10.1071/CH20149_AC

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SUPPLEMENTARY MATERIAL

Application of the Variable Oxygen Probe to Derivatives of 2,6-Dimethyltetrahydropyran-2-ol: Evidence for Through-bond n_O-σ_{CC}-σ^*_{CO} Interactions

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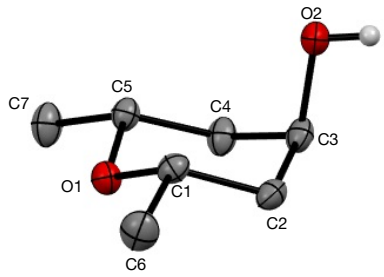
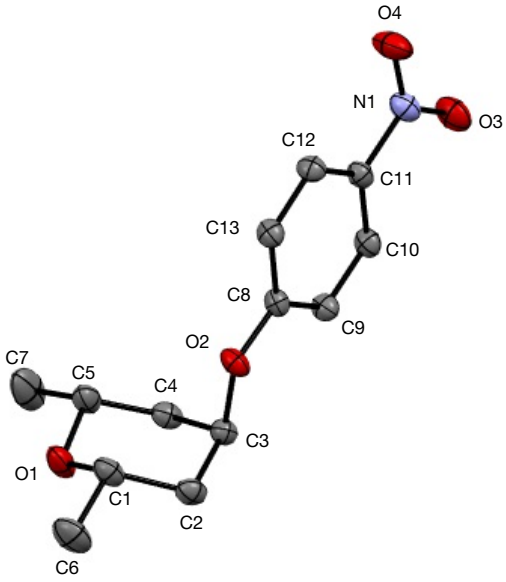
[B]Département de chimie, Faculté des sciences et de génie, Université Laval,1045 avenue de la médecine, Québec, Québec, G1V 0A6, Canada.

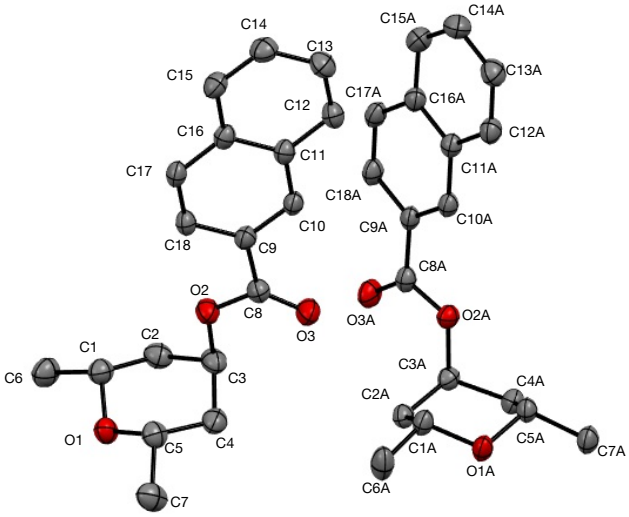
[C]Corresponding author. Email: whitejm@unimelb.edu.au



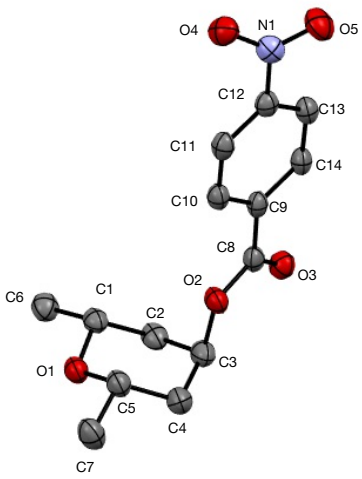
SI Figure S1: Compound **4a**.



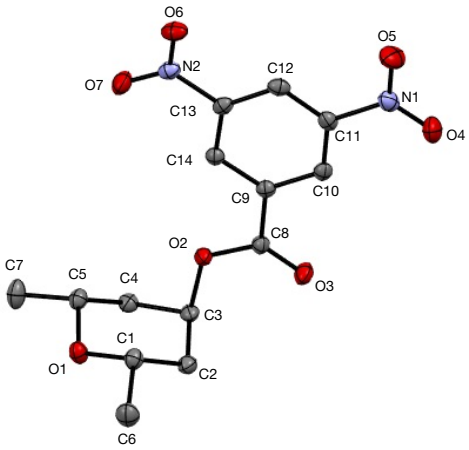
SI Figure S2: Compound **4b**.



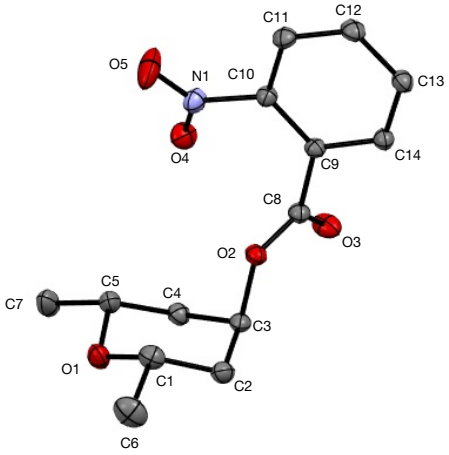
SI Figure S3: Compound **4c**.



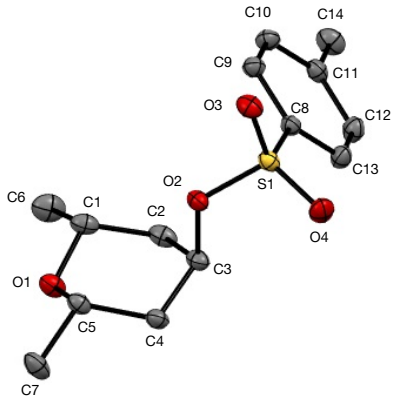
SI Figure S4: Compound **4d**.



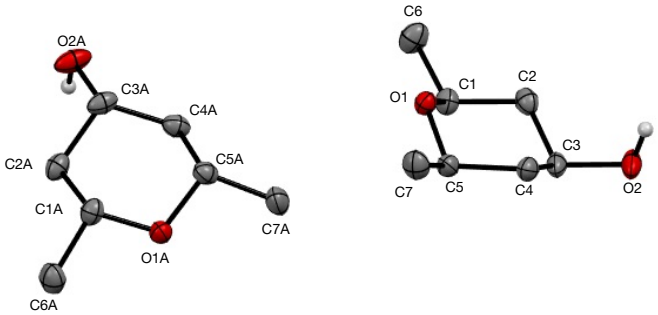
SI Figure S5: Compound **4e**.



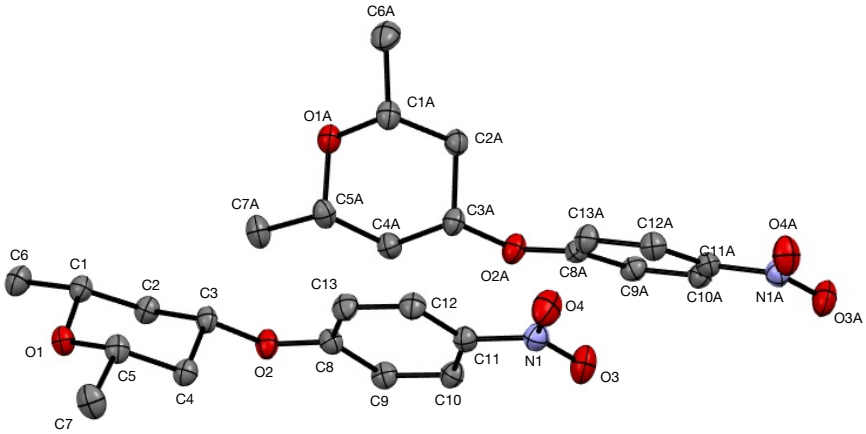
SI Figure S6: Compound **4f**.



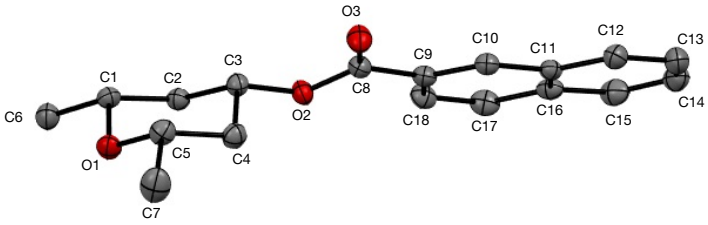
SI Figure S7: Compound **4h**.



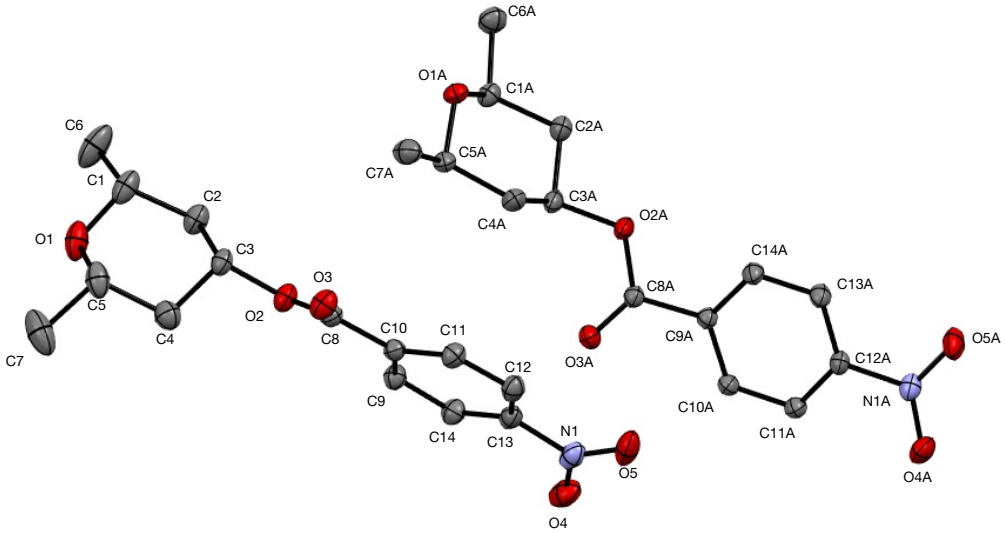
SI Figure S8: Compound **5a**.



