Supporting Information

Synthesis and Desymmetrization of *Meso*-Tricyclic Systems Derived from Benzene Oxide

Desirée M. Matías and Jeffrey S. Johnson*

Department of Chemistry, University of North Carolina at Chapel Hill, Chapel Hill, NC 27599-3290, United States

jsj@unc.edu

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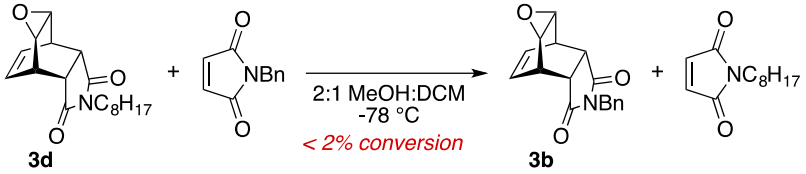
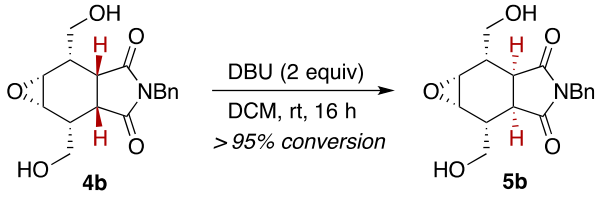
Mechanistic Experiments

Crossover experiment



A solution of **3d** (25.0 mg, 0.08 mmol) in dichloromethane (0.5 mL) was cooled to -78 °C. *N*-benzyl maleimide (15.4 mg, 0.08 mmol) was added and the reaction was stirred for 20 min. The reaction was moved to an ice bath and stirred for 1 h. The solvent was evaporated under reduced pressure and the crude oil analyzed. Only returned starting materials were observed, indicating that no retro Diels-Alder reaction had taken place.

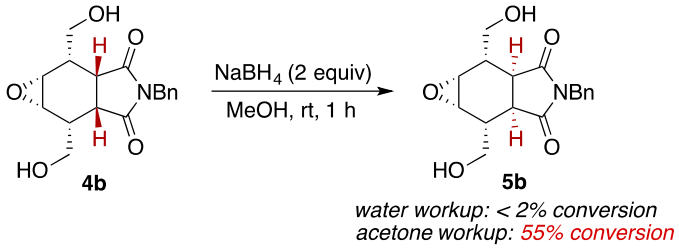
Double epimerization experiments



To a solution of **4b** (10 mg, 0.06 mmol) in dichloromethane (0.1 mL) in a flame-dried 1 dram vial was added DBU (8 µL, 0.05 mmol) and the reaction was allowed to stir at room temperature for 16 h. A sat. aq. $NaHCO_3$ solution (1 mL) was added and the solution was extracted with EtOAc (3 x 1 mL). The organics were combined, dried with sodium sulfate and concentrated. Full conversion to the opposite diastereoisomer was observed by ^1H NMR spectroscopy.

Table S1: Effect of base:

