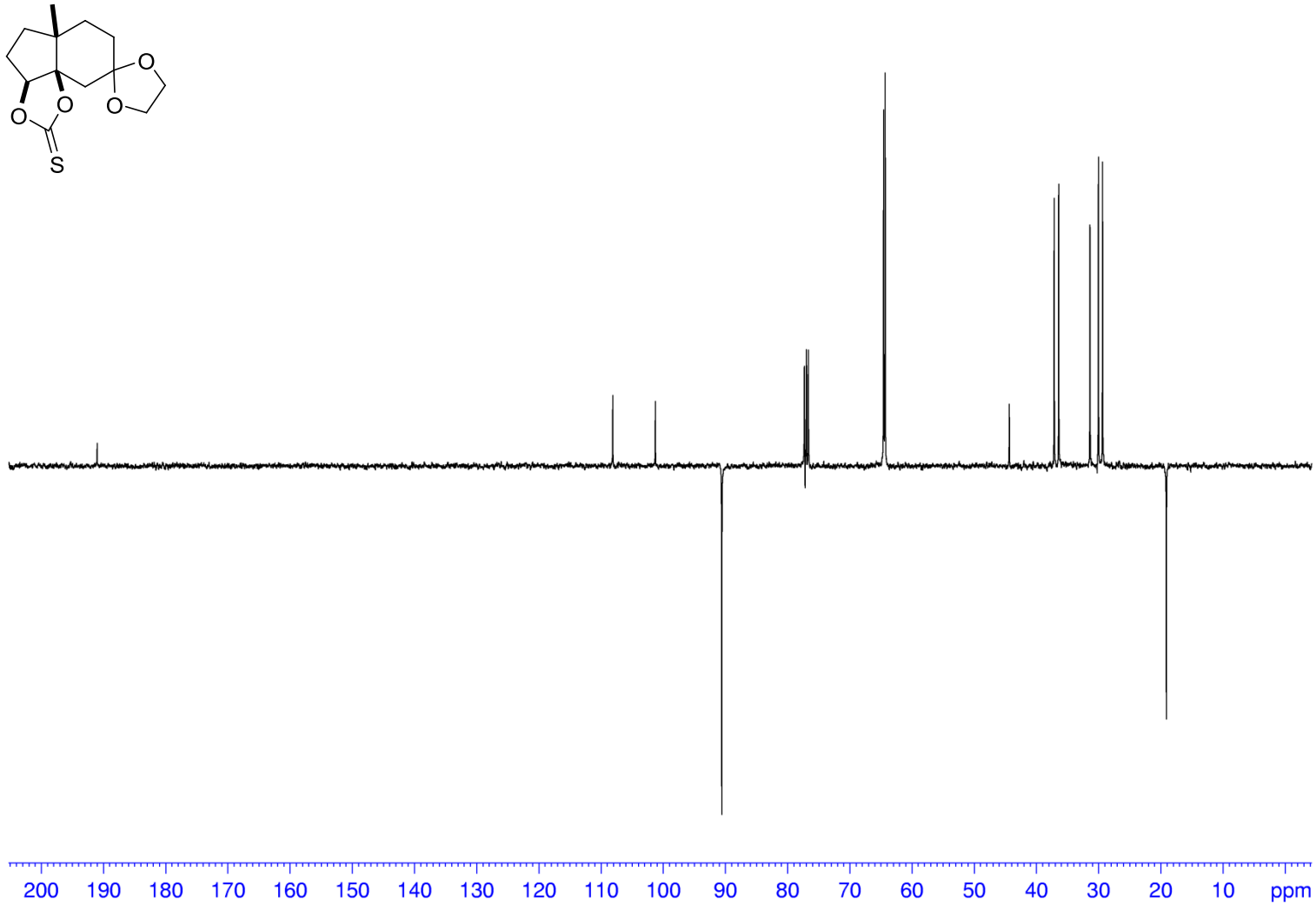


(±)-(3a'*S**,5a'*R**,9a'*R**)-5a'-Methylhexahydro-3a'*H*-spiro[[1,3]dioxolane-2,8'-indeno[1,7a-*d*][1,3]dioxole]-2'-thione **10**; $CDCl_3$, 400 MHz.

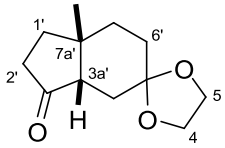


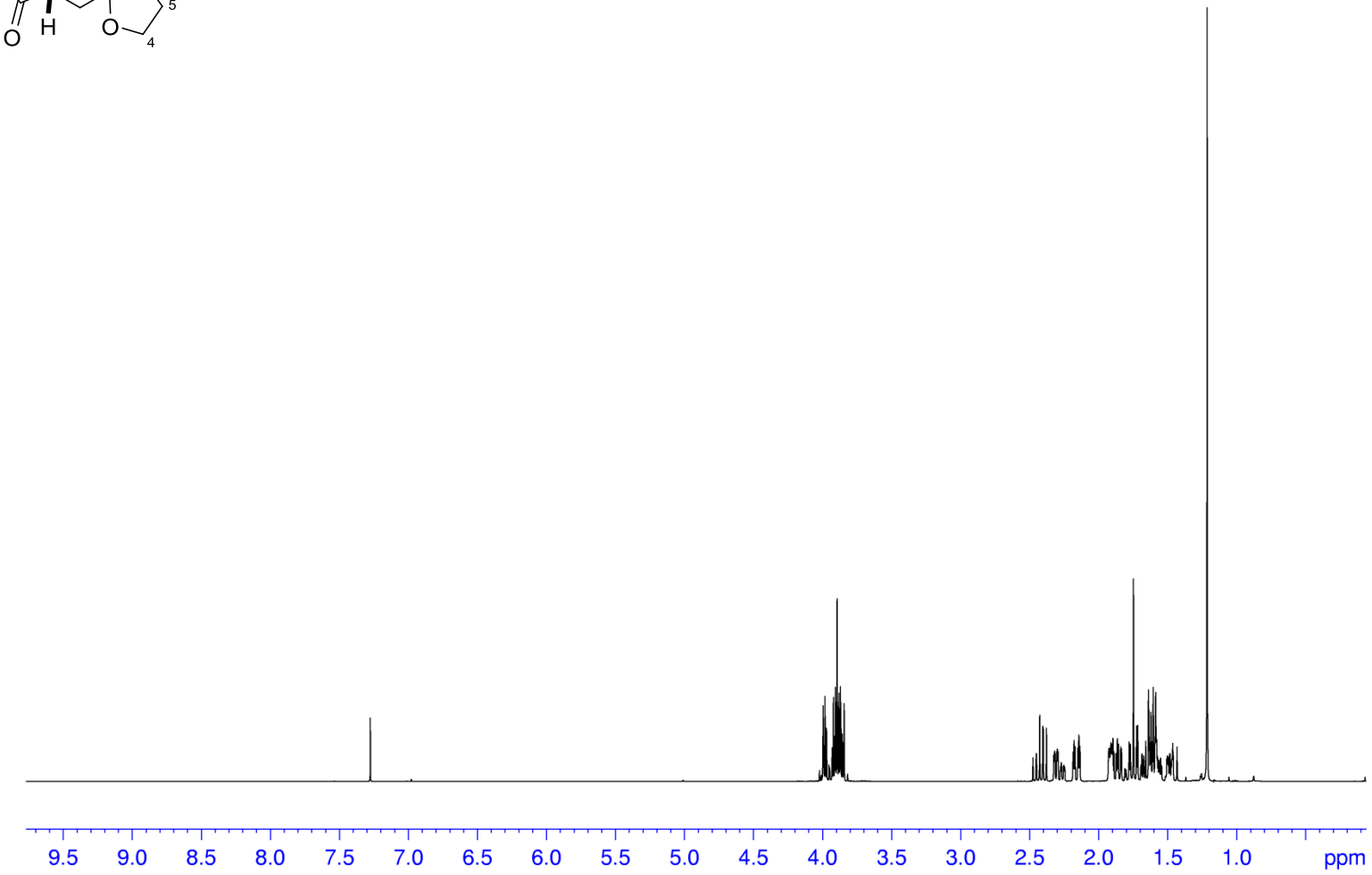


(±)-(3a'*S**,5a'*R**,9a'*R**)-5a'-Methylhexahydro-3a'*H*-spiro[[1,3]dioxolane-2,8'-indeno[1,7a-*d*][1,3]dioxole]-2'-thione **10**; $CDCl_3$, 100 MHz.

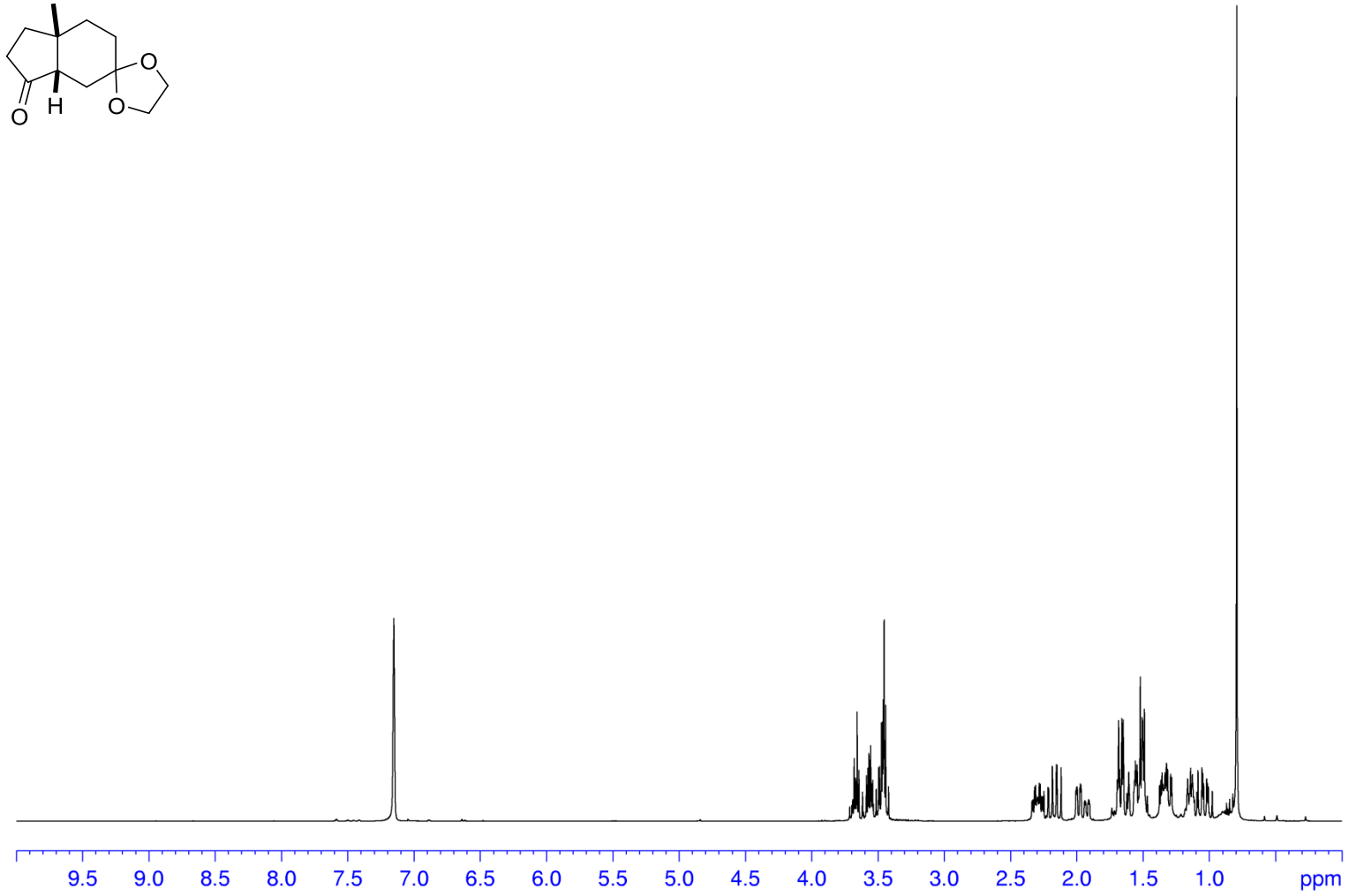


(±)-(3a'*S**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **6** (synthesised *via* Pinacol rearrangement); $CDCl_3$, 400 MHz.