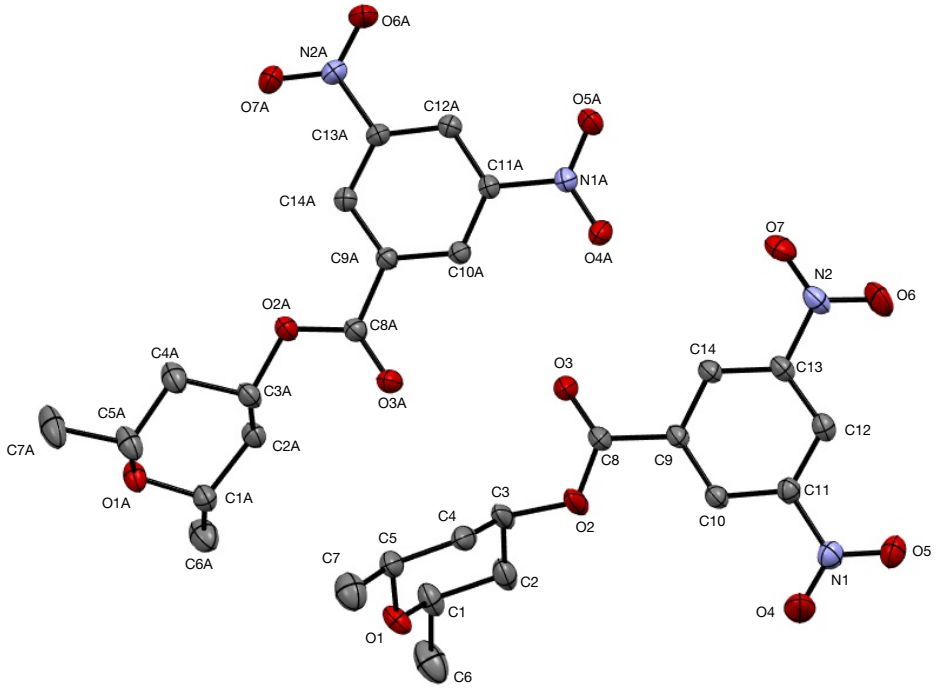
SI Figure S9: Compound **5b**.



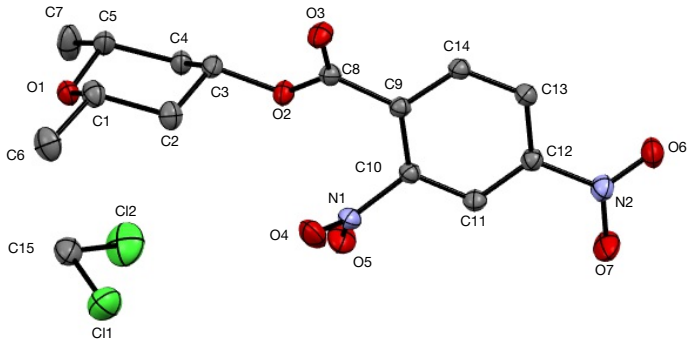
SI Figure S10: Compound **5c**.



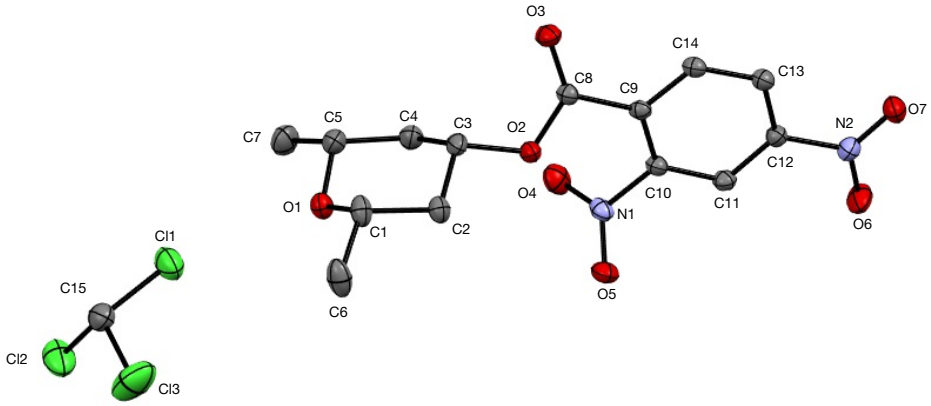
SI Figure S11: Compound **5d**.



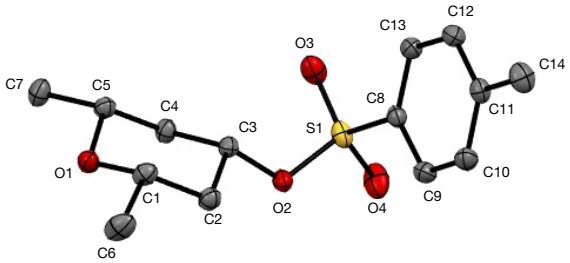
SI Figure S12: Compound **5e**.



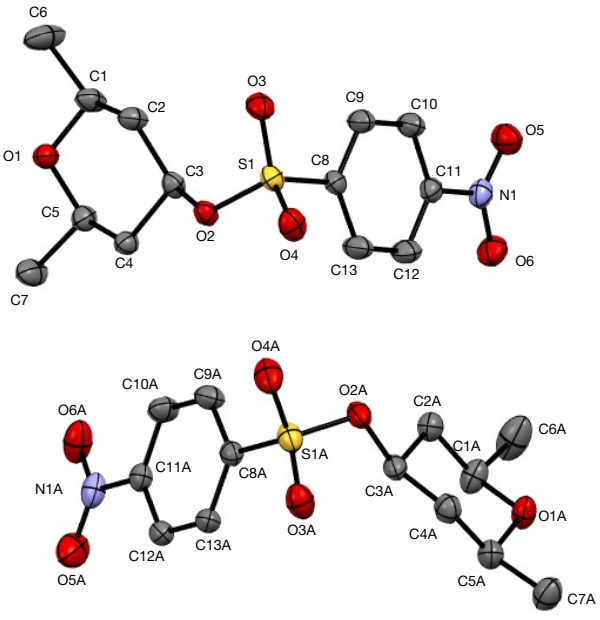
SI Figure S13: Compound **5g**. CH_2Cl_2



SI Figure S14: Compound **5g**. $CHCl_3$

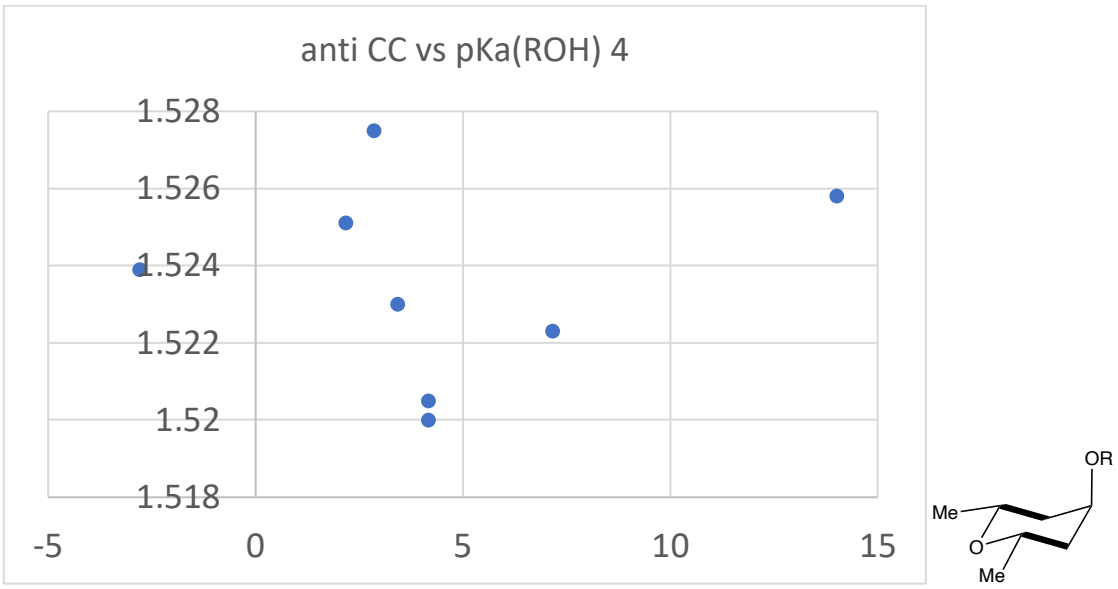


SI Figure S15: Compound **5h**.

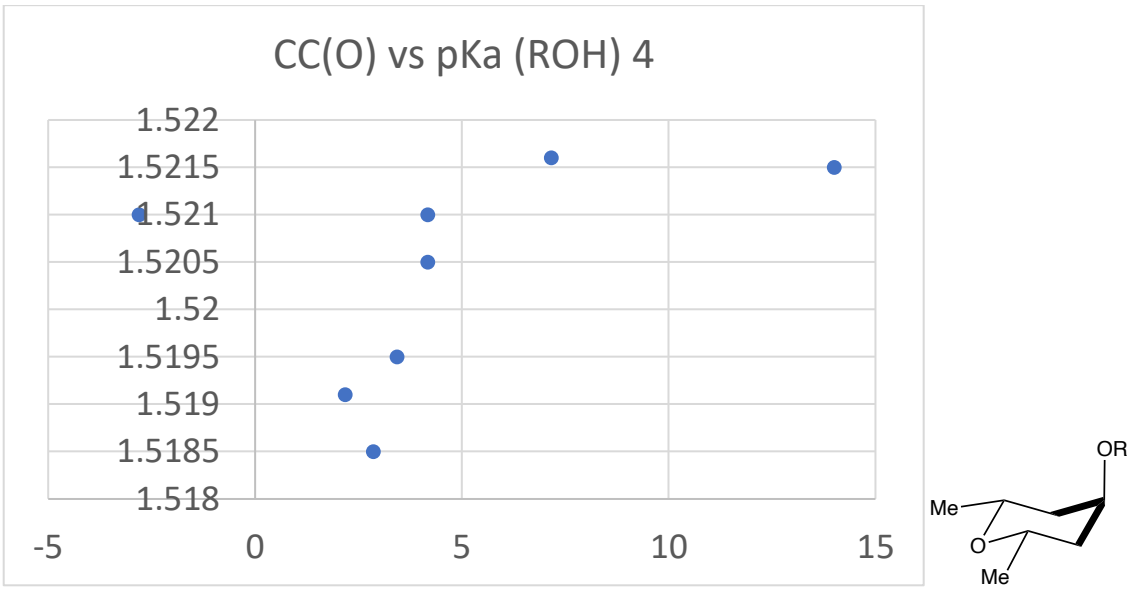


SI Figure S16: Compound **5i**.