Entry	Base	Conversion to **5b**
1	Et_3N	< 2%
2	$NaHCO_3$	< 2%
3	K_2CO_3	21%

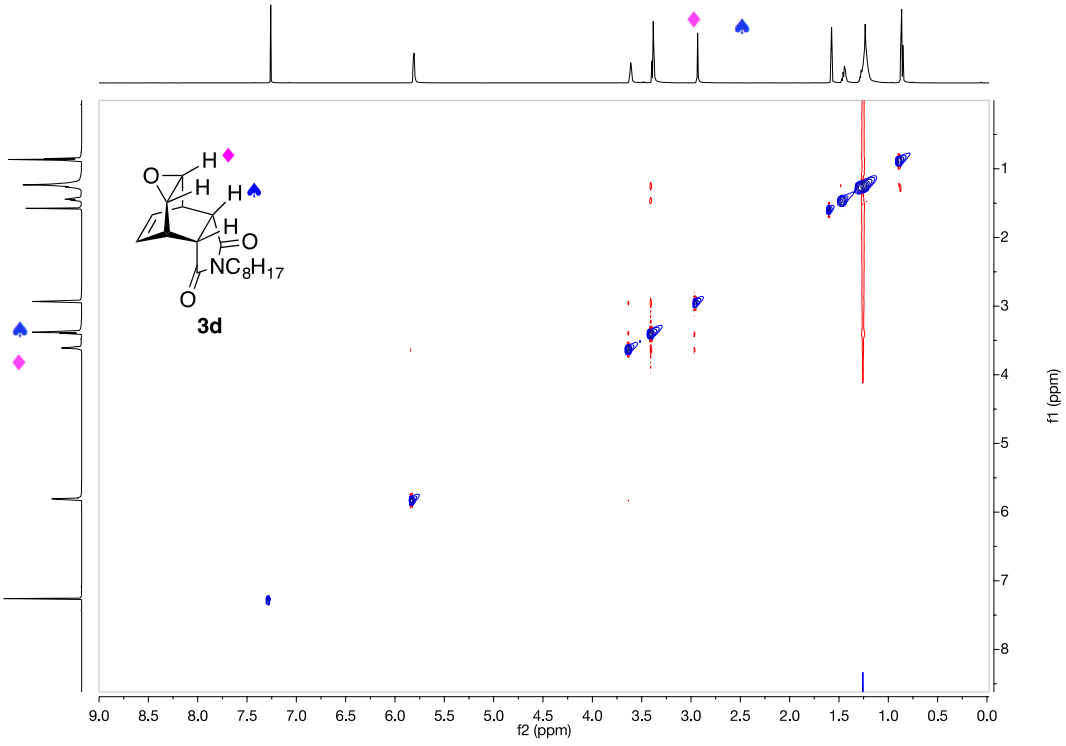


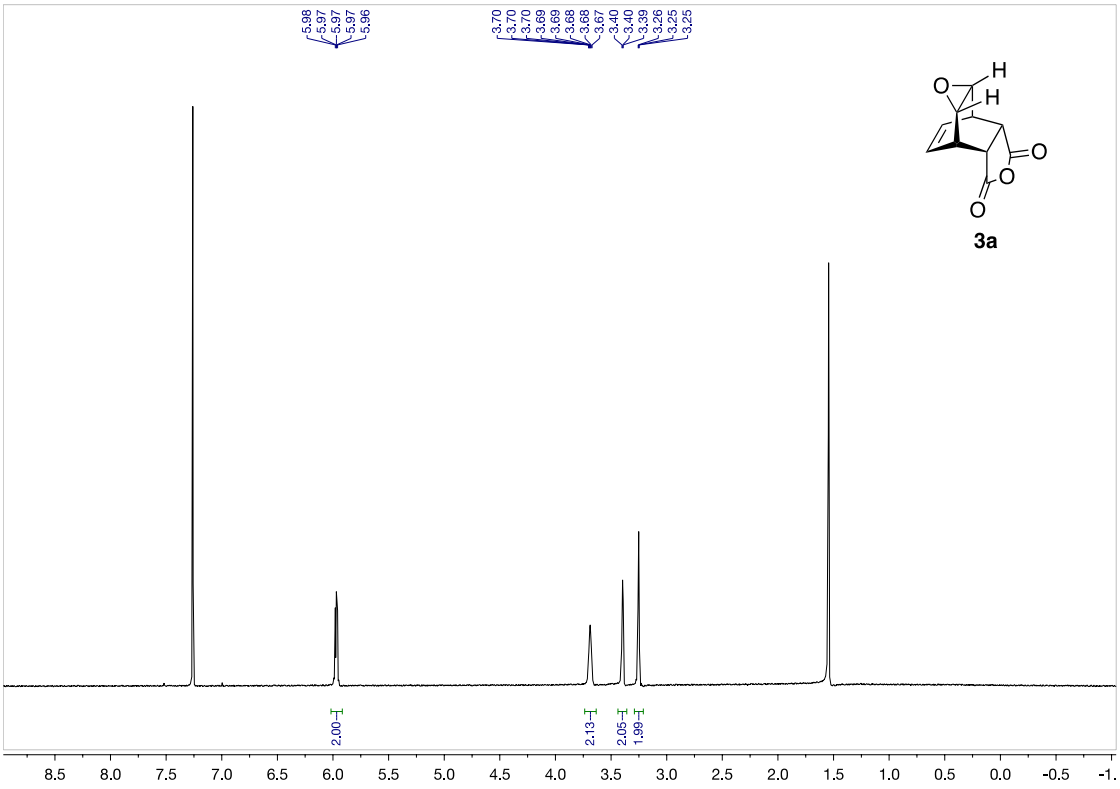
To a solution of **4b** (30.0 mg, 0.09 mmol) in methanol (0.7 mL) was added $NaBH_4$ (7.15 mg, 0.19 mmol) at room temperature and the reaction was allowed to stir for 1 h.

Water workup: 1 mL of water and 1 mL of ethyl acetate were added and the layers were separated. The aqueous layer was extracted using ethyl acetate (3 x 2 mL) and the organics were combined, dried with sodium sulfate and concentrated. Only **4b** was observed by ^{1}H NMR.

Acetone workup: 1 mL of acetone was added and the solvent was removed under reduced pressure. The residue was passed through a silica gel plug using ethyl acetate and the solvent was removed to obtain a 1:1.2 mixture of diastereoisomers, favoring **5b**.