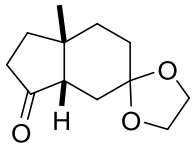


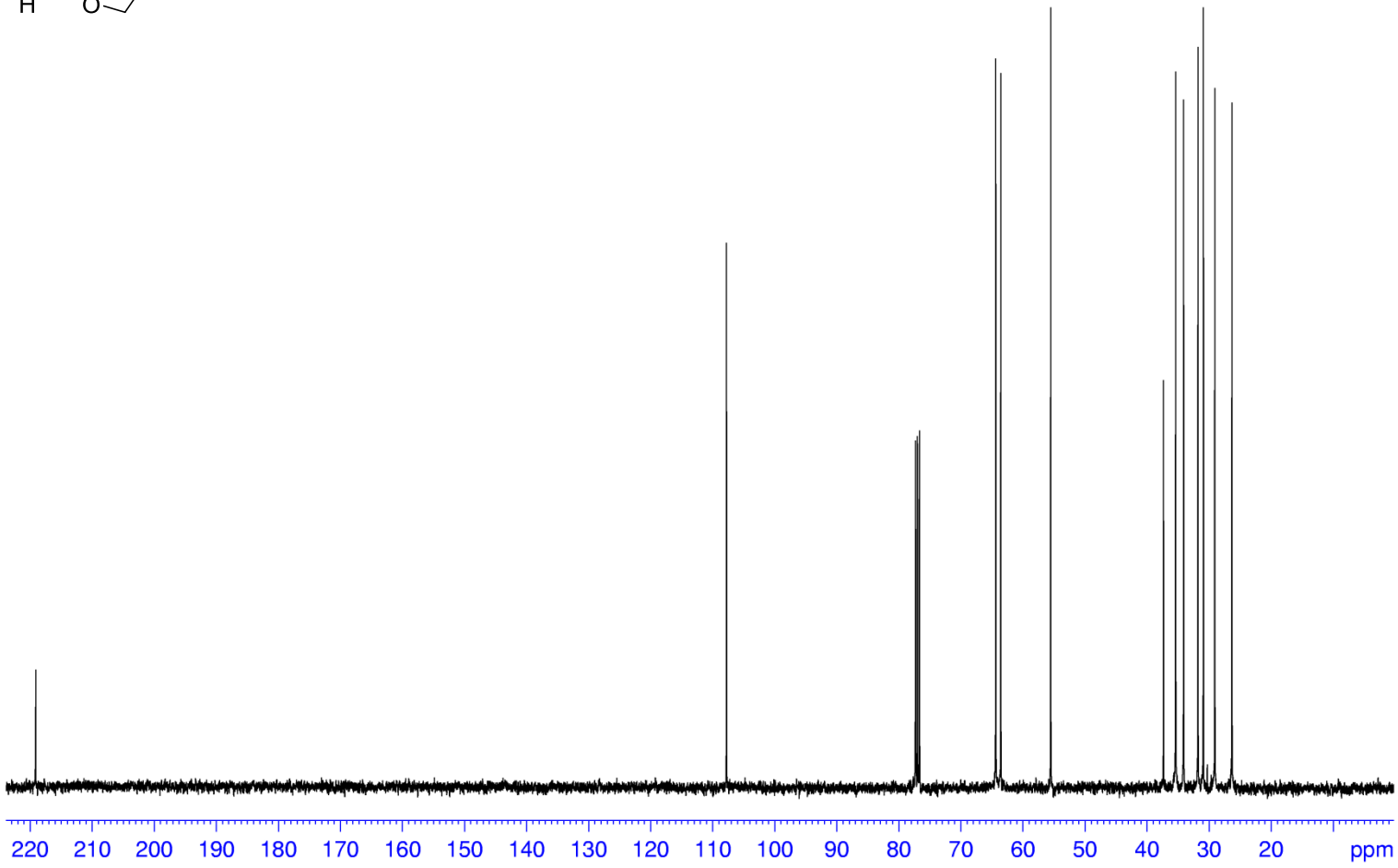


(±)-(3a'*S**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **6** (synthesised *via* Pinacol rearrangement); C_6D_6, 300 MHz.

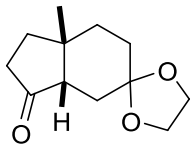


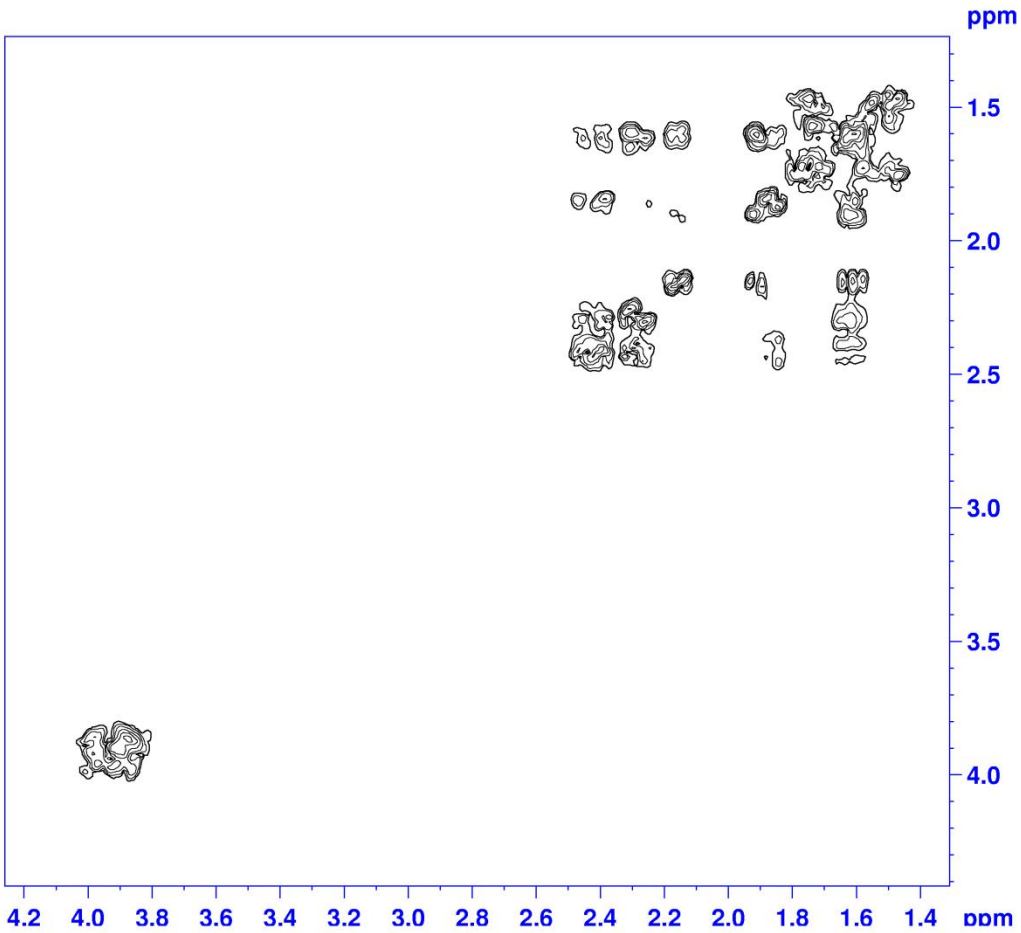
(±)-(3a'*S**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **6** (synthesised *via* Pinacol rearrangement); $CDCl_3$, 100 MHz.



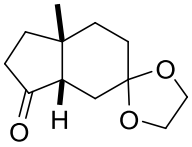


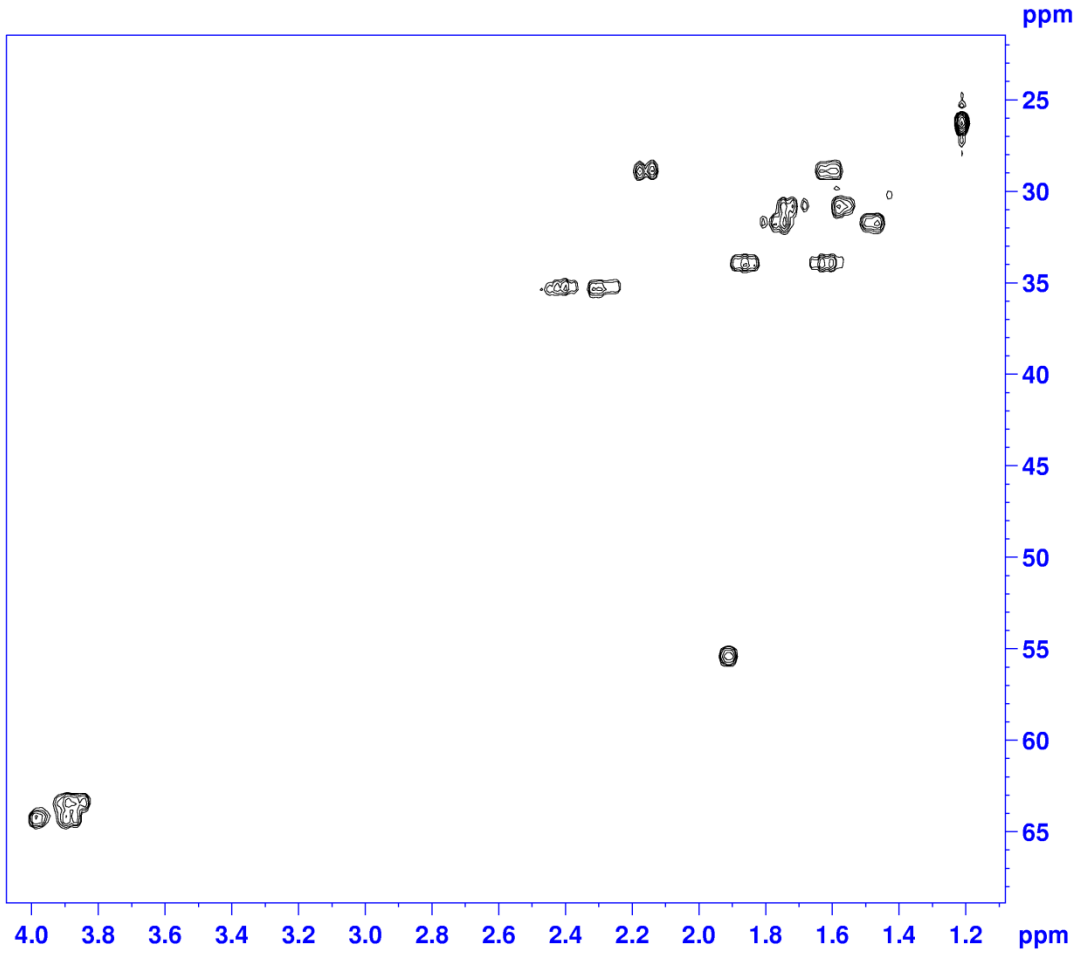
(±)-(3a'*S**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **6** (synthesised *via* Pinacol rearrangement); $CDCl_3$, 400 MHz.



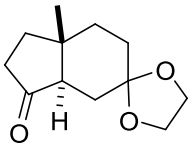


(±)-(3a'*S**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **6** (synthesised *via* Pinacol rearrangement); $CDCl_3$, 400 MHz/100 MHz.

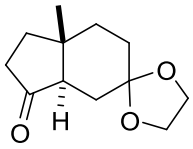




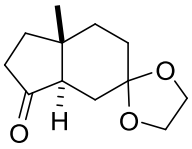
(±)-(3a'*R**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **2** (synthesised *via* Pinacol rearrangement); C_6D_6, 400 MHz.



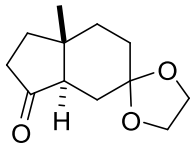
(±)-(3a'*R**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **2** (synthesised *via* Pinacol rearrangement); $CDCl_3$, 300 MHz.