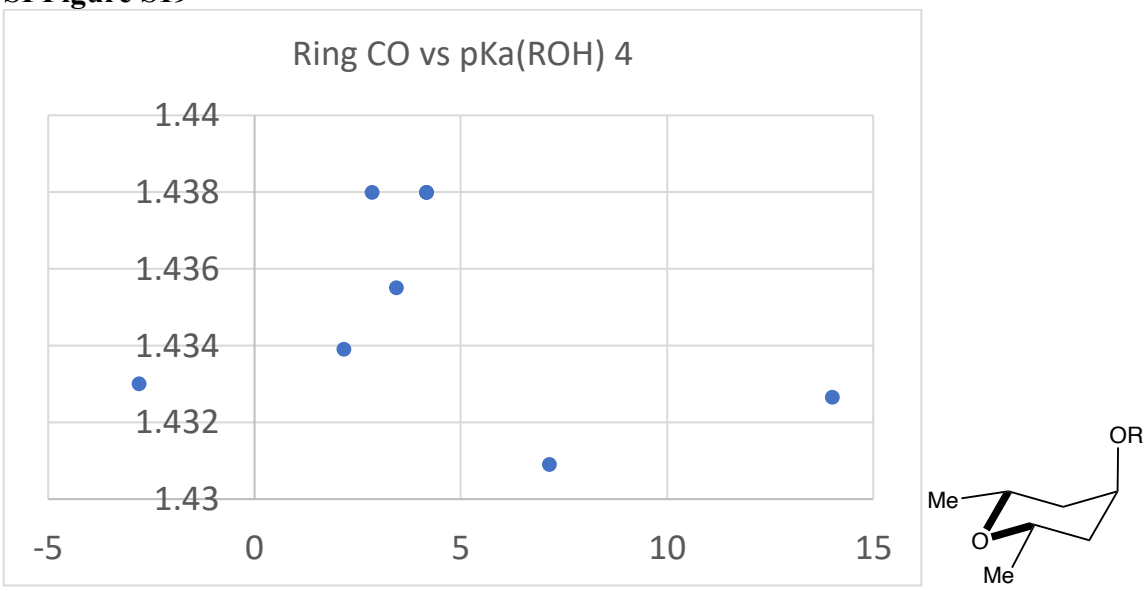
SI Figure S17



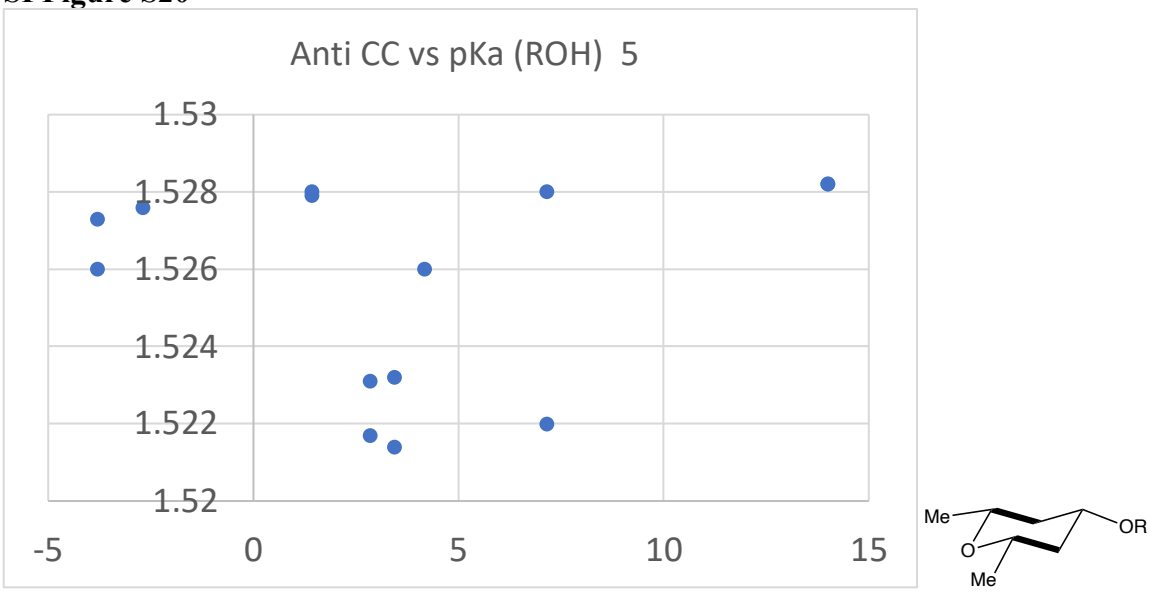
SI Figure S18



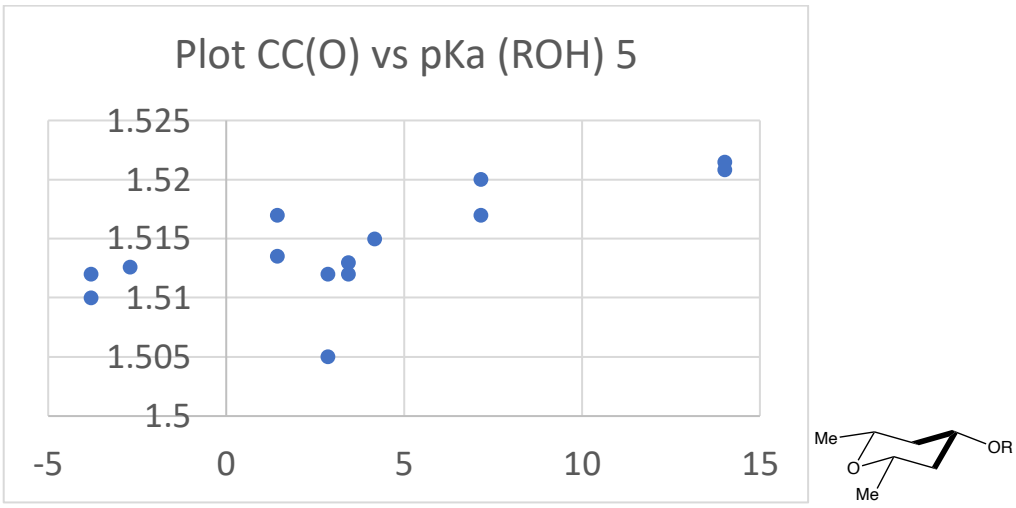
SI Figure S19



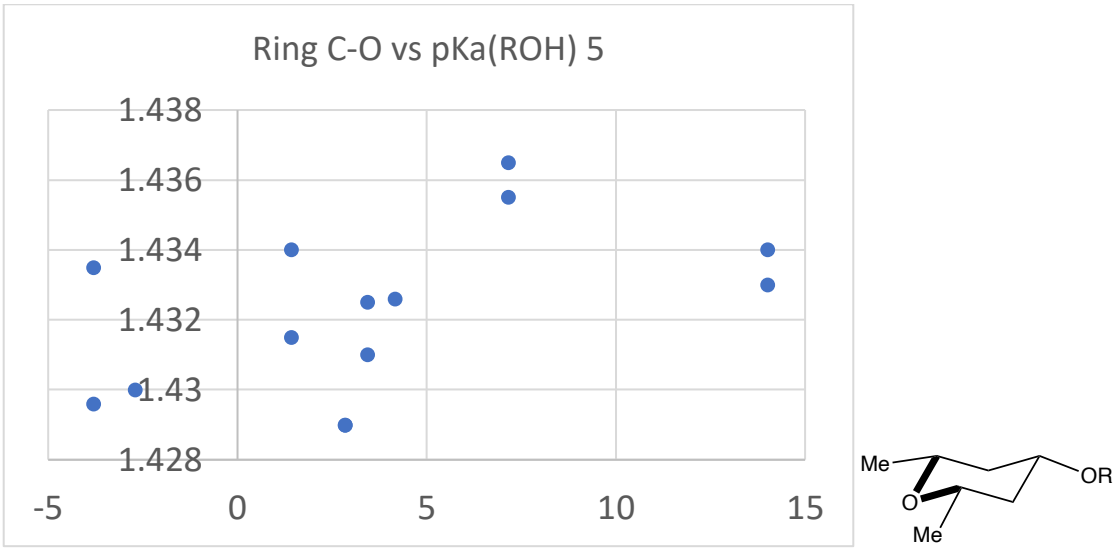
SI Figure S20



SI Figure S21



SI Figure S22



Synthetic procedures.
Procedure for preparation of 4a, 5a, 5h, 4d and 5d are adapted from references[1,2]

Preparation of alcohols 4a and 5a
Step 1:
To a solution of homoallylic alcohol (300 mg, 3.48 mmol) in toluene (7.0 mL) at 0 °C were added acetaldehyde (0.097 mL, 1.74 mmol) p-TSA (1.04 g, 5.58 mmol) then the reaction was slowly warm to room temperature and stirred at 70 °C for 3 h. The reaction mixture was diluted with brine solution and extracted with ethyl acetate (2 x 15 mL). The combined organic layers were washed with saturated potassium carbonate solution (15 mL), dried over sodium sulfate and evaporated the solvent to give crude product which was purified by silica gel column chromatography using 20% ethyl acetate-hexanes afforded Prins product (**5h**, 500 mg, 52%). The product was confirmed by ^{1}H NMR spectroscopy.**Error! Bookmark not defined.**,**Error! Bookmark not defined.**
Step 2:
To a solution of OTs compound (**5h**, 500 mg, 1.76 mmol) in MeOH (10 mL) was added Mg powder (105.6 mg, 4.40 mmol) at ambient temperature and the resulting mixture was continued to stir at the same temperature for 6 h. After completion of the reaction cooled to 0°C were added conc. HCl (3.0 mL) and water (10.0 mL) and extracted with diethyl ether (3 X 15 mL). The combined organic layers were dried over sodium sulfate, filtered and concentrated under vacuum to give crude product which was purified by silica gel column chromatography using 50% diethyl ether-hexanes obtained desired product (**5a**, 200 mg, 59%). The NMR spectra were identical to literature data.[1,2]
Step 3:
To a stirred solution of alcohol (**5a**, 200 mg, 1.53 mmol) in anhydrous benzene (6 mL) under argon atmosphere were added benzoic acid (1.15 g, 6.92 mmol) and triphenylphosphine (2.0 g, 7.67 mmol) at room temperature. After 5 min DAID (1.52 mL, 7.67 mmol) was added to the reaction mixture at the same temperature and allowed to continue for 24 h. Then the reaction mixture was concentrated as such (without further work-up) and subjected to silica gel column chromatography using 20% ethyl acetate-hexanes acquired the desired benzoate compound The product was confirmed by ^{1}H NMR spectroscopy.
Step 4:
A solution of benzoate above (350 mg, 1.25 mmol) in THF (18 mL) was added LiOH (263 mg, 6.27 mmol) in water (6 mL) at ambient temperature and the resulting mixture was continued to stir at the same temperature for 24 h. THF was removed under reduced pressure and the aqueous layer was diluted with more water (15 mL) and extracted with diethyl ether (3 X 20 mL). The combined organic layers were dried with sodium sulfate, filtered and concentrated under vacuum to give crude product, which was purified by silica gel column chromatography using 50% diethyl ether-hexanes obtained desired product (**4a**, 150 mg, 92%). The NMR spectra were identical to literature data.**Error! Bookmark not defined.**,**Error! Bookmark not defined.**

General procedure to prepare benzoates and sulfonates:
To a stirred solution of alcohol (**4a** or **5a**, 100 mg, 0.76 mmol) in pyridine (5 mL) was added acid chloride (293 mg, 1.53 mmol) at 0 °C. Then the reaction was continued to stir for 24 h. Pyridine was removed from the reaction then diluted with water (10 mL) and extracted with ethyl acetate (2 x 10 mL), then the organic layer was washed with aq. HC1 (2 x 10 mL). The organic layer was dried over sodium sulfate, filtered, concentrated under reduced pressure and purified by silica gel column chromatography using 5% ethyl acetate-hexanes gave desired product (**4c**, 120 mg, 55%). The products were confirmed by ^{1}H NMR spectroscopy.