nOe experiment for 3d

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H
H
O
O
O
O
3a
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2.13
2.05
1.99
8.5
8.0
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7.0
6.5
6.0
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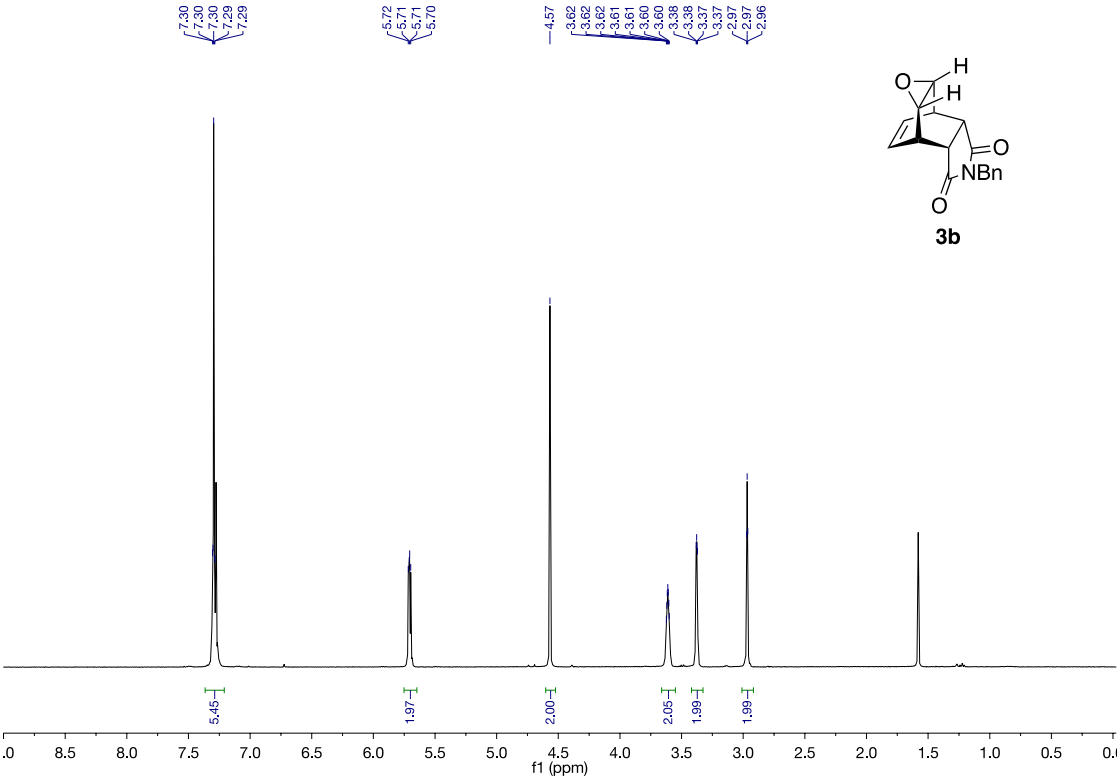
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3.61
3.61
3.60
3.60