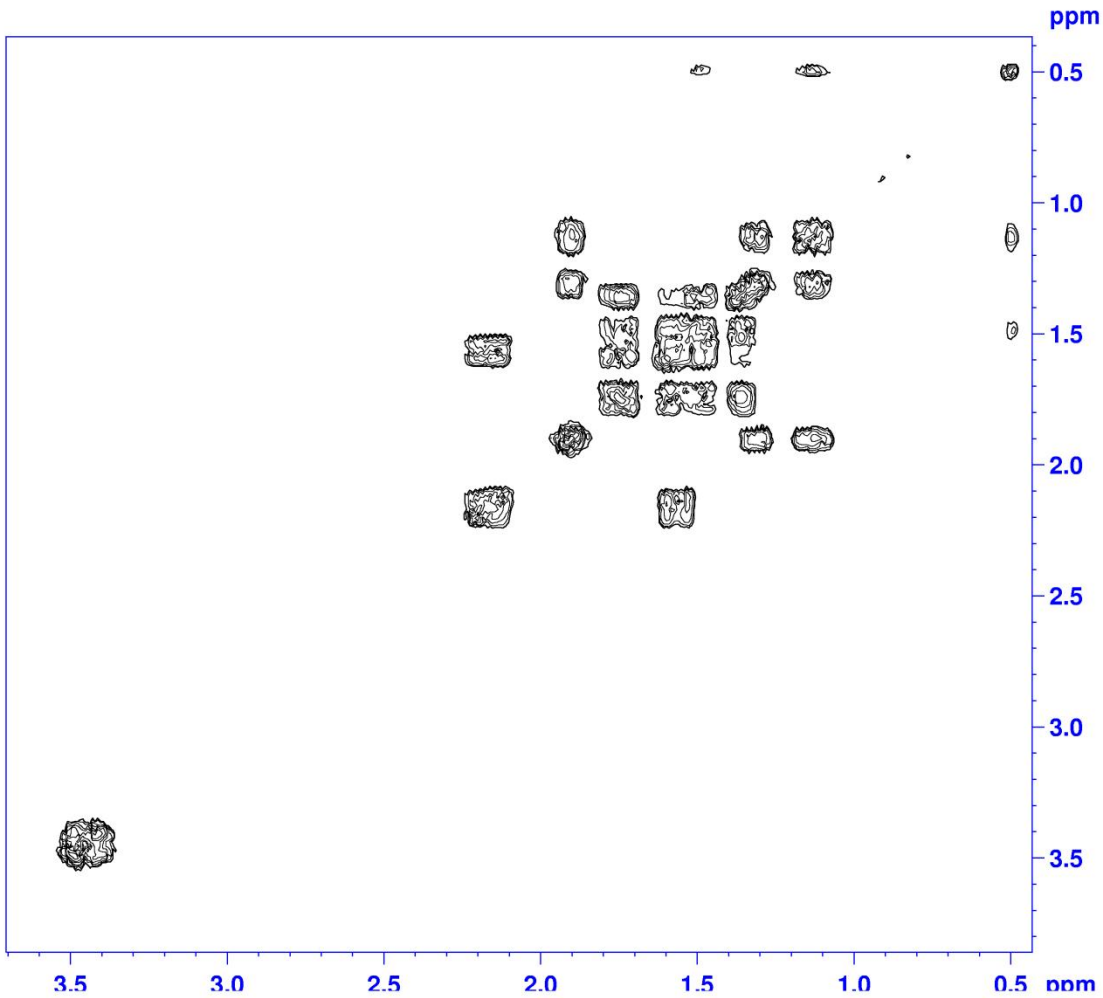


(±)-(3a'*R**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **2** (synthesised *via* Pinacol rearrangement); C_6D_6, 100 MHz.

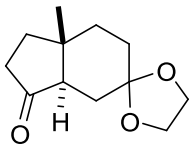


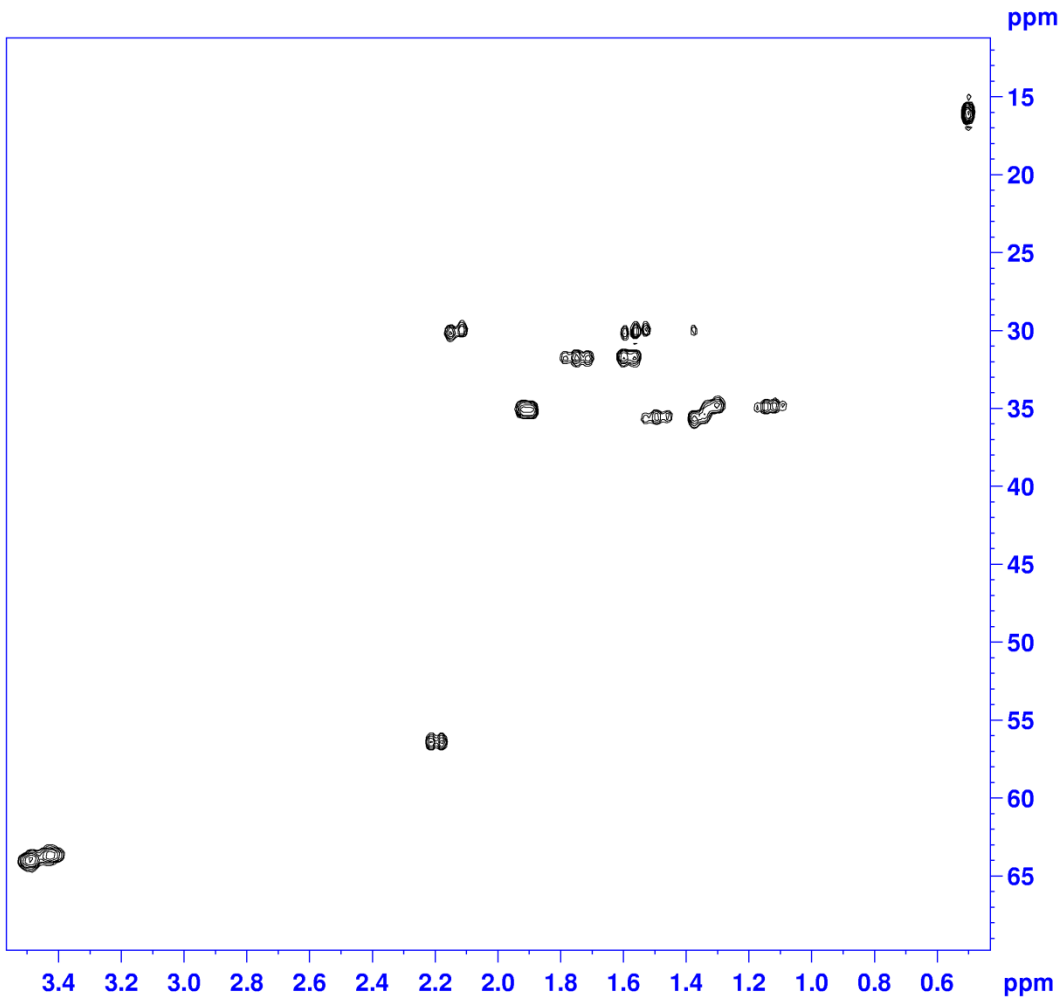
(±)-(3a'*R**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **2** (synthesised *via* Pinacol rearrangement); C_6D_6, 400 MHz.



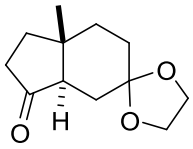


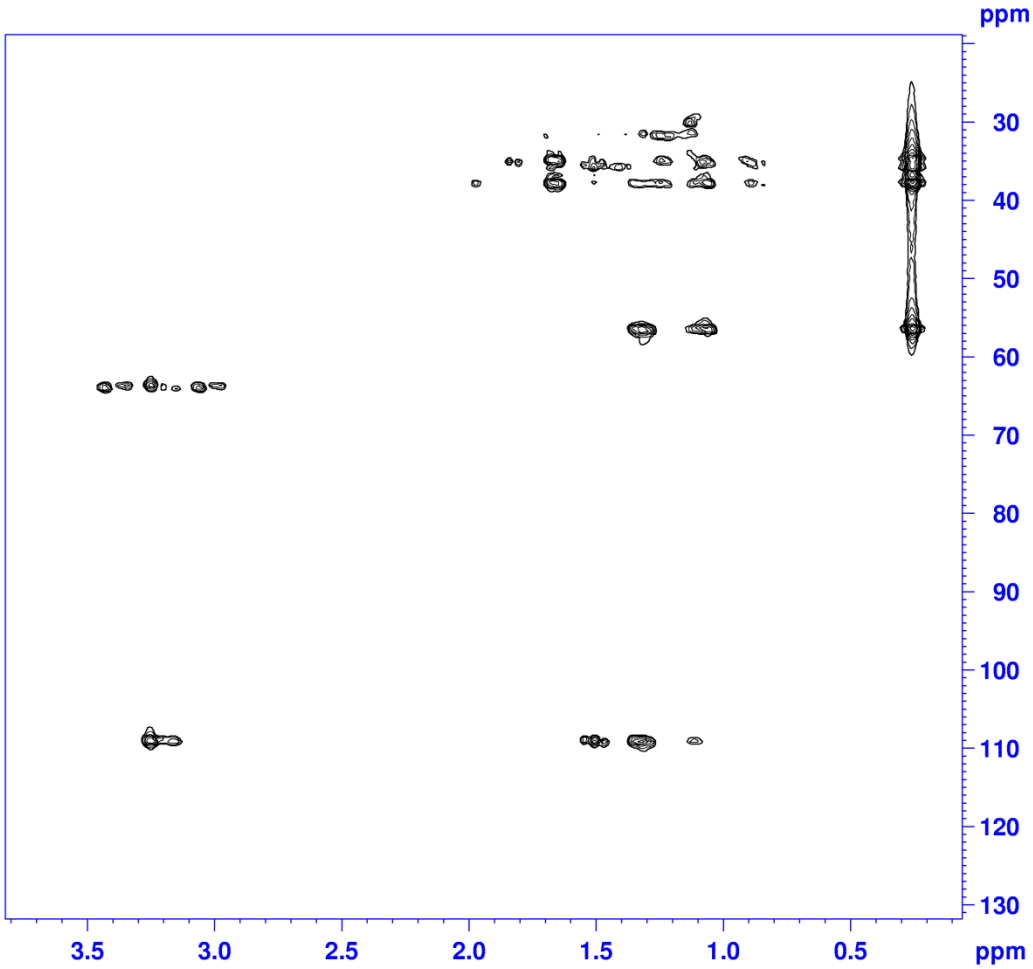
(±)-(3a'*R**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **2** (synthesised *via* Pinacol rearrangement); C_6D_6, 400 MHz/100 MHz.



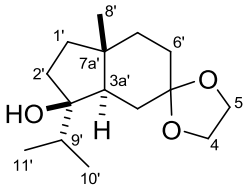


(±)-(3a'*R**,7a'*R**)-7a'-Methylhexahydrospiro[[1,3]dioxolane-2,5'-inden]-3'(2'*H*)-one **2** (synthesised *via* Pinacol rearrangement); C_6D_6, 400 MHz/100 MHz.