Melting points: 4c 85-86.4°C**, 5c** 101-107°C, **4d** 103-104.5°C**, 5d** 93-95.3°C**, 4e** 181-182°C, **5e** 99.3-101.6°C, **4f** 62.2-63.7°C, **4h** 60.2-61.3°C, **5h** 85.2-87°C, **4i** 75.6-79.9°C.

General procedure to prepare p-nitrophenoxyl ethers

The alcohol **4a** or **5a** (100 mg) in dry ether (20 ml) was treated with excess potassium hydride (50 mg) and stirred at 0°C for 30 minutes before treatment with p-nitrofluorobenzene (1.05 eq.). After 2 hours the reaction was quenched carefully with water. Excess water was added, and the mixture extracted with ether (3 x 20 ml), dried ($MgSO_4$), and evaporated down to a residue.

Compound **4b**

^{1}H NMR (CDCl3, 400MHz): δ = 8.16 - 8.21 (m, 2 H) (Ar-**H**), 6.91 - 6.96 (m, 2 H) (Ar-**H**), 4.82 (bs 1H) (C**H**eq-OR), 3.87 - 3.96 (m, 2 H) (CO-C**H**ax-CR), 1.91 (dd, *J*=14.3, 2.7 Hz, 2 H) (RC-C**H**eq-CR), 1.52 (ddd, *J*=14.3, 11.5, 2.7 Hz, 2 H) (RC-C**H**ax-CR), 1.18 ppm (d, J = 6Hz 6H) (C**H**3) ^{13}C NMR (CDCl3, 101MHz): δ = 162.5, 141.3, 126.0, 115.2, 71.5, 67.9, 36.4, 31.9, 29.7, 21.8, 14.1 ppm

Compound **5b**

^{1}H NMR (CDCl3, 400MHz): δ = 8.12 - 8.17 (m, 2 H) (Ar-**H**), 6.90 - 6.95 (m, 2 H) (Ar-**H**), 4.47 - 4.56 (m, 1 H) (C**H**ax-OR), 3.56 (ddd, *J*=11.4, 6.0, 1.4 Hz, 2 H) (CO-C**H**ax-CR), 2.05 - 2.12 (m, 2 H) (RC-C**H**eq-CR), 1.29 - 1.40 (m, 2 H) (RC-C**H**ax-CR), 1.24 ppm (d, *J*=6.0 Hz, 6 H) (C**H**3) ^{13}C NMR (CDCl3, 101MHz): δ = 206.9, 162.6, 141.3, 125.9, 115.3, 74.2, 71.3, 38.7, 30.9, 29.7, 21.7 ppm

Compound 4f

^{1}H NMR (CDCl3, 400MHz): δ = 7.91 (d, *J*=8.2 Hz, 1 H) (Ar-**H**), 7.59 - 7.74 (m, 3 H) (Ar-**H**), 5.09 - 5.20 (m, 1 H) (C**H**eq-OR), 3.51 - 3.62 (m, 2 H) (CO-C**H**ax-CR), 2.08 (dd, *J*=13.1, 4.5 Hz, 2 H) (RC-C**H**eq-CR), 1.27 - 1.38 (m, 3 H) (RC-C**H**ax-CR), 1.22 - 1.26 ppm (d J = 6Hz, 6 H) (C**H**3) ^{13}C NMR (CDCl3, 101MHz): δ = 164.9, 148.1, 132.9, 131.6, 129.8, 127.9, 123.9, 72.9, 71.3, 59.5, 38.1, 31.2, 29.7, 21.7 ppm

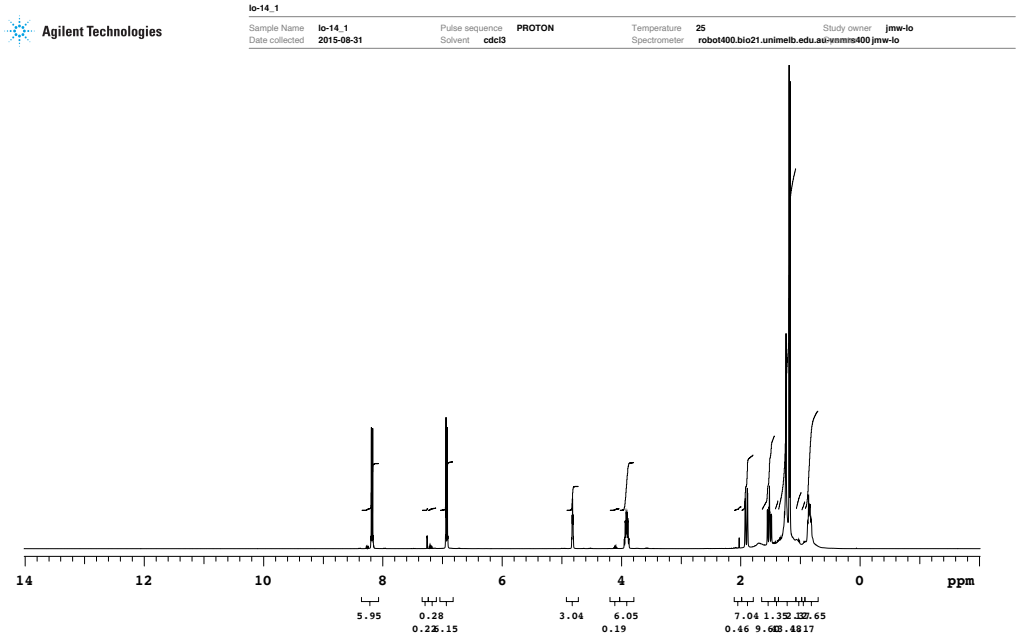
Compound 4g

^{1}H NMR (CDCl3, 400MHz): δ = 8.77 (d, *J*=2.3 Hz, 1 H) (Ar-**H**), 8.51 - 8.56 (m, 1 H) (Ar-**H**), 7.98 (d, *J*=8.6 Hz, 1 H) (Ar-**H**), 5.46 - 5.51 (m, 1 H) (C**H**eq-OR), 3.67 - 3.77 (m, 2 H) (CO-C**H**ax-CR), 1.91 (dd, *J*=14.9, 2.3 Hz, 2 H) (RC-C**H**eq-CR), 1.52 - 1.60 (m, 2 H) (RC-C**H**ax-CR), 1.17 - 1.22 ppm (d J = 6Hz, 6 H) (C**H**3) ^{13}C NMR (CDCl3, 101MHz): δ = 162.9, 132.9, 131.4, 127.3, 124.4, 119.5, 72.4, 68.3, 36.5, 31.4, 29.7, 21.8 ppm

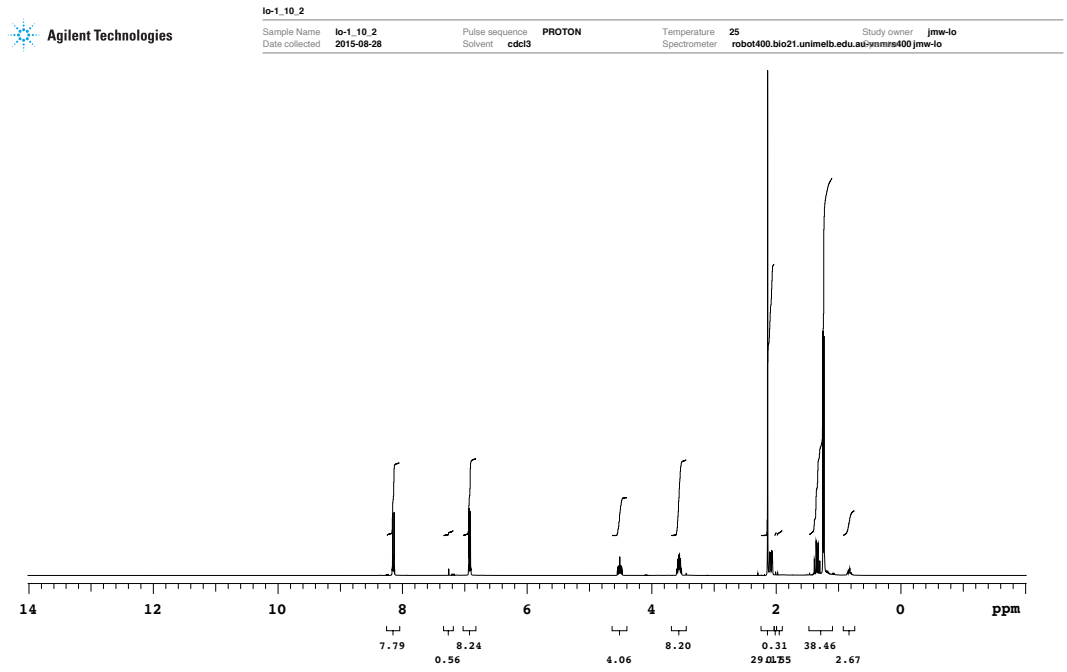
Compound 5g 78%

^{1}H NMR (CDCl3, 400MHz): δ = 8.76 - 8.79 (m, 1 H) (Ar-**H**), 8.52 (dt, *J*=8.2, 1.8 Hz, 1 H) (Ar-**H**), 7.91 (dd, *J*=8.2, 1.2 Hz, 1 H) (Ar-**H**), 5.13 - 5.23 (m, 1 H) (C**H**ax-OR), 3.51 - 3.62 (m, 2 H) (CO-C**H**ax-CR), 2.05 - 2.12 (m, 2 H) (RC-C**H**eq-CR), 1.27 - 1.38 (m, 2 H) (RC-C**H**ax-CR), 1.21 - 1.26 ppm (d, J = 6Hz, 6 H) (C**H**3) ^{13}C NMR (CDCl3, 101MHz): δ = 163.2, 148.9, 148.0, 133.1, 131.2, 127.5, 119.6, 74.0, 71.3, 38.0, 21.7 ppm