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H
H
O
O
NBn
O
3b
5.45
1.97
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2.05
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1.99
8.5
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6.0
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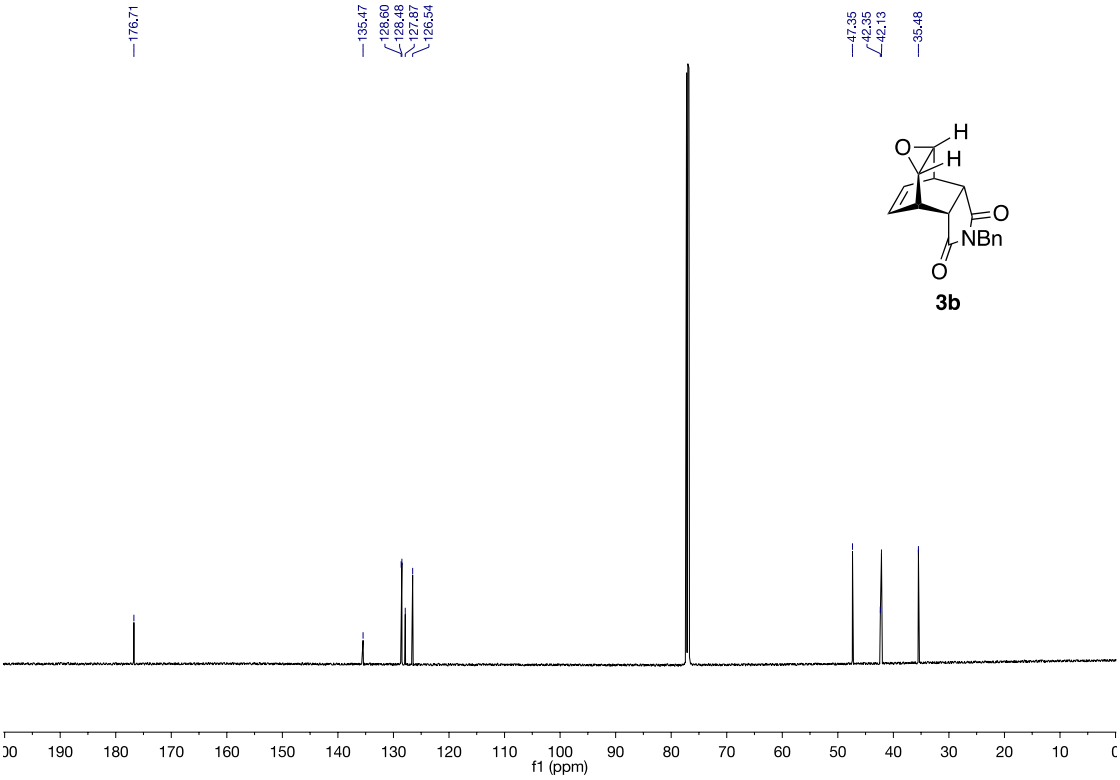
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O