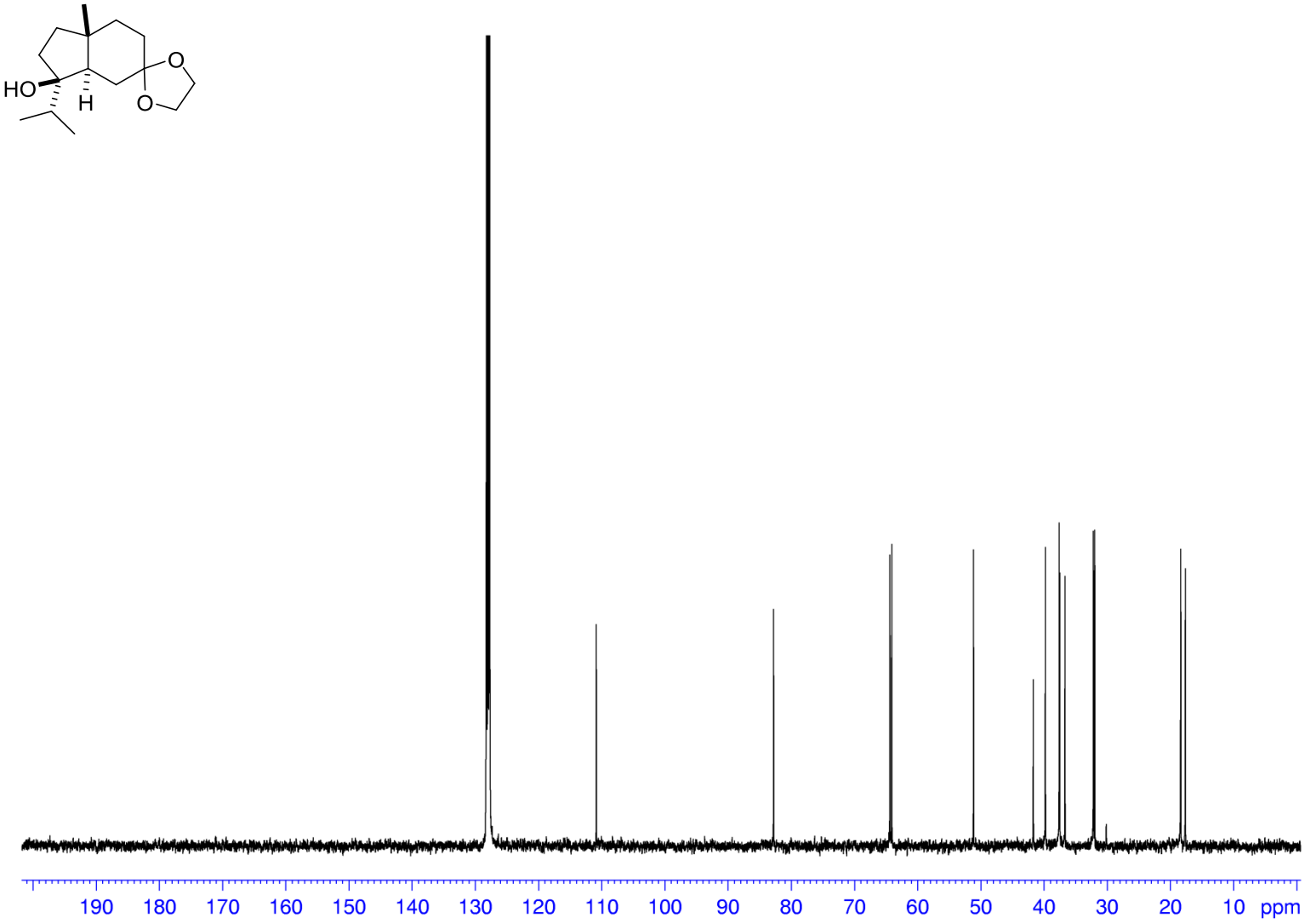




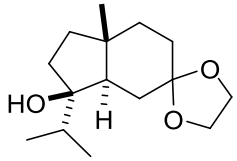
(±)-(3'*R**,3a'*R**,7a'*R**)-3'-*iso*Propyl-7a'-methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **12;** C_6D_6, 400 MHz.

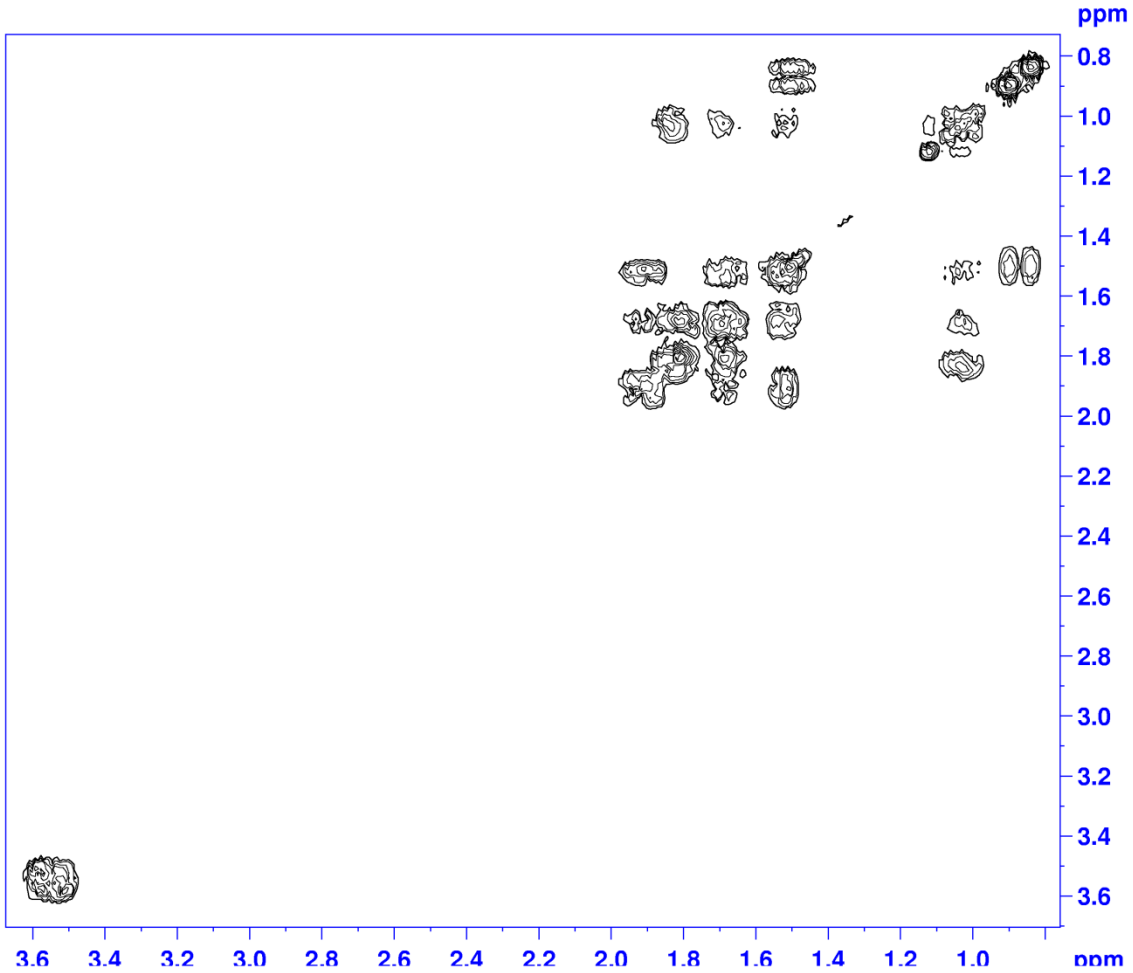


(±)-(3'*R**,3a'*R**,7a'*R**)-3'-*iso*Propyl-7a'-methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **12;** C_6D_6, 100 MHz.

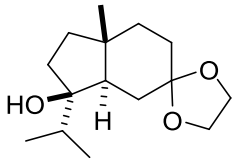


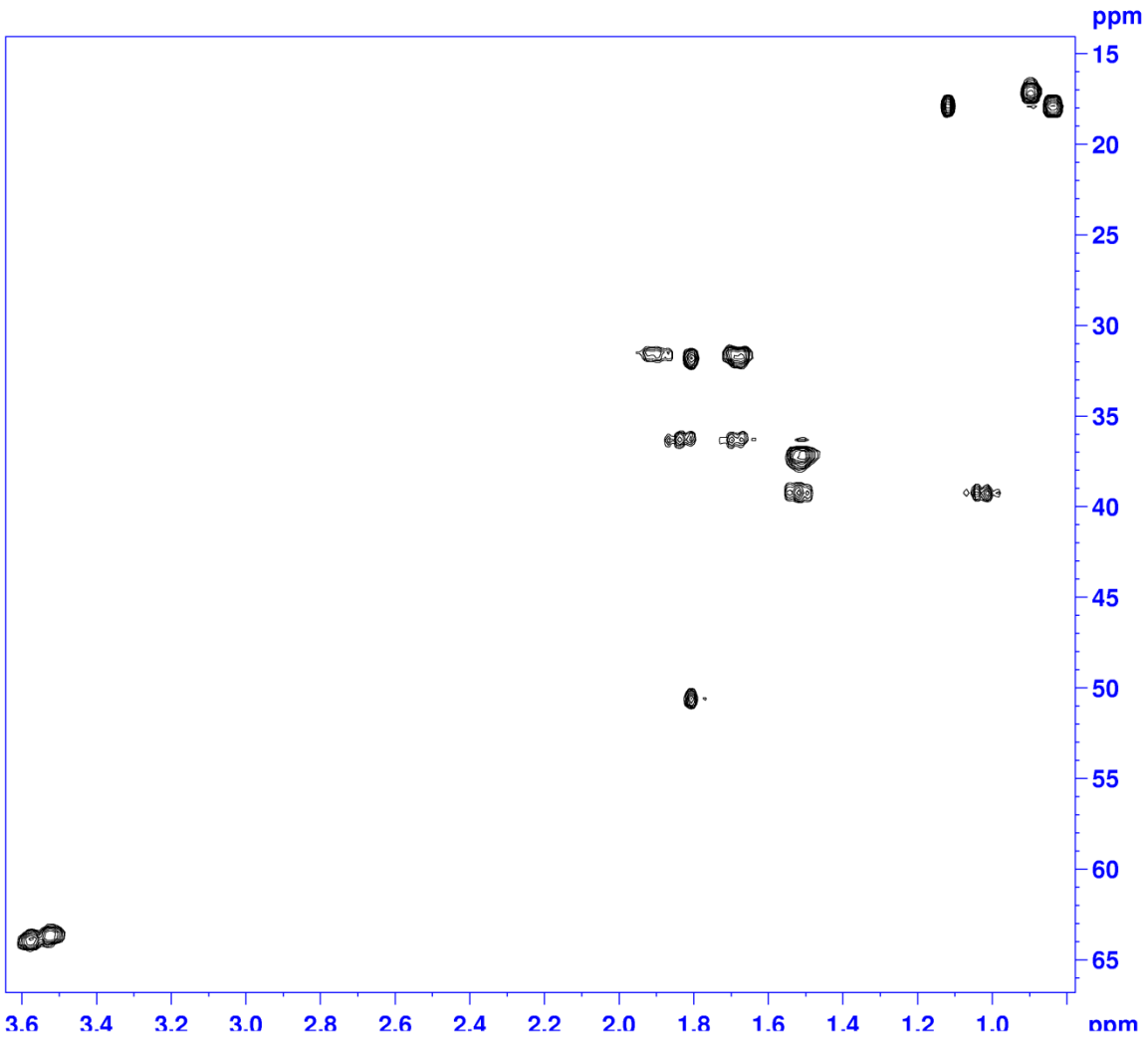
(±)-(3'*R**,3a'*R**,7a'*R**)-3'-*iso*Propyl-7a'-methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **12;** C_6D_6, 400 MHz.