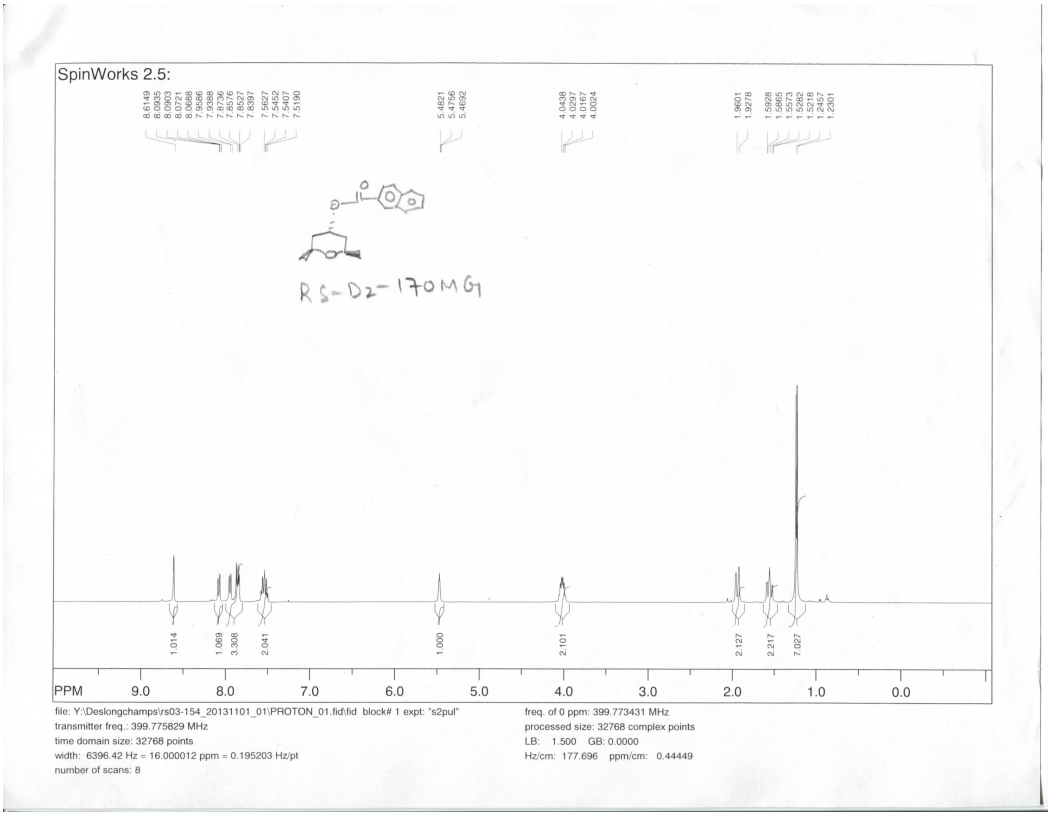
Compound 4b



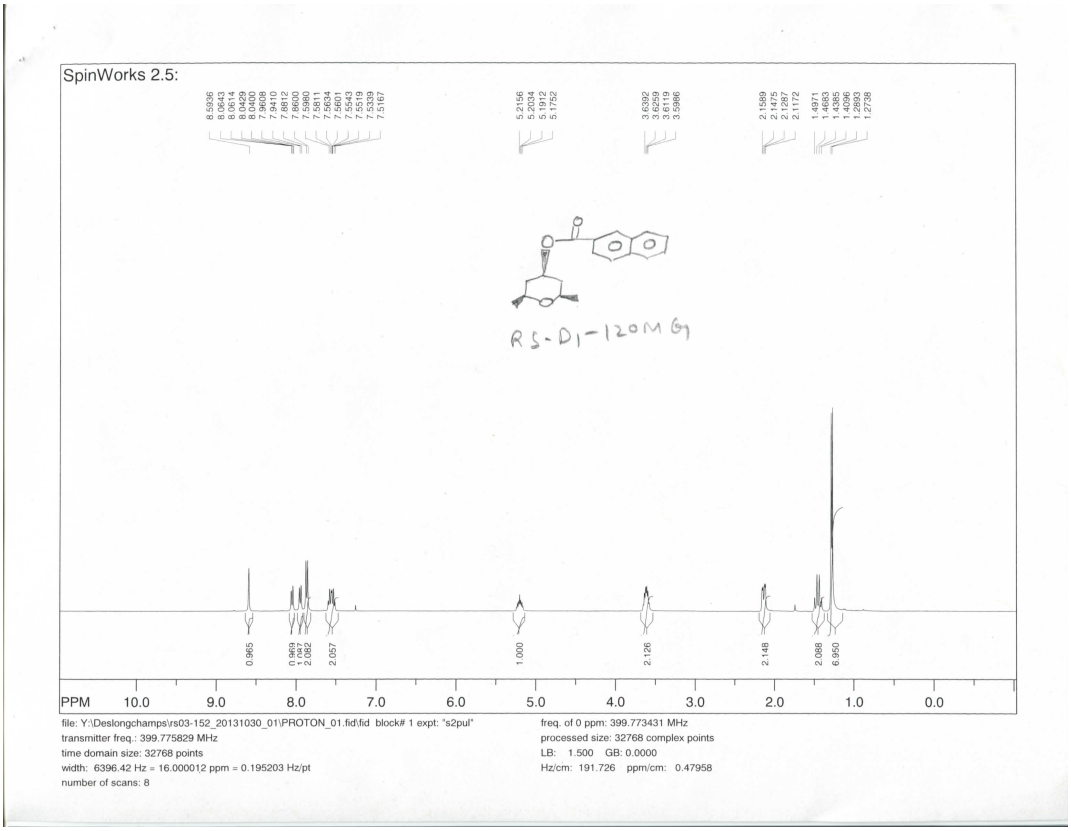
Compound 5b



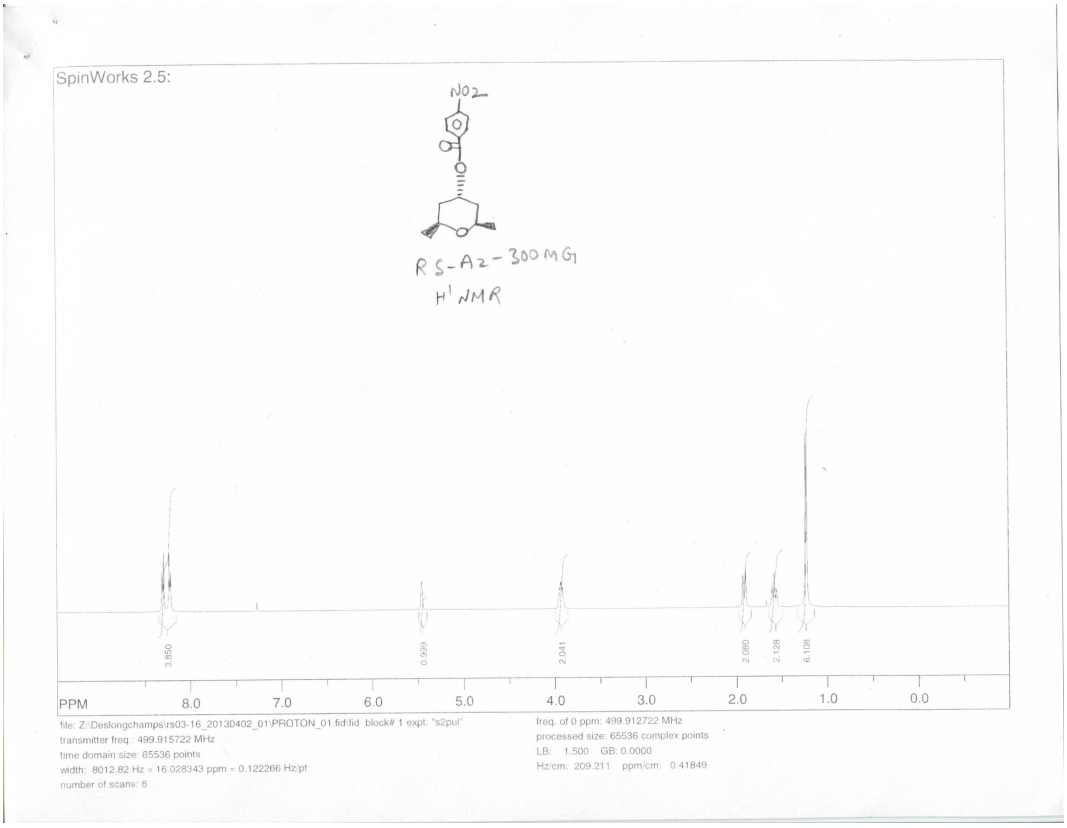
Compound 4c



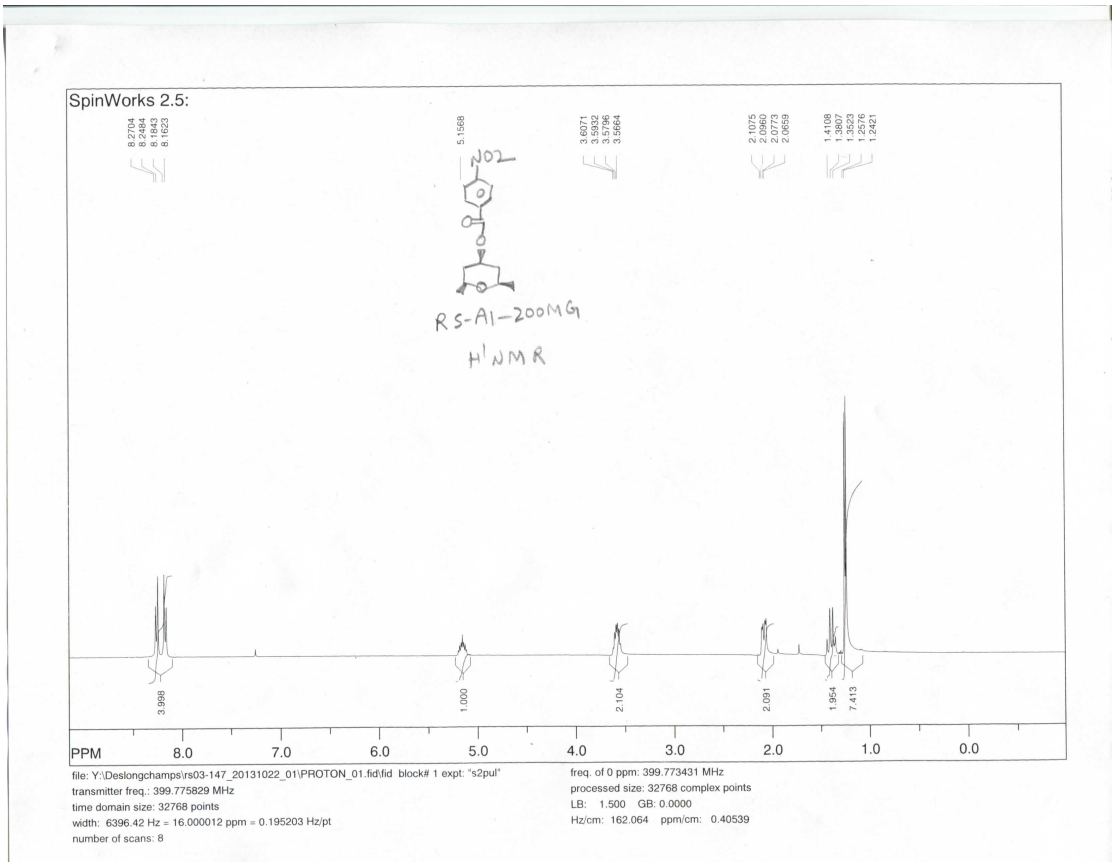
Compound 5c



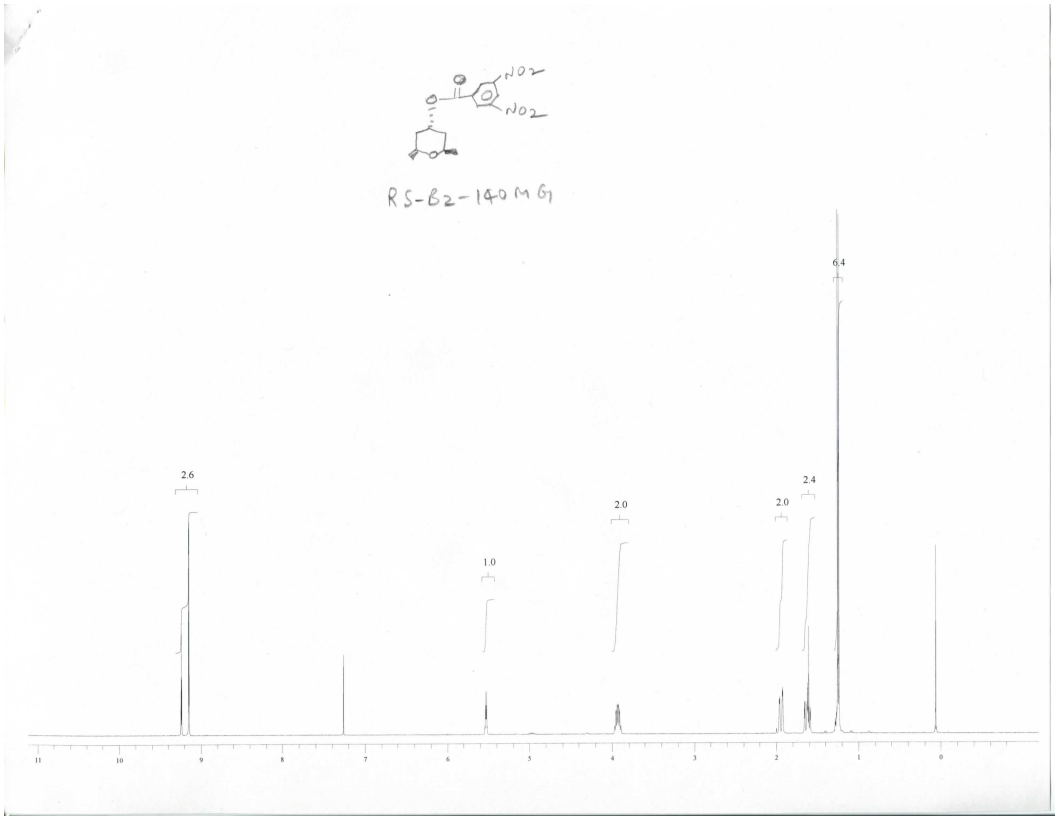
Compound 4d



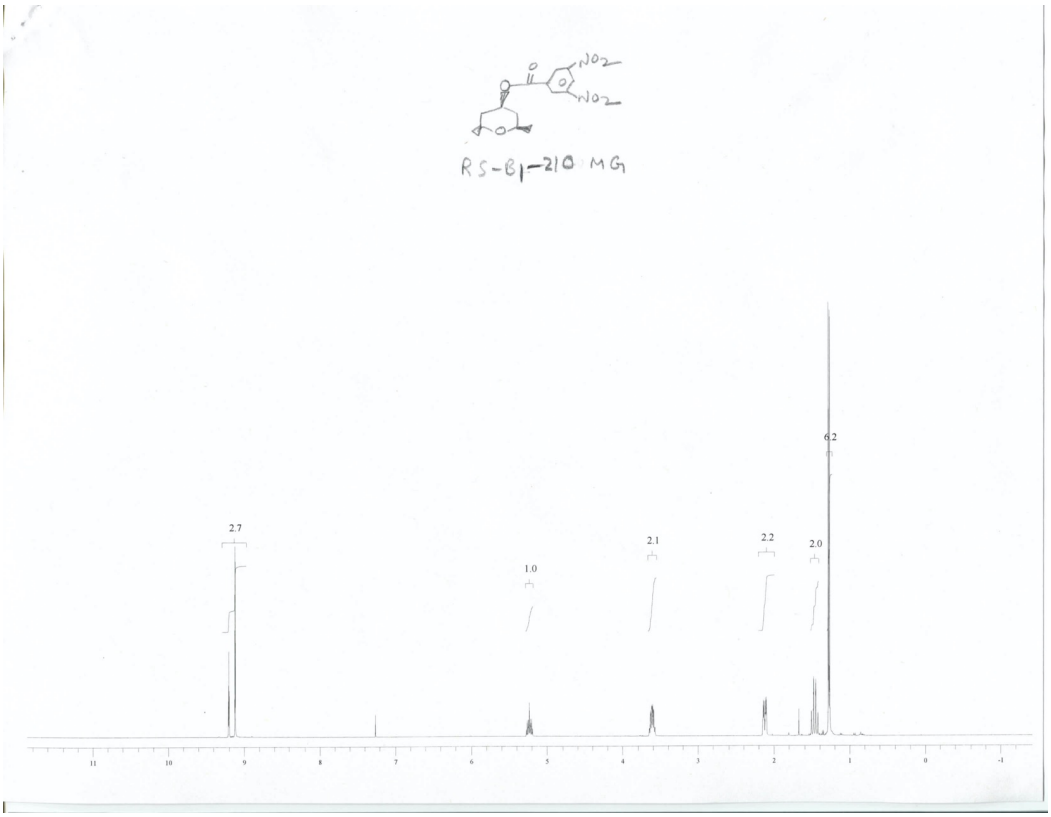
Compound 5d



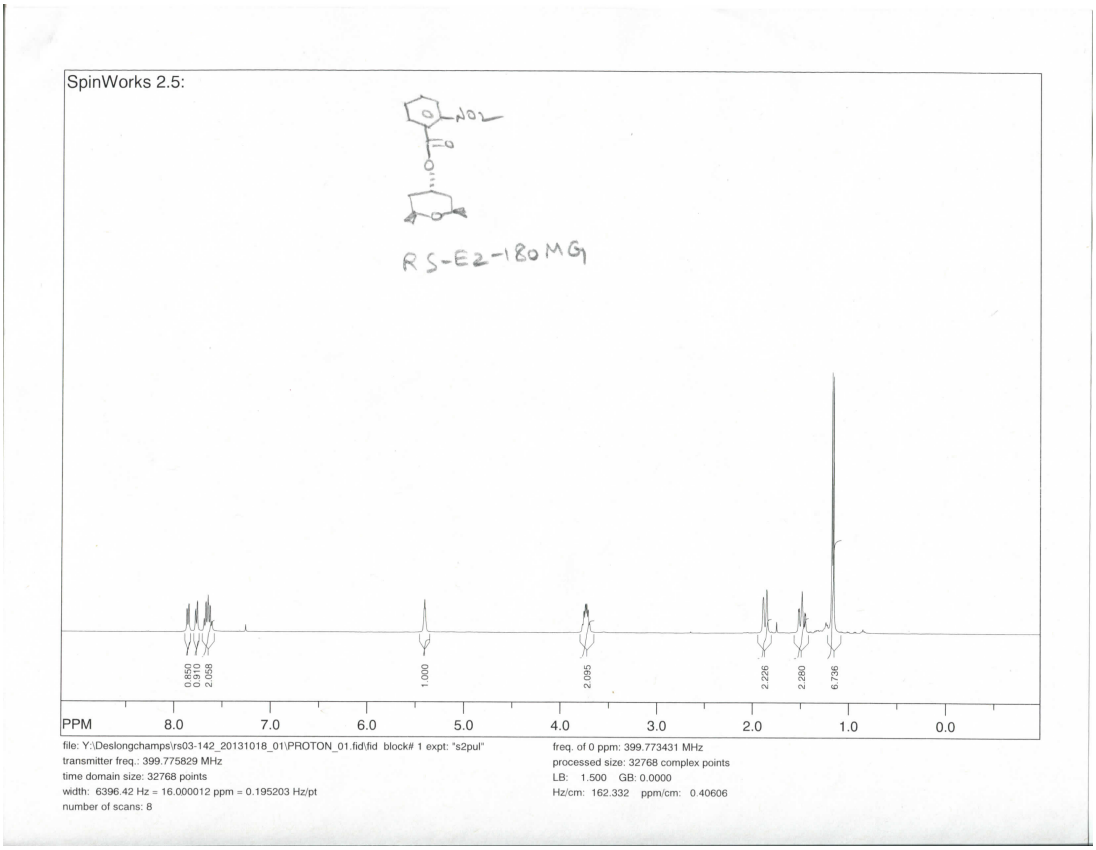
Compound 4e



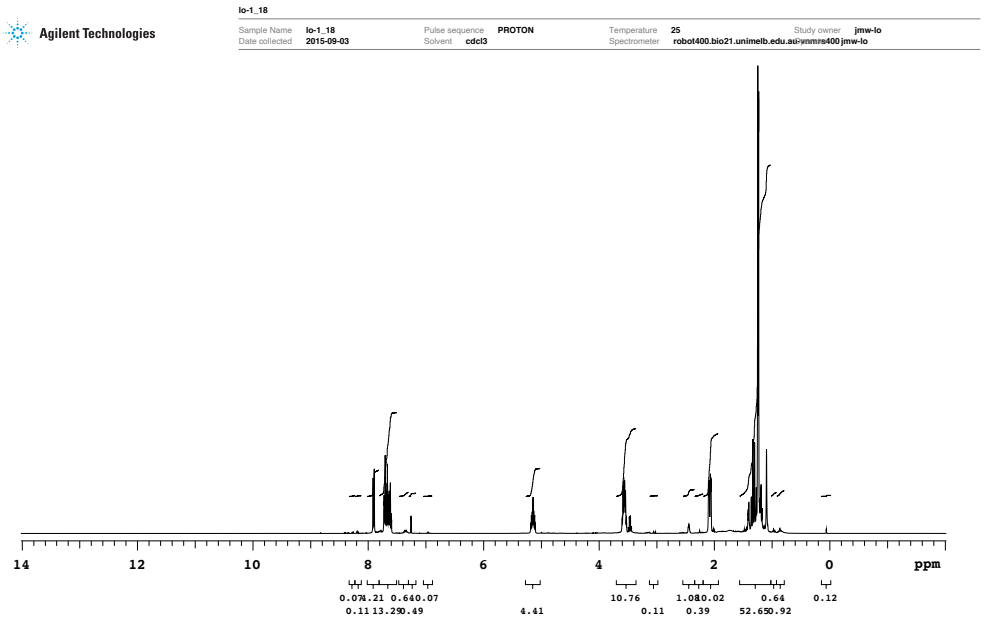
Compound 5e