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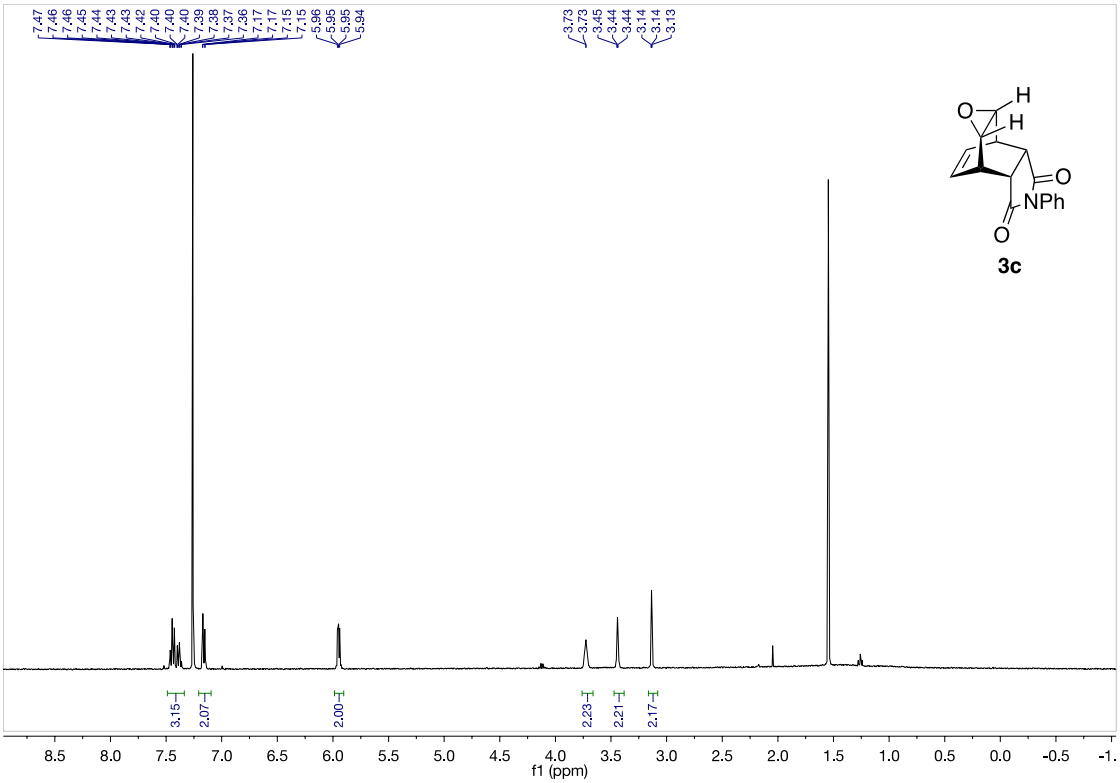
O
3b
f1 (ppm)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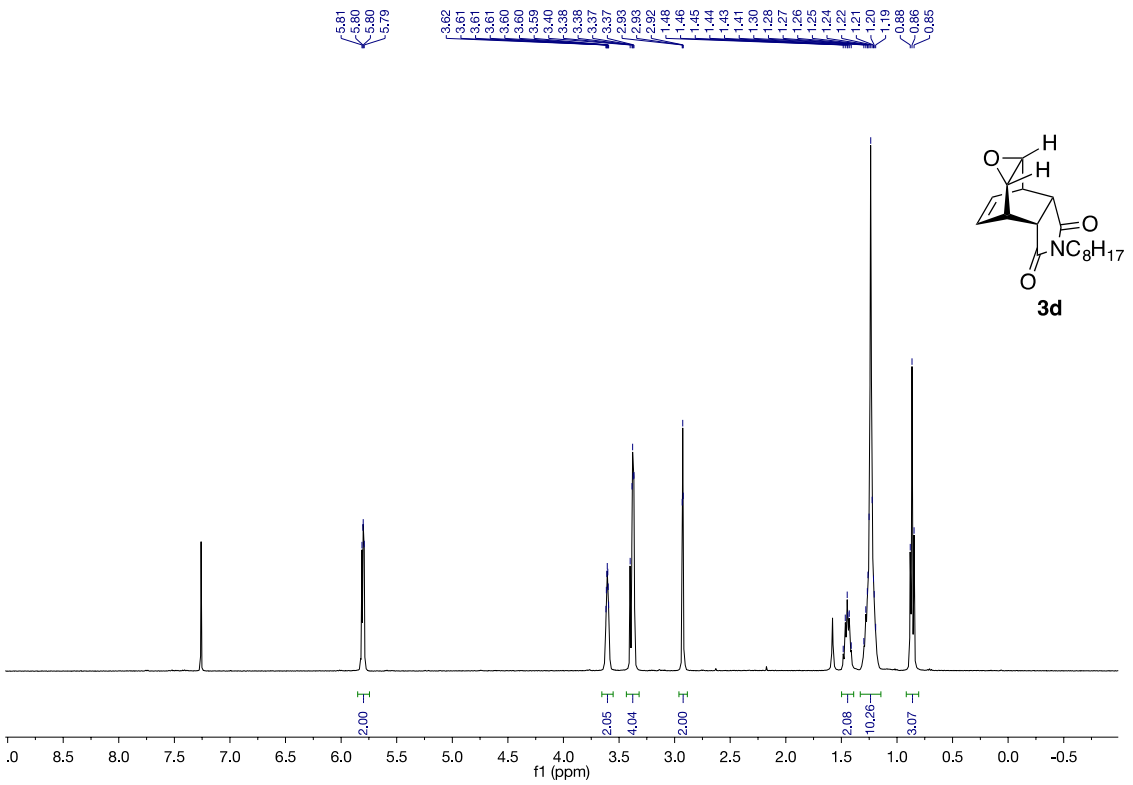
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H
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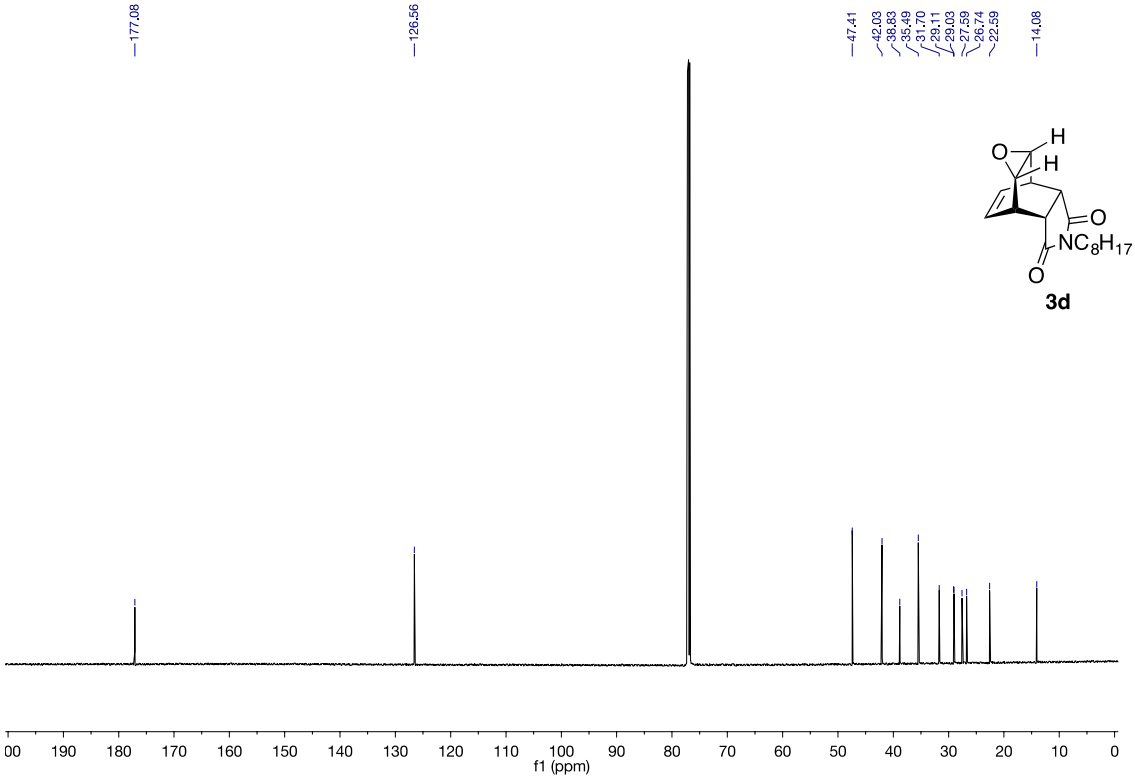
f1 (ppm)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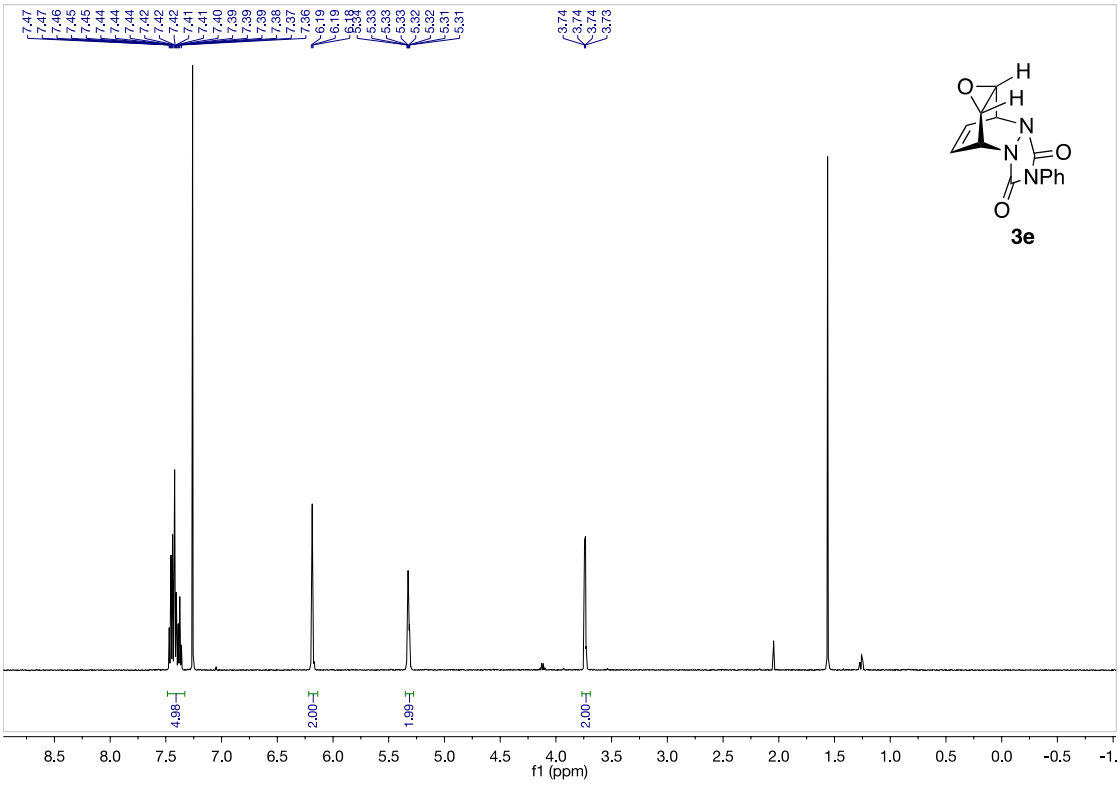