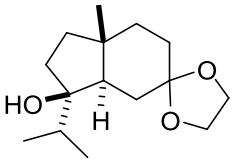


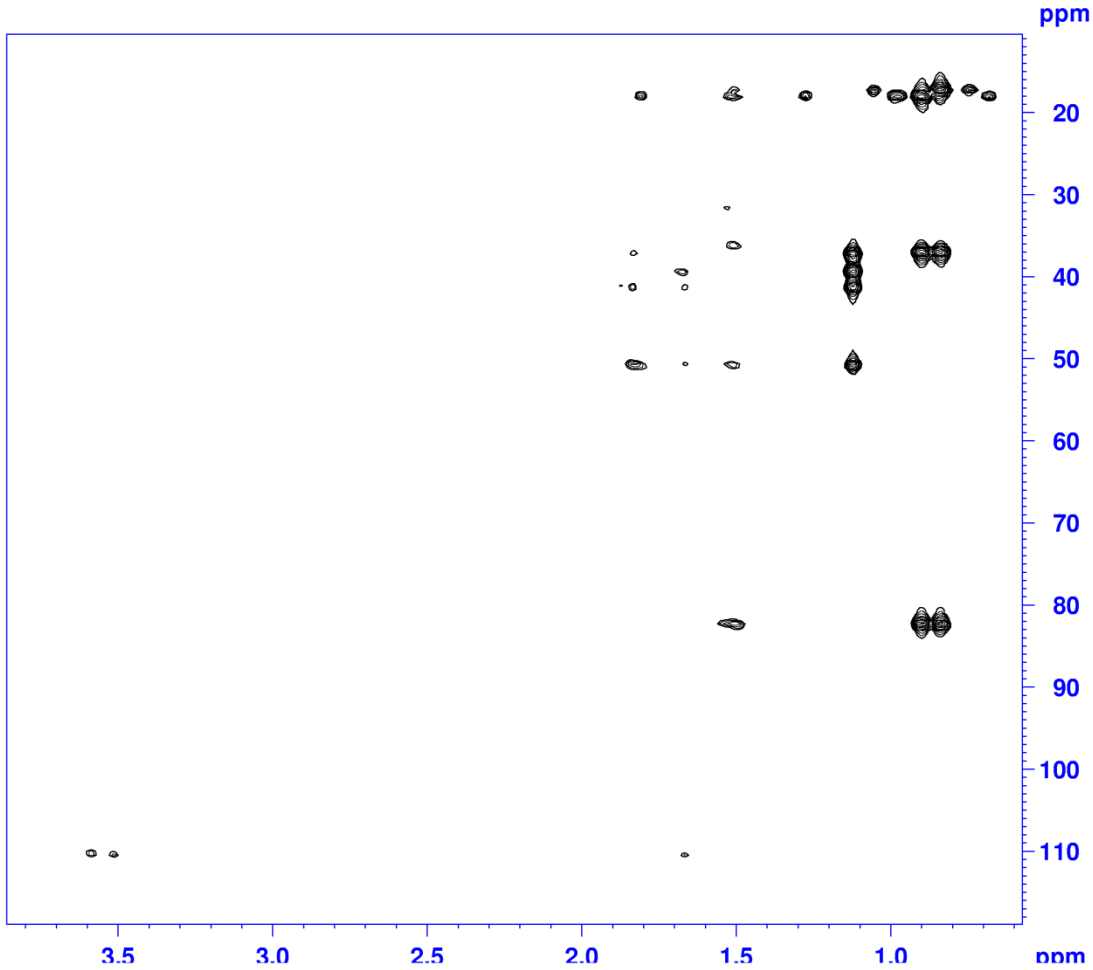
(±)-(3'*R**,3a'*R**,7a'*R**)-3'-*iso*Propyl-7a'-methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **12;** C_6D_6, 400 MHz/100 MHz.



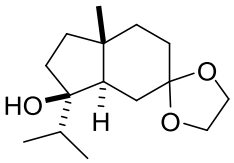


(±)-(3'*R**,3a'*R**,7a'*R**)-3'-*iso*Propyl-7a'-methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **12;** C_6D_6, 400 MHz/100 MHz.

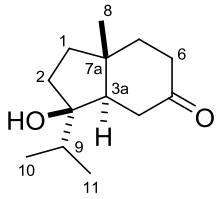




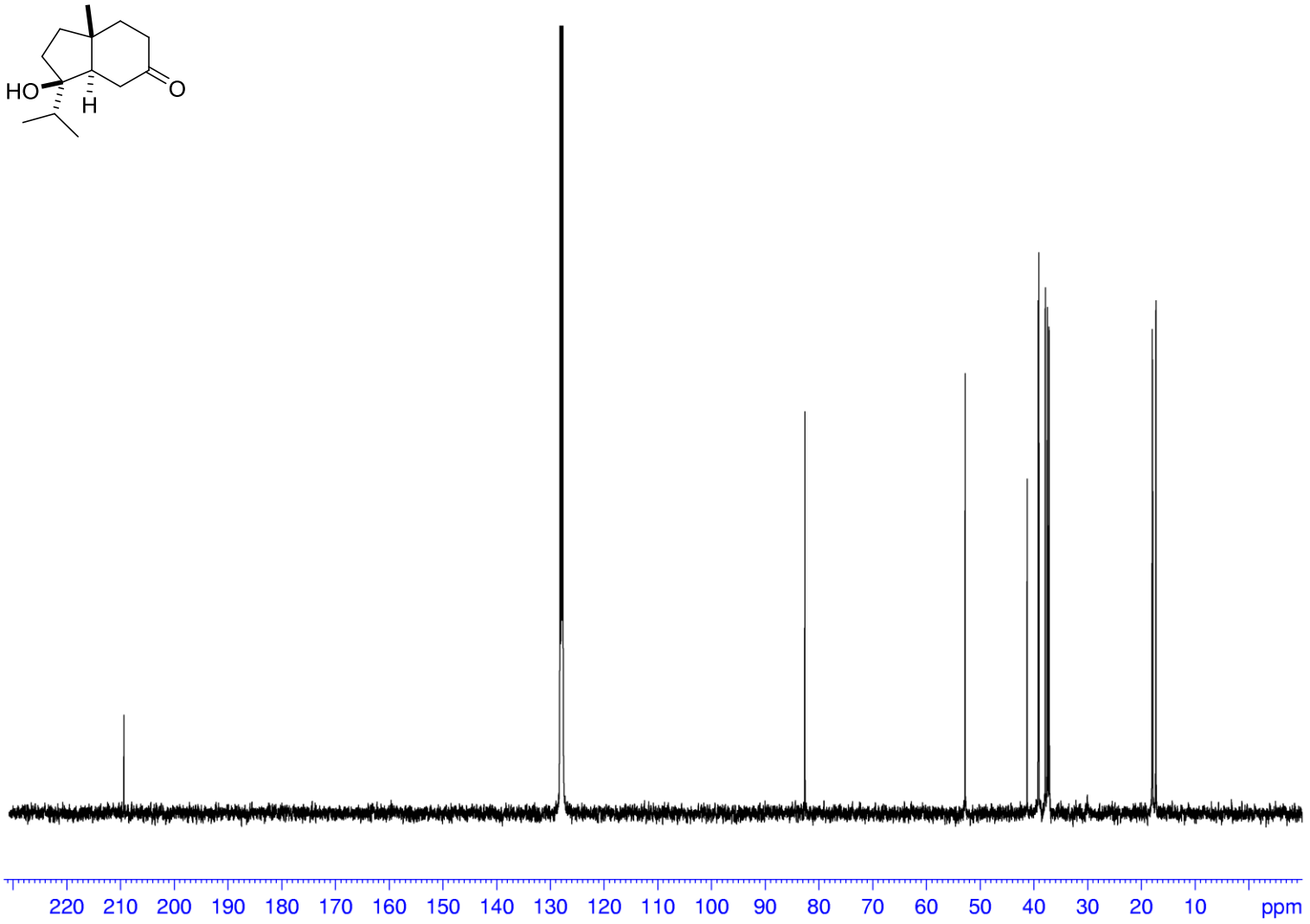
(±)-(3'*R**,3a'*R**,7a'*R**)-3'-*iso*Propyl-7a'-methyloctahydrospiro[[1,3]dioxolane-2,5'-inden]-3'-ol **12;** C_6D_6, 400 MHz.



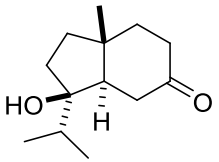
(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 400 MHz.

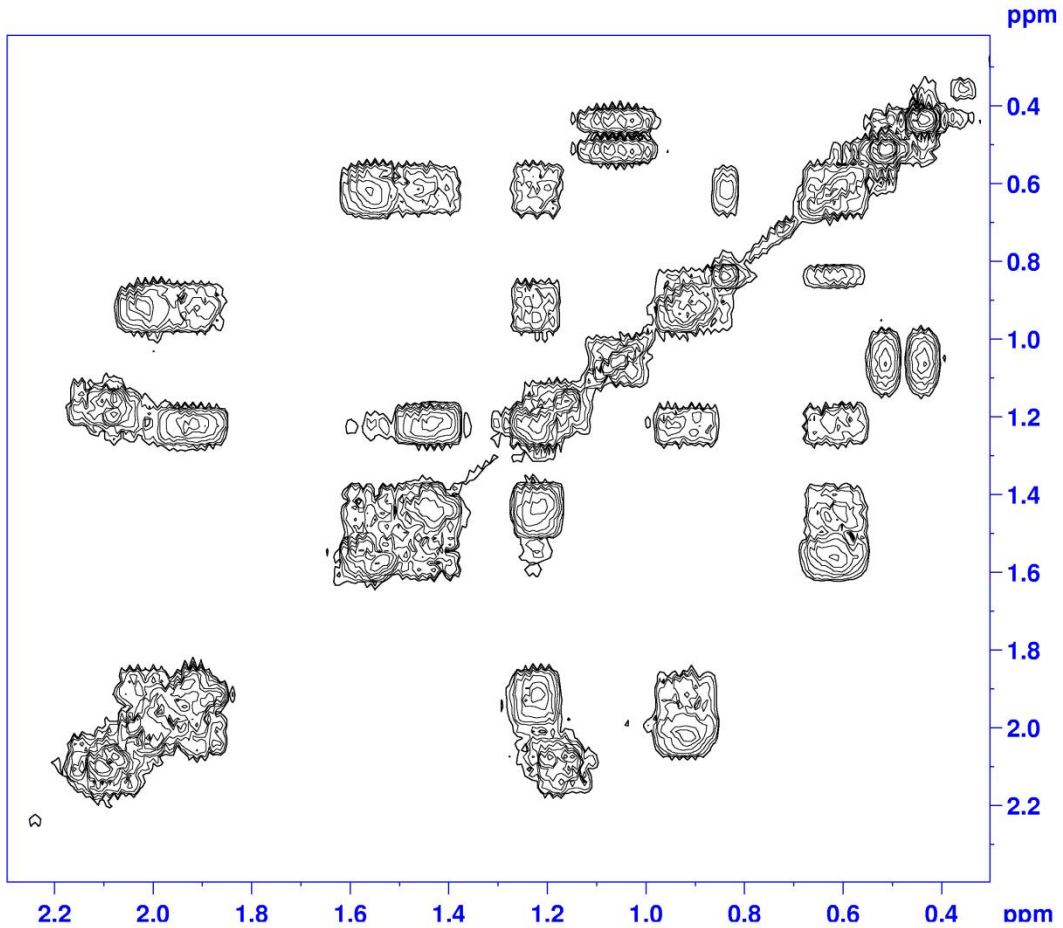


(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 100 MHz.