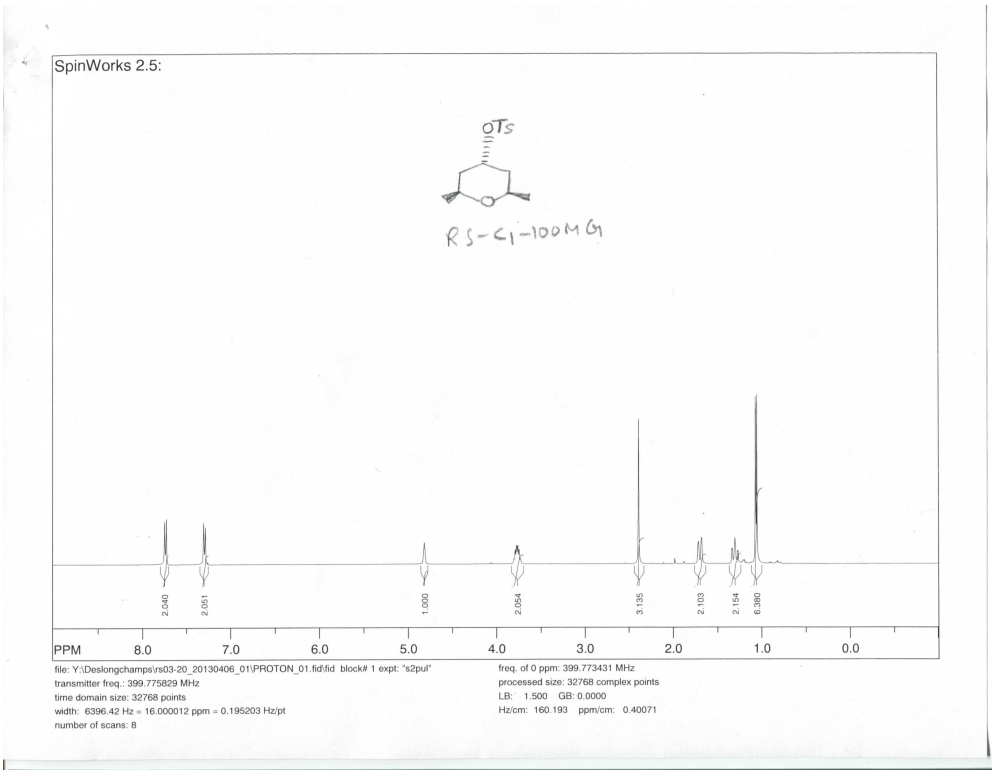
Compound 4f



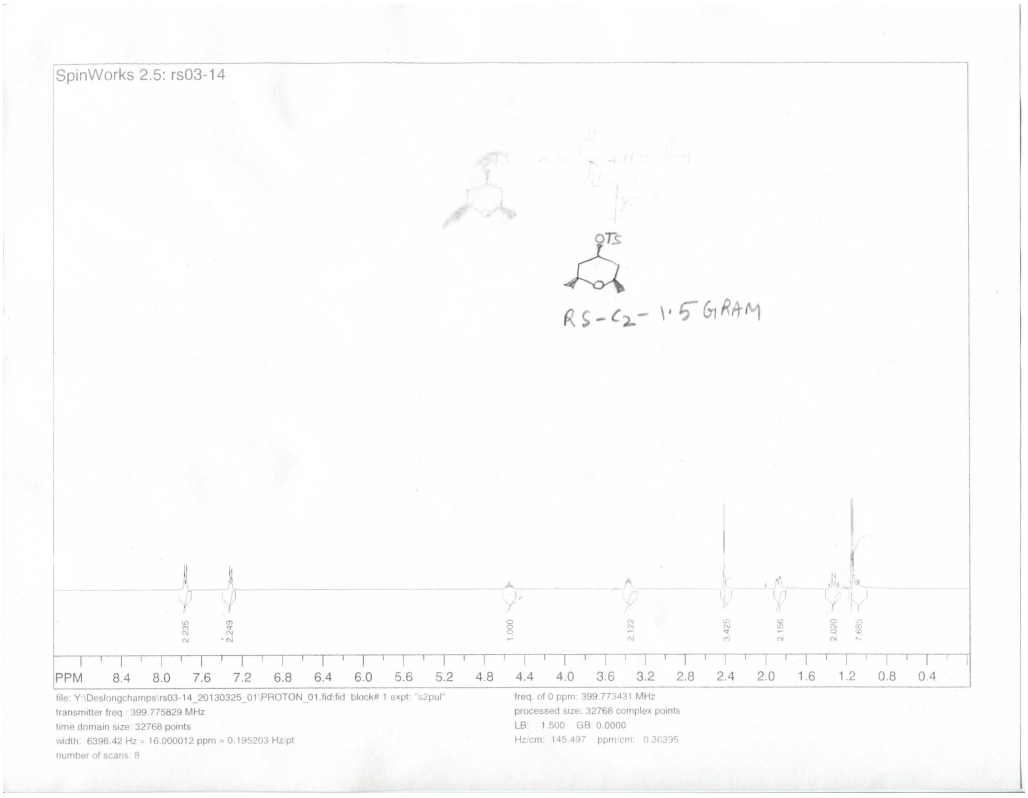
Compound 5f



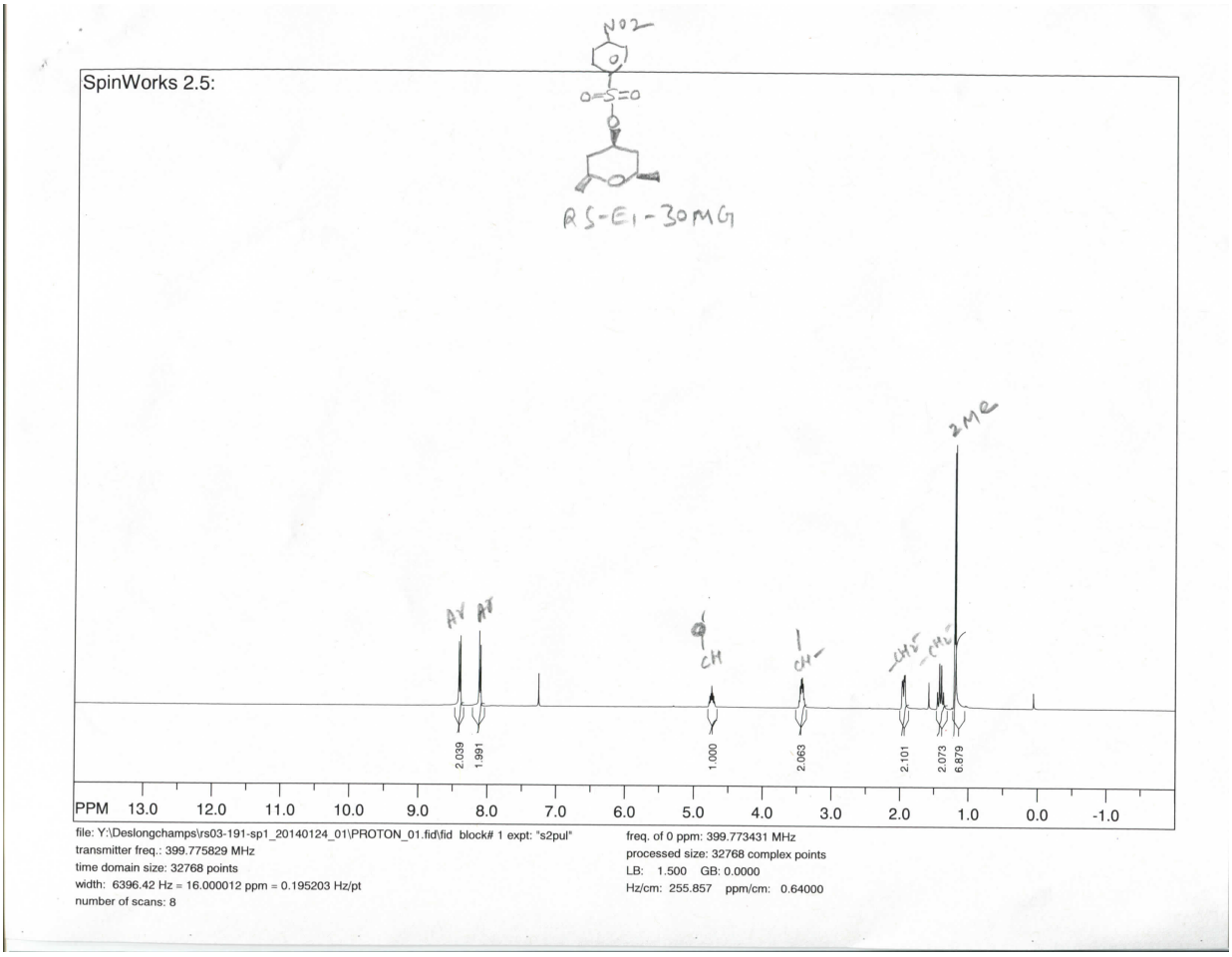
Compound 4h



Compound 5h



Compound 5i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2.073
6.879
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transmitter freq.: 399.775829 MHz
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width: 6396.42 Hz = 16.000012 ppm = 0.195203 Hz/pt
number of scans: 8
freq. of 0 ppm: 399.773431 MHz
processed size: 32768 complex points
LB: 1.500 GB: 0.0000
Hz/cm: 255.857 ppm/cm: 0.64000

Gaussian Cartesian Coordinates and Energies Calculated at B3LYP/6-311++G(d,p):
Energies given as sum of electronic and zero-point vibrational energies (E), sum of electronic and thermal enthalpies (H), sum of electronic and thermal Free energies (G), and zero-point vibrational energy (ZPVE) in Hartrees.