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2.00
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H
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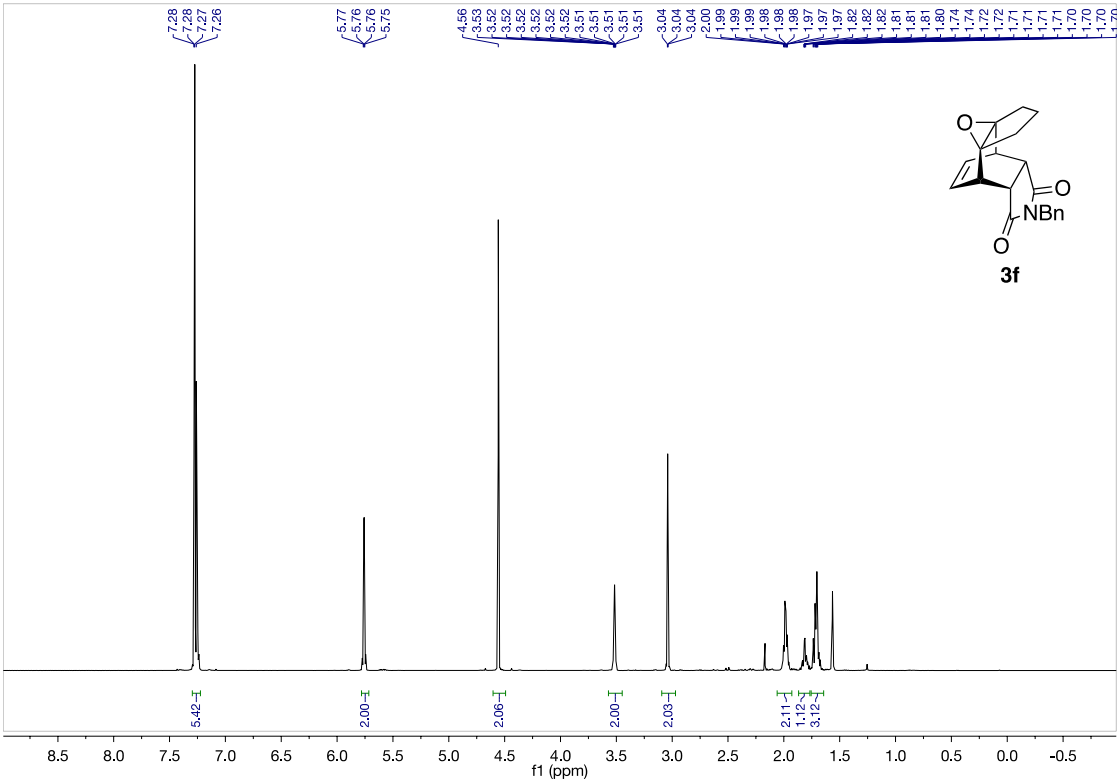
NPh
O
3e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2.00
2.03
2.11
1.12
3.12
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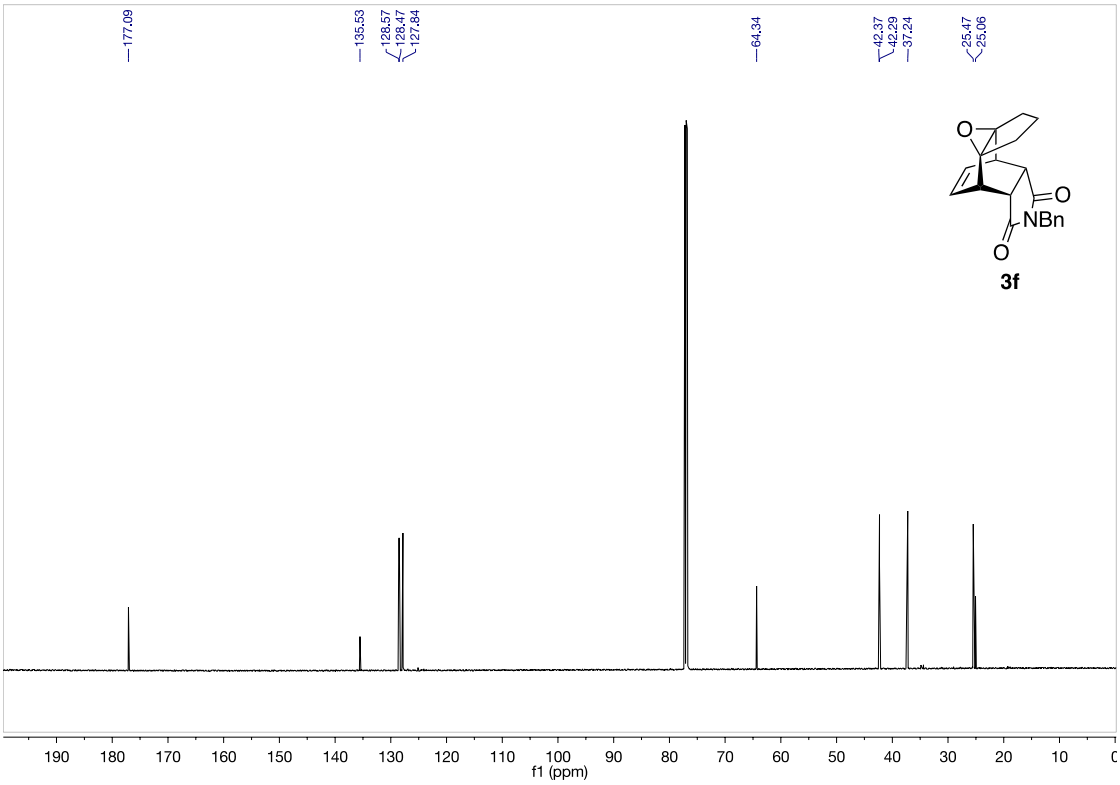