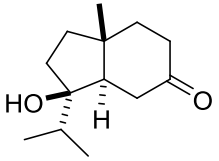


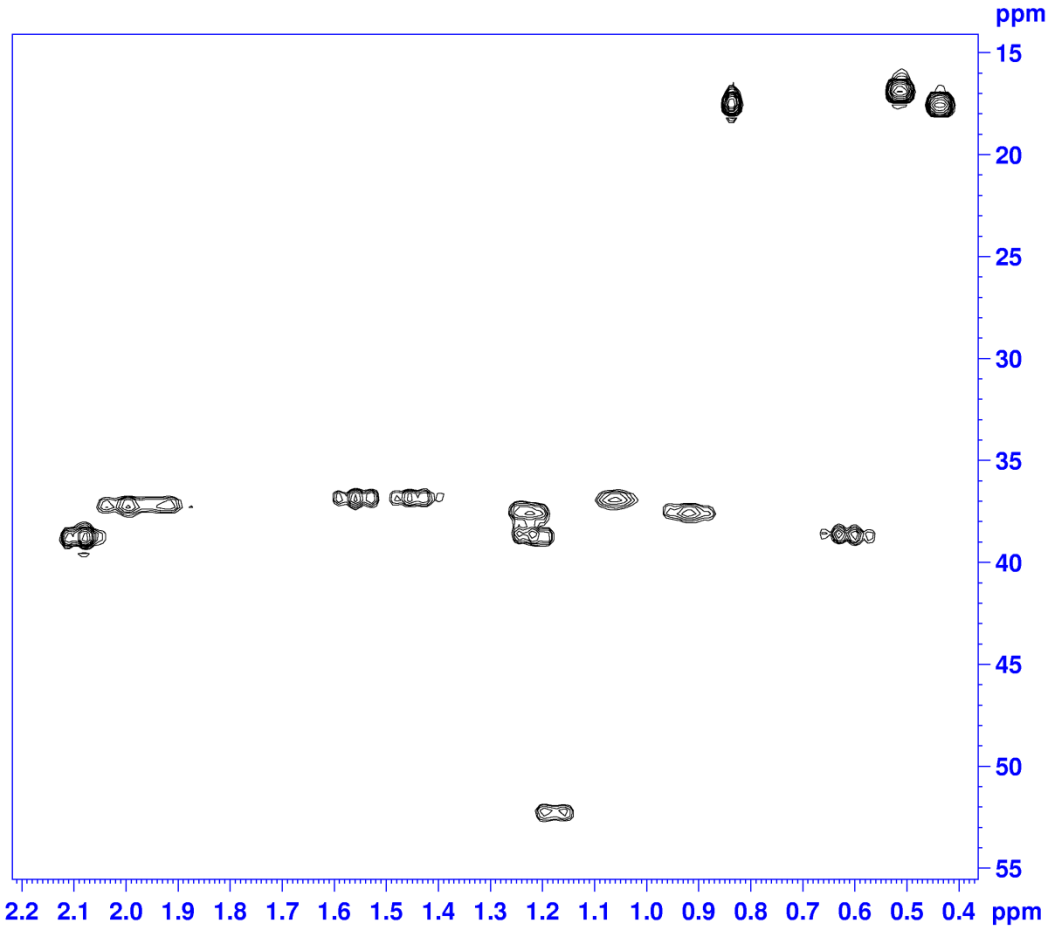
(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 400 MHz.



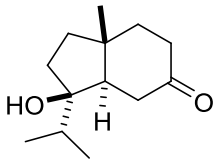


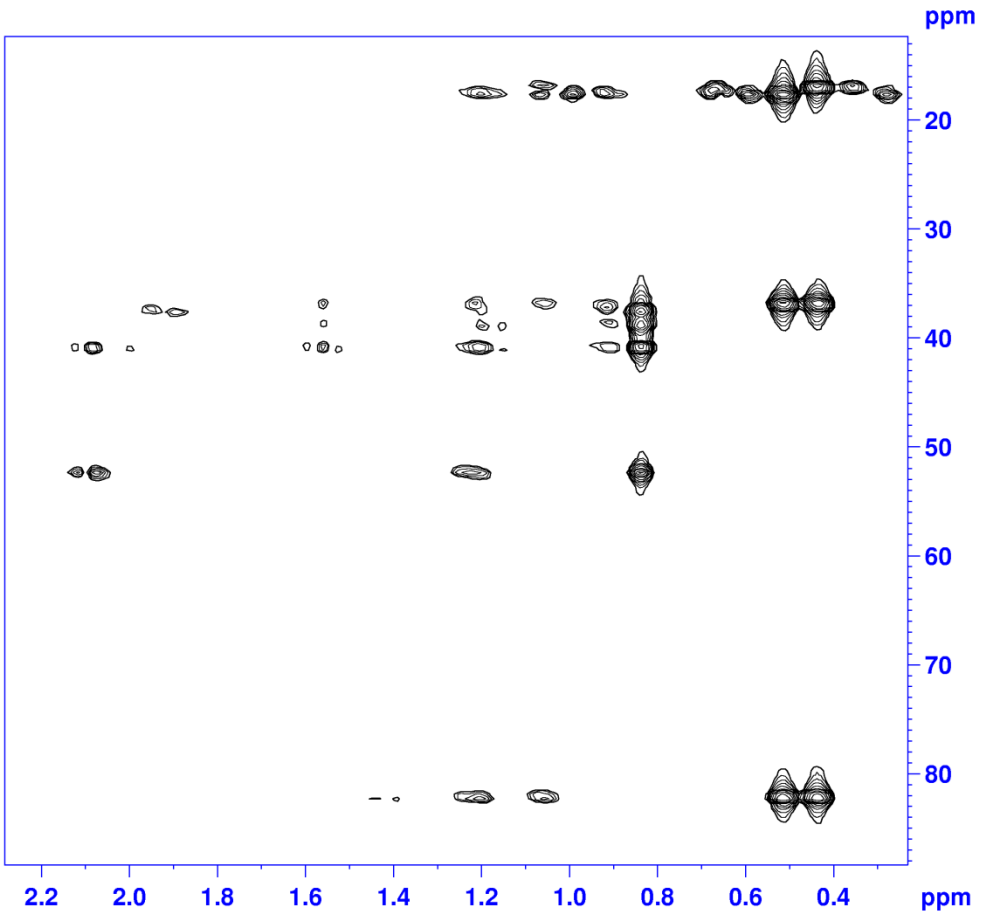
(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 400 MHz/100 MHz.



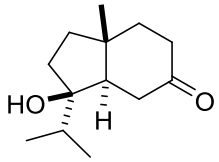


(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 400 MHz/100 MHz.

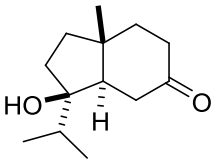


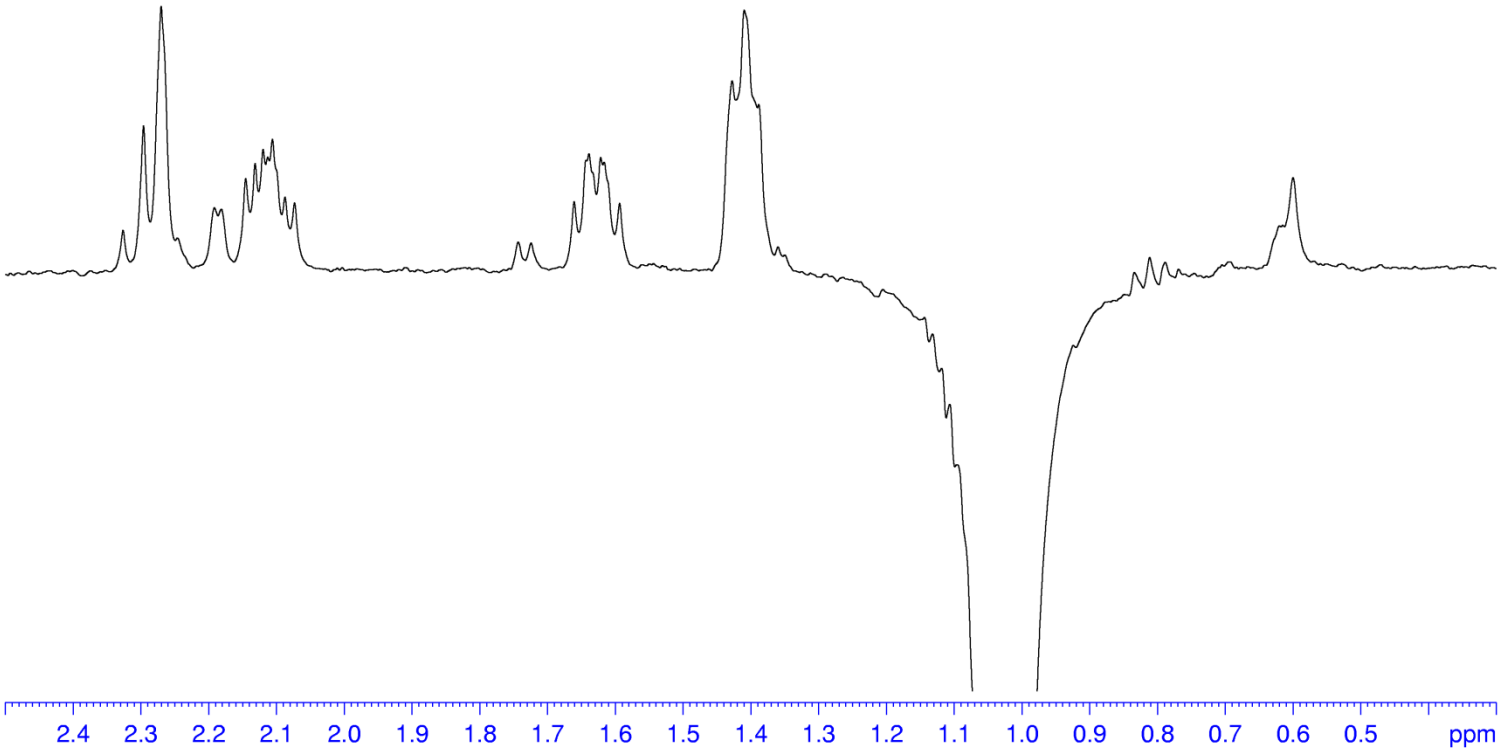


(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 400 MHz.

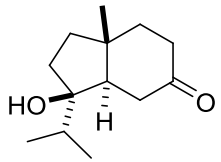


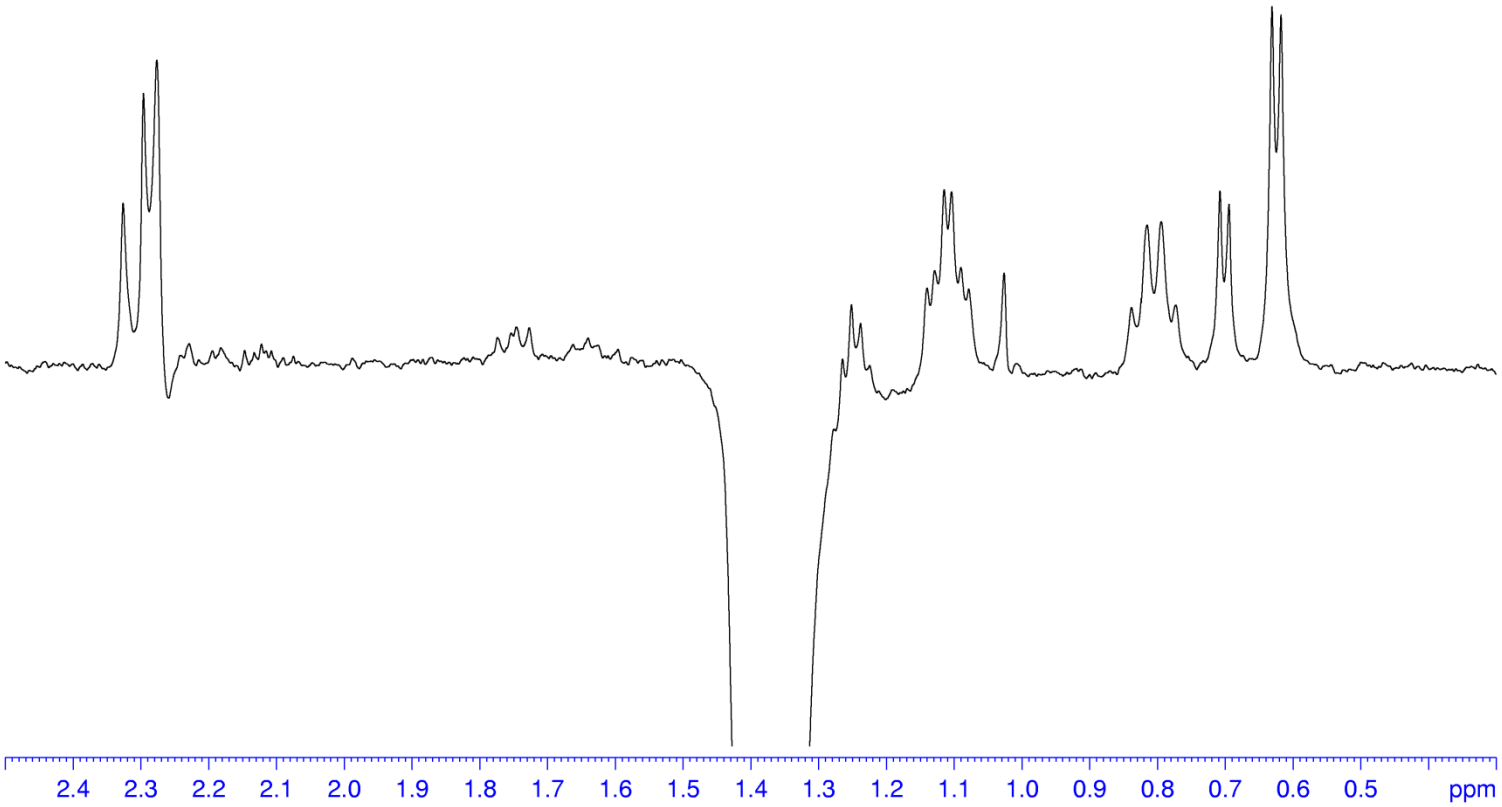
(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 500 MHz; nOe irradiation of methyl group protons.