4

E = -425.546958
H = -425.536438
G = -425.580672
ZPVE = 0.205259

Symbol	X	Y	Z
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C	-0.02213100	1.59409600	0.42974500
H	-0.03219900	2.38116000	1.19534400
C	-1.18691600	-0.54752500	-0.28480000
H	-1.17244300	-0.24502800	-1.34266000
C	-1.26779100	0.71131400	0.58630900
H	-2.16847400	1.28524500	0.33560700
H	-1.35981200	0.41017500	1.63661700
C	1.20688300	-0.50284600	-0.28921600
H	1.17809000	-0.20111800	-1.34596900
C	1.24855900	0.75774600	0.58104000
H	1.35399400	0.45958400	1.63015200
H	2.11471300	1.37113500	0.31662500
C	-2.34116300	-1.50850700	-0.04896100
H	-2.23641100	-2.39104900	-0.68355300
H	-3.29609000	-1.02737900	-0.27853700
H	-2.35895900	-1.83663900	0.99401100
C	2.39343900	-1.42336100	-0.05540200
H	2.42687100	-1.74802300	0.98839500
H	3.32928100	-0.90873600	-0.28981500
H	2.31760000	-2.31045200	-0.68784300
H	-0.75561400	2.73945900	-0.99576000

4-H_2O^+
E = -425.851573
H = -425.840257
G = -425.886039
ZPVE = 0.216107

Symbol	X	Y	Z
O	-0.00696300	2.28310400	-0.82716700
O	0.01129800	-1.27061900	-0.00507800
C	-0.00725000	1.48682200	0.63234800
H	-0.01463000	2.33311800	1.31807200
C	-1.18916800	-0.55922800	-0.29456700
H	-1.16105700	-0.23030200	-1.34482700
C	-1.27530300	0.67726500	0.62151600
H	-2.14922400	1.29924000	0.39817900
H	-1.40384200	0.34140200	1.65921300
C	1.20508300	-0.54505100	-0.28641900
H	1.18031200	-0.21645600	-1.33687100
C	1.27040800	0.69239100	0.63018100
H	1.39566600	0.35799100	1.66873900
H	2.13839300	1.32475900	0.41292800
C	-2.35847600	-1.50637200	-0.09173500
H	-2.25882400	-2.36551400	-0.75586000
H	-3.30576700	-1.00812500	-0.31289900
H	-2.38459700	-1.86978400	0.93826200
C	2.38424200	-1.47814000	-0.07550700
H	2.40766600	-1.84114000	0.95469900
H	3.32698000	-0.96857800	-0.29026800
H	2.29942400	-2.33845600	-0.74017200
H	0.78502500	2.82884300	-0.97508300
H	-0.80291100	2.82175700	-0.97971600

5

E = -425.547872
H = -425.537276
G = -425.581692
ZPVE = 0.205119

Symbol	X	Y	Z
O	1.33495800	0.00266500	-0.14184000
O	-2.87026400	-0.00557700	-0.11326800
C	0.71099900	1.19945000	0.33252000
H	0.74306000	1.19036200	1.43516500
C	-0.75673400	1.25248800	-0.11297000
H	-1.24550400	2.14224100	0.29538600
H	-0.78646000	1.32875100	-1.20805400
C	-1.51080400	-0.00294100	0.32951500
H	-1.58938000	-0.00319900	1.42200300
C	0.71568200	-1.19664100	0.33232600
H	0.74768900	-1.18758900	1.43497400
C	-0.75182500	-1.25533300	-0.11321600
H	-0.78124600	-1.33150400	-1.20830700
H	-1.23714100	-2.14705500	0.29495800
C	1.53467200	2.37019800	-0.17968400
H	2.56866400	2.28678000	0.16132600
H	1.12347200	3.31585400	0.18331700
H	1.53396300	2.38701400	-1.27277600
C	1.54395000	-2.36407300	-0.18002400
H	1.54318400	-2.38087700	-1.27311600
H	1.13656600	-3.31136900	0.18300200
H	2.57763900	-2.27653700	0.16087200
H	-2.87699300	-0.00437900	-1.07704900

5-H_2O^+

E = -425.854075
H = -425.842602
G = -425.888806
ZPVE = 0.216351

Symbol	X	Y	Z
O	-1.36930800	0.01969500	-0.17725900
O	2.92916800	-0.10150500	-0.15621200
C	-0.81321100	-1.17866600	0.34075800
H	-0.86752200	-1.15901700	1.43920600
C	0.69172800	-1.26924200	-0.08069000
H	1.14940600	-2.15892900	0.35515400
C	1.35241600	-0.01474800	0.39521600
H	1.58400000	-0.02258600	1.45938100
C	-0.78327300	1.20325600	0.33717800
H	-0.83552600	1.18812400	1.43592300
C	0.72439900	1.25397600	-0.08919800
H	0.75258500	1.32061100	-1.18077100
H	1.19439200	2.14037300	0.34439900
C	-1.61440400	-2.35319700	-0.19238800
H	-2.65373800	-2.26020800	0.12490900
H	-1.21985200	-3.29626800	0.19198200
H	-1.58790500	-2.37518500	-1.28399700
C	-1.55407200	2.39744600	-0.19778800
H	-1.52730600	2.41679900	-1.28940600
H	-1.13637600	3.33119100	0.18511100
H	-2.59535900	2.33100800	0.11976400
H	2.98280500	-0.31003600	-1.10558700
H	0.71517500	-1.34077700	-1.17329200
H	3.45458300	0.69955500	0.01946600

6

E = -389.609996
H = -389.599237
G = -389.643880
ZPVE = 0.229258

Symbol	X	Y	Z
O	-0.04660100	2.18113200	-0.79413400
C	0.01290400	1.54933200	0.49751500
H	0.01299800	2.32857400	1.27244400
C	-1.27429000	-0.51513800	-0.28956800
H	-1.27134000	-0.14557400	-1.32186900
C	-1.25674500	0.71001100	0.63968200
H	-2.12439800	1.34823900	0.44566500
H	-1.33132200	0.37525500	1.68268100
C	1.28684100	-0.53508000	-0.27857800
H	1.29533000	-0.16890500	-1.31270700
C	1.27550900	0.69064900	0.64968800
H	1.34054300	0.35690400	1.69432900
H	2.16431700	1.31022100	0.47150600
C	-2.54191000	-1.35023000	-0.08367000
H	-2.56893200	-2.20445500	-0.76712500
H	-3.44293700	-0.75445800	-0.25880700
H	-2.59478900	-1.74103000	0.93883600
C	2.54133800	-1.38767100	-0.06333500
H	2.57935000	-1.78116000	0.95859100
H	3.45332100	-0.80542600	-0.22897500
H	2.56203400	-2.24085800	-0.74814100
H	0.74148800	2.72213500	-0.90701800
C	-0.00097300	-1.35307400	-0.08650200
H	-0.00341000	-2.20384900	-0.77818300
H	-0.00791800	-1.77947200	0.92819400

6-H_2O^+

E = -389.921079
H = -389.909514
G = -389.955751
ZPVE = 0.240467

Symbol	X	Y	Z
O	0.01740500	2.24446000	-0.85494100
C	0.00764000	1.47899500	0.62209000
H	0.00903900	2.34090500	1.28897400
C	-1.28172300	-0.53434900	-0.29611000
H	-1.28278600	-0.17697100	-1.33414400
C	-1.27640500	0.69578100	0.63207300
H	-2.13658800	1.35213600	0.45394900
H	-1.38293900	0.36961400	1.67738400
C	1.28513400	-0.54671700	-0.28550600
H	1.29822100	-0.18943600	-1.32349100
C	1.28397400	0.68346300	0.64262200
H	1.37864800	0.35637400	1.68878400
H	2.15188800	1.33150000	0.47166400
C	-2.55253500	-1.36199000	-0.07993000
H	-2.57996400	-2.21113500	-0.76598300
H	-3.45466700	-0.76841700	-0.25179000
H	-2.59814600	-1.75617800	0.94000200
C	2.54610600	-1.38650400	-0.05877900
H	2.57945000	-1.78101000	0.96150400
H	3.45529400	-0.80162900	-0.22312800
H	2.57110700	-2.23593600	-0.74456700
H	0.81486800	2.77925400	-1.01106500
H	-0.77295200	2.78786600	-1.01732400
C	-0.00310300	-1.36137100	-0.08292400
H	-0.00433400	-2.21127400	-0.77094300
H	-0.00932100	-1.78661800	0.93016300

7

E = -389.610813
H = -389.599988
G = -389.644788
ZPVE = 0.229204

Symbol	X	Y	Z
O	-0.00064600	2.86140400	0.08450400
C	-1.28224000	-0.71237800	-0.33471200
H	-1.29483100	-0.71565300	-1.43420400
C	-1.26326100	0.75579000	0.12511200
H	-2.14425700	1.28827600	-0.24714600
C	-0.00033700	1.49083800	-0.33113700
H	-0.00034400	1.54900400	-1.42499100
C	1.28255500	-0.71179800	-0.33470700
H	1.29514300	-0.71506200	-1.43420000
C	1.26291200	0.75635700	0.12513000
H	1.31273200	0.78822900	1.22420900
H	2.14367000	1.28924700	-0.24711300
C	-2.54180900	-1.43798100	0.14997900
H	-2.56787600	-2.47093400	-0.20987900
H	-3.44893500	-0.93793900	-0.20222200
H	-2.58052500	-1.46590900	1.24436800
C	2.54245000	-1.43683800	0.14997500
H	2.58118300	-1.46475300	1.24436400
H	3.44934900	-0.93638500	-0.20222400
H	2.56898400	-2.46977800	-0.20988600
H	-0.00098000	2.88666200	1.04792300
H	-1.31310500	0.78764100	1.22419300
C	0.00031800	-1.43048900	0.11772800
H	0.00055300	-2.45939300	-0.26049900
H	0.00033100	-1.50412500	1.21511100

7-H_2O^+

E = -389.922171
H = -389.910533
G = -389.956983
ZPVE = 0.240882

Symbol	X	Y	Z
O	-2.81414500	-0.74147400	0.11460100
C	1.06409400	-1.06019600	-0.33994700
H	1.08448700	-1.07274200	-1.43731500
C	-0.39046900	-1.40067900	0.10364700
H	-0.68976400	-2.37228800	-0.29548500
C	-1.29360800	-0.31052800	-0.39064200
H	-1.48372700	-0.36104300	-1.46195700
C	0.45983600	1.43391500	-0.33689500
H	0.46987800	1.45491500	-1.43431600
C	-0.98791600	1.06920200	0.11269100
H	-1.03121800	1.09567400	1.20776300
H	-1.69197500	1.80745200	-0.28316200
C	2.03323000	-2.13274800	0.16683300
H	3.04764000	-1.92077200	-0.17771200
H	1.75945100	-3.12555900	-0.19835500
H	2.05153700	-2.16455500	1.26035200
C	0.83204300	2.83060000	0.17048700
H	0.83490600	2.86633400	1.26397700
H	0.13499600	3.58950700	-0.19348500
H	1.83088300	3.10566600	-0.17485500
H	-2.83453400	-1.00653000	1.05090000
H	-0.41292700	-1.44709400	1.20022900
H	-3.48590300	-0.05216900	-0.03248500
C	1.44364900	0.35136700	0.13314200
H	2.44456600	0.59483300	-0.23398400
H	1.50563900	0.36534700	1.22956700

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