O
3f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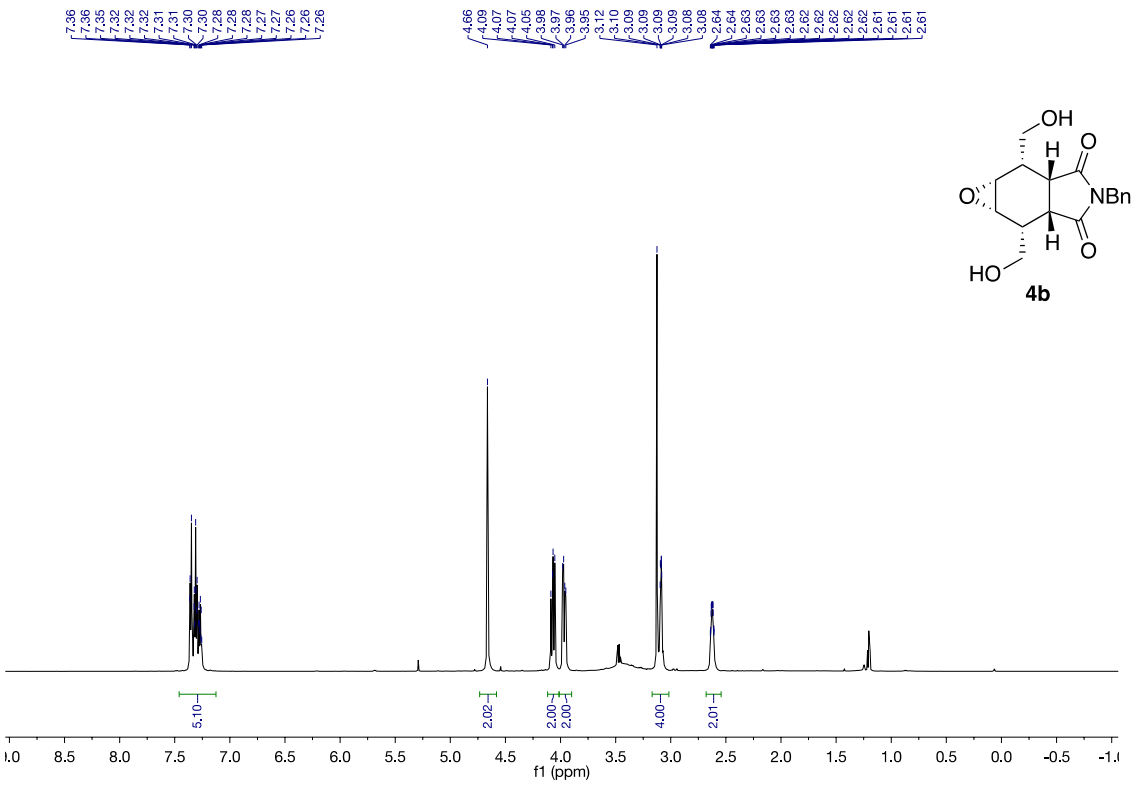
f1 (ppm)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2.61
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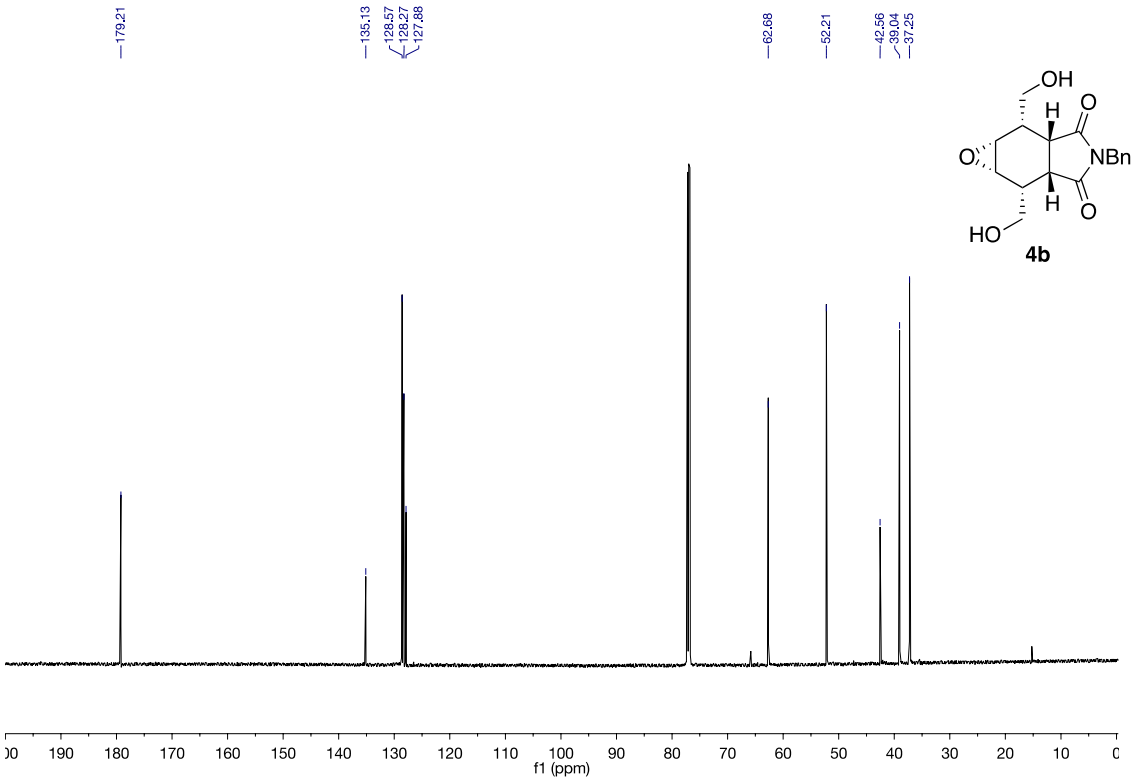
f1 (ppm)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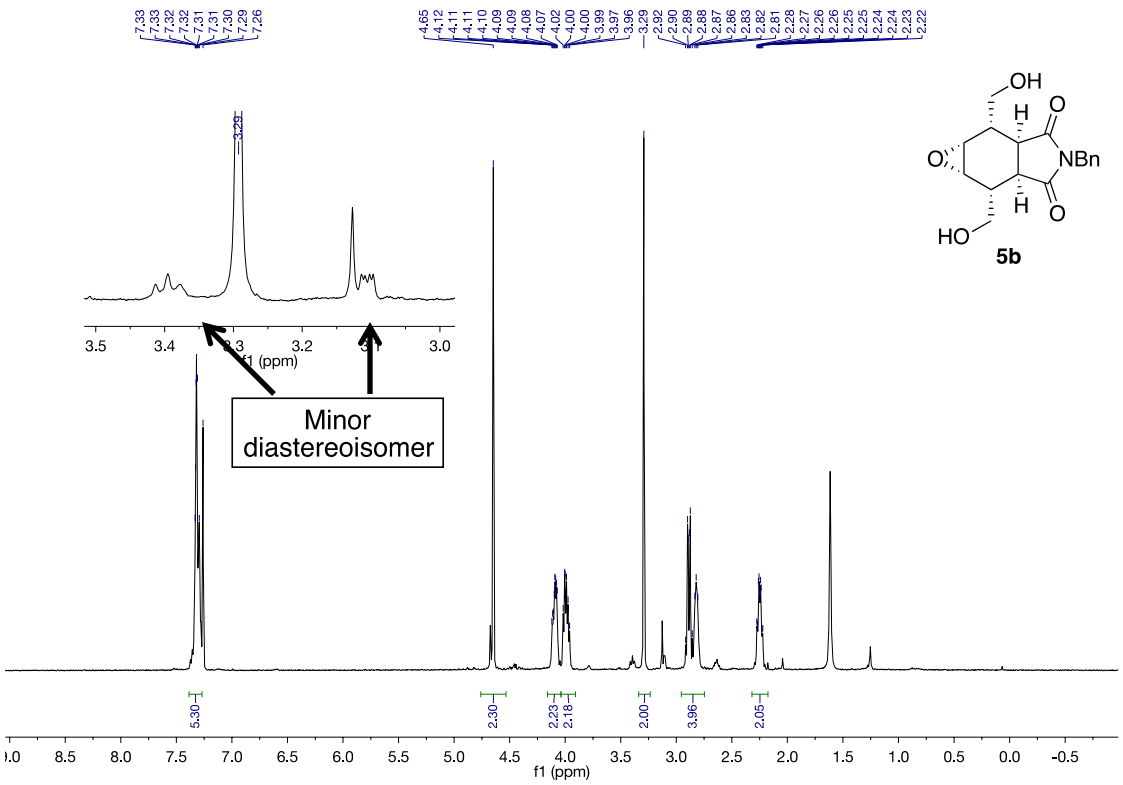
O
H
HO
4b
f1 (ppm)