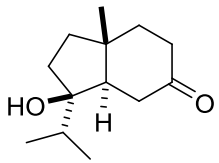


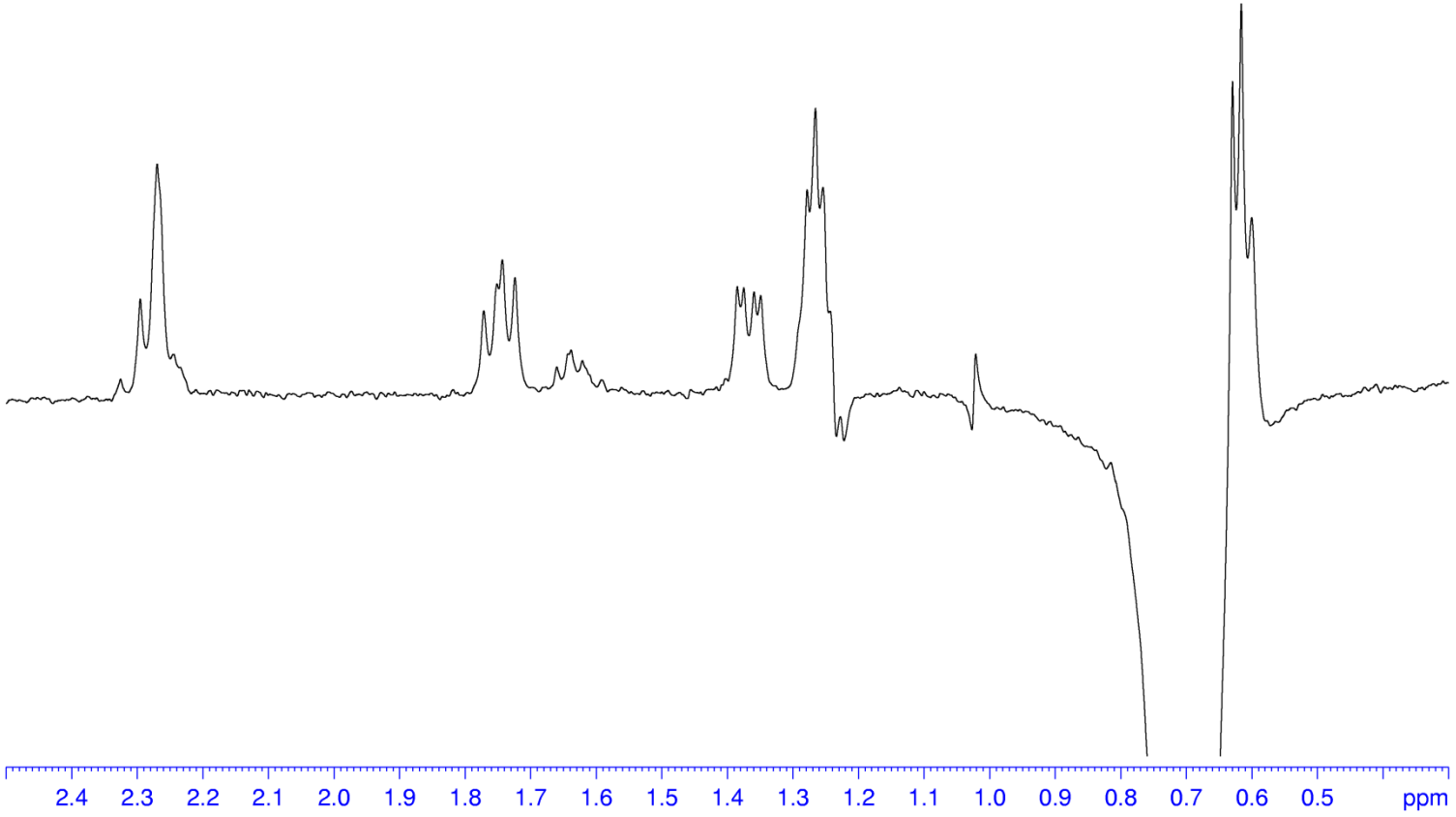
(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 500 MHz; nOe irradiation of H-3a.



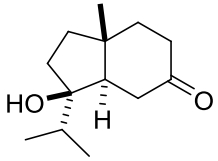


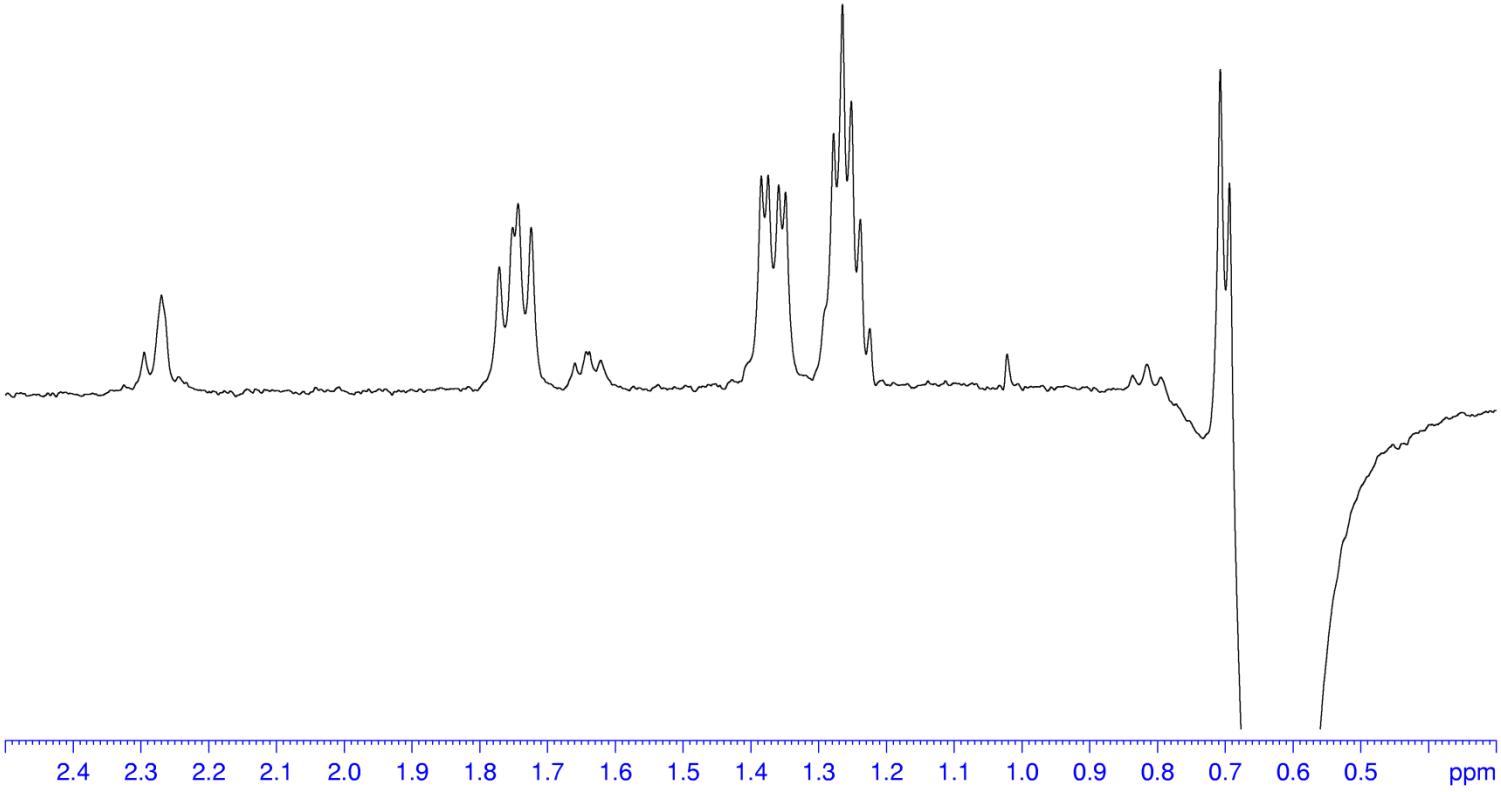
(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 500 MHz; nOe irradiation of high frequency ^{i}Pr methyl group.



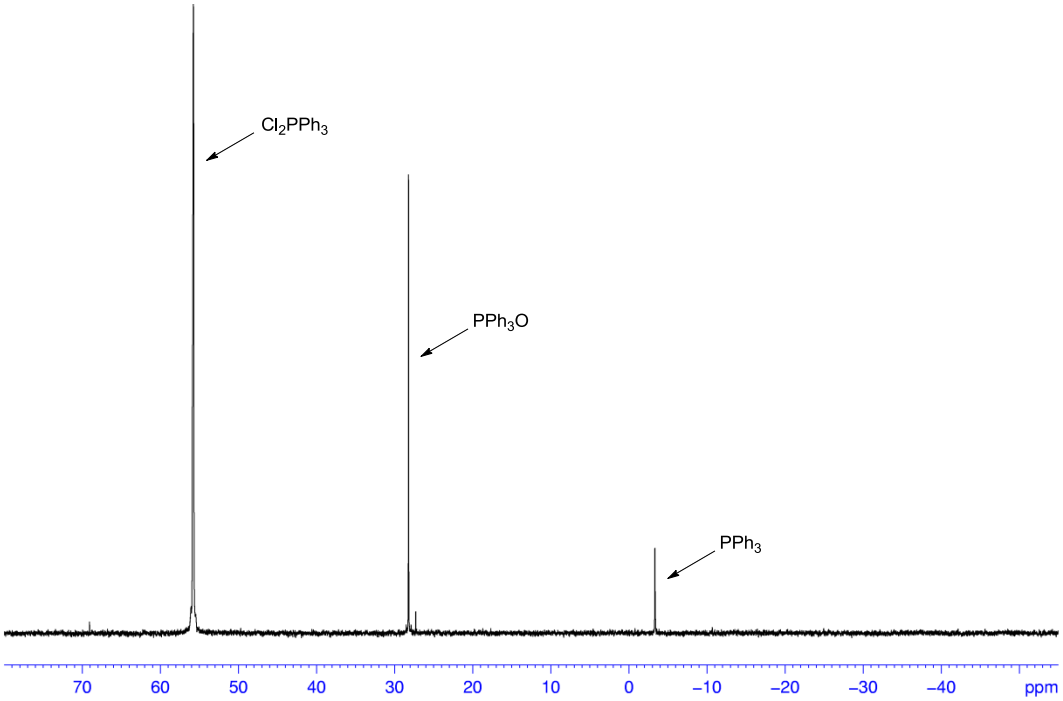


(±)-(3*R**,3a*R**,7a*R**)-3-Hydroxy-3-*iso*propyl-7a-methylhexahydro-1*H*-inden-5(6*H*)-one **1**; C_6D_6, 500 MHz; nOe irradiation of lower frequency ^{i}Pr methyl group.

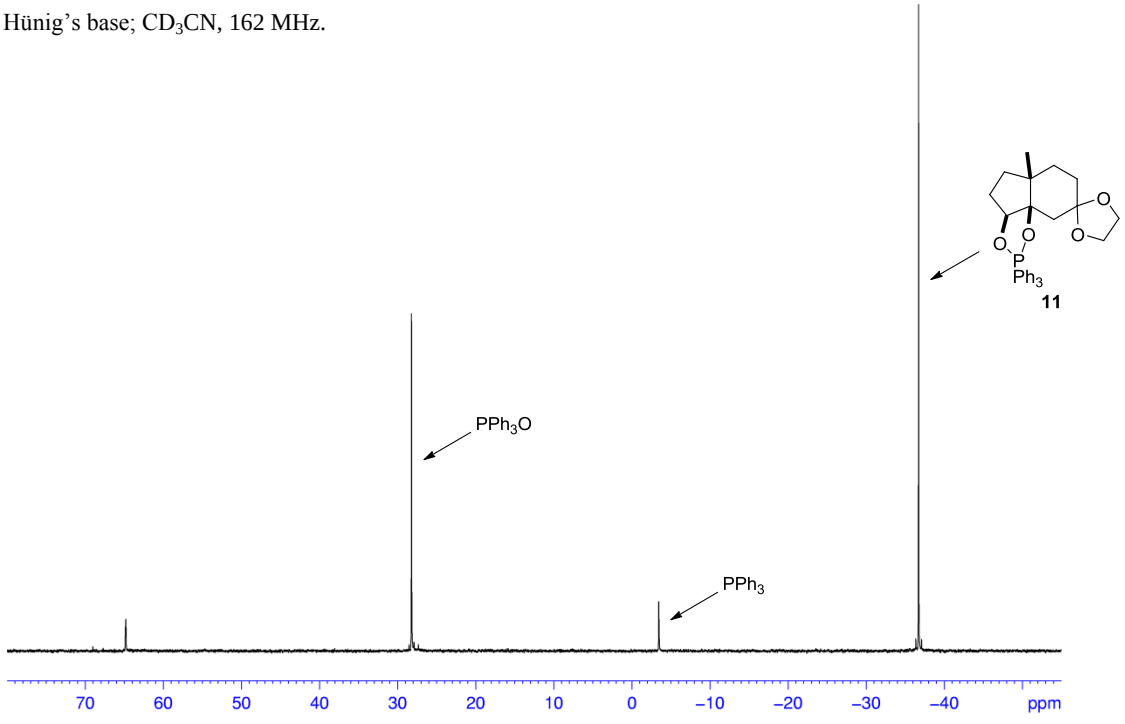




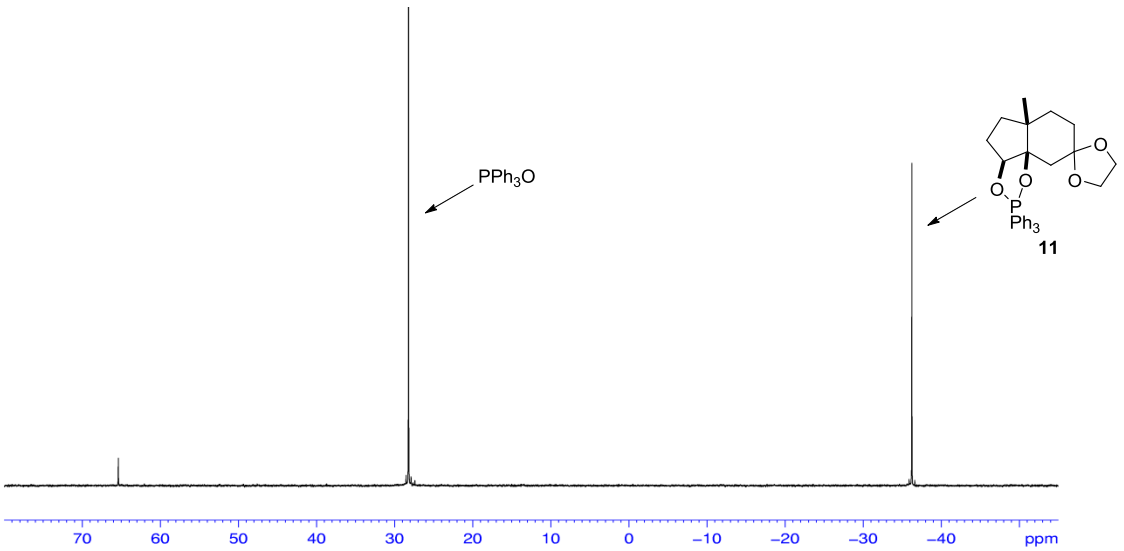
^{31}P NMR of a freshly prepared mixture of Cl_2PPh_3 and Hünig's base; CD_3CN, 162 MHz.



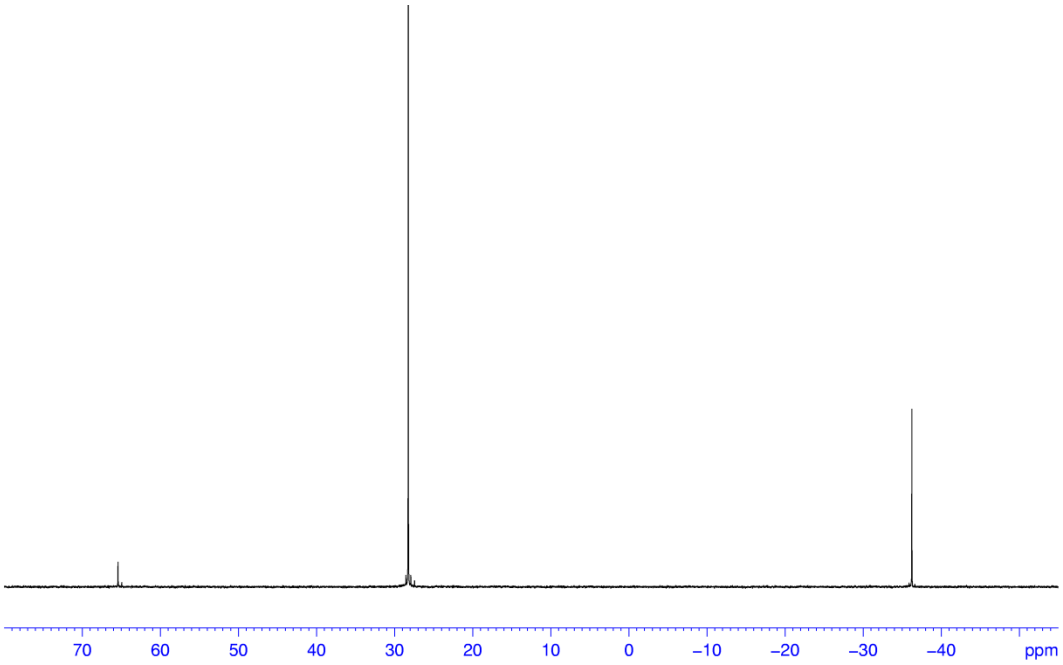
^{31}P NMR immediately after addition of **9** to a freshly prepared solution of Cl_2PPh_3 and Hünig's base; CD_3CN, 162 MHz.



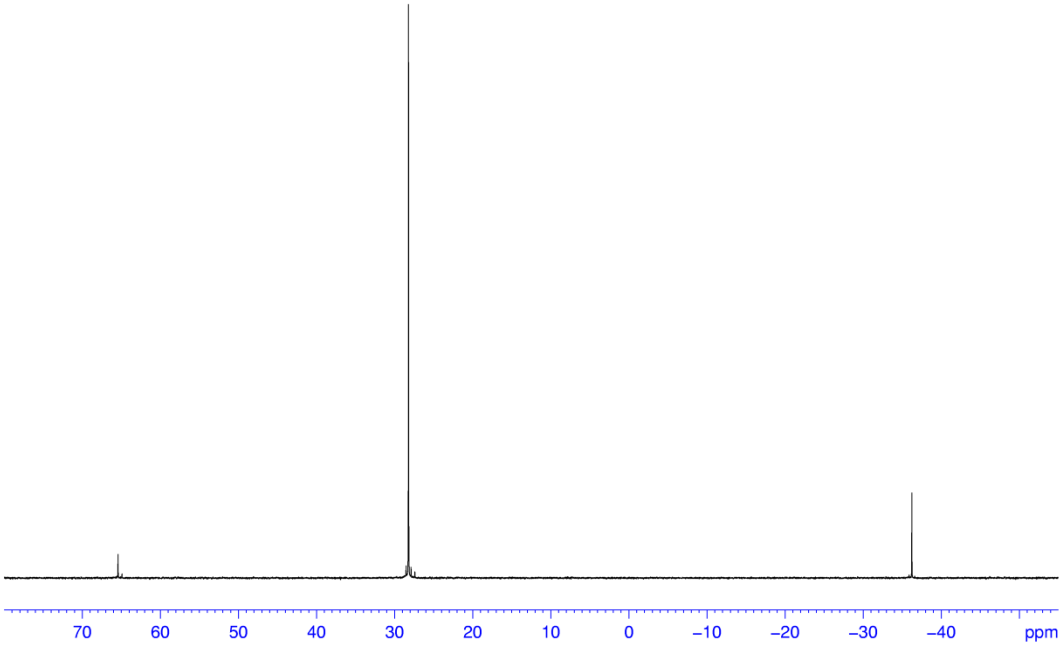
^{31}P NMR of **11** after 10 min at 60 °C; CD_3CN, 162 MHz.



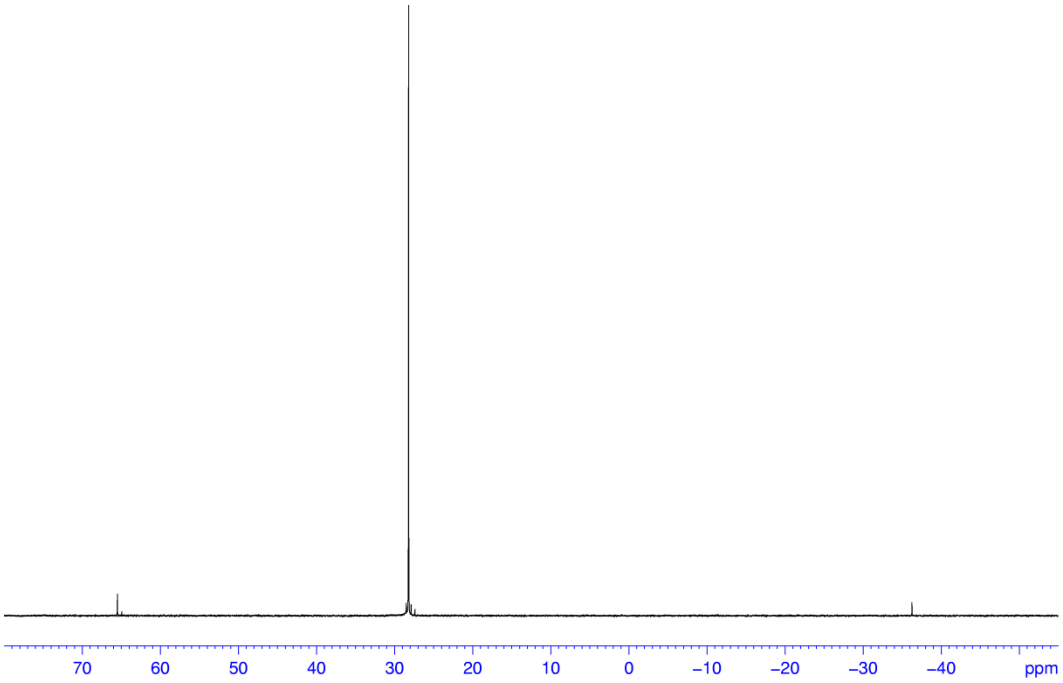
^{31}P NMR of **11** after 30 min at 60 °C; CD_3CN, 162 MHz.



^{31}P NMR of **11** after 50 min at 60 °C; CD_3CN, 162 MHz.



^{31}P NMR of **11** after 110 min at 60 °C; CD_3CN, 162 MHz.



^{31}P NMR of **11** after 170 min at 60 °C; CD_3CN, 162 MHz.