Minor
diastereoisomer
f1 (ppm)
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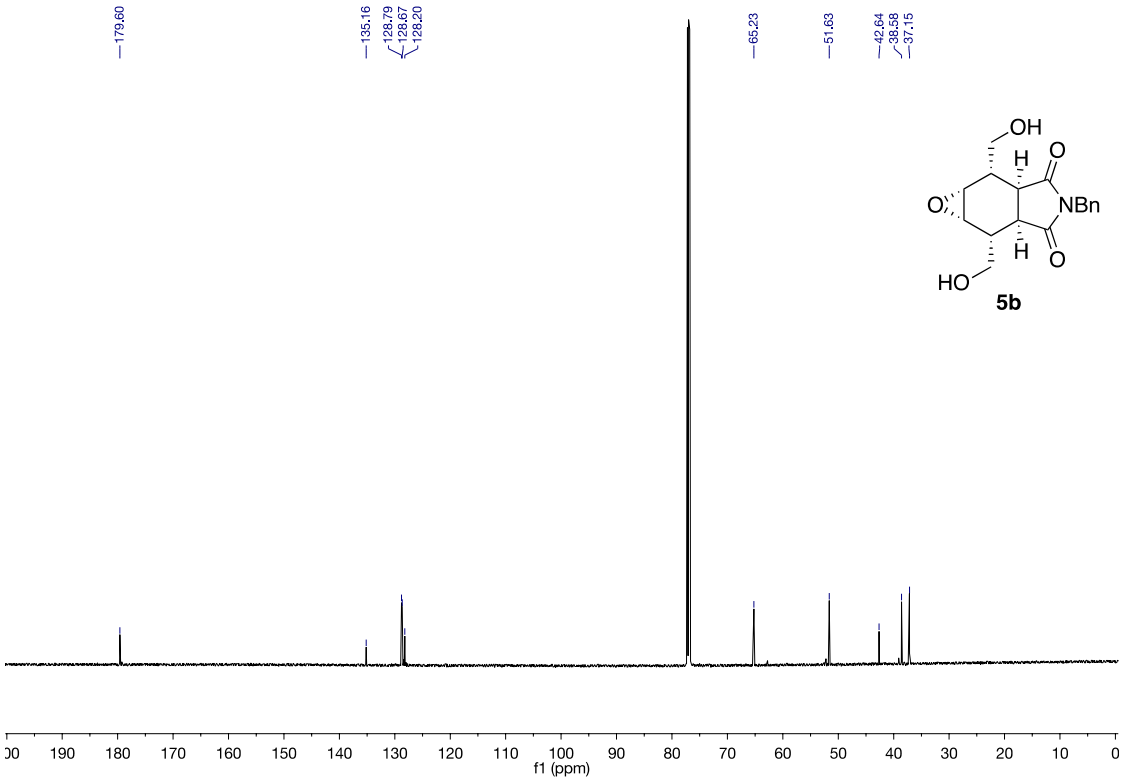
H
O
NBn
O
H
O
HO
5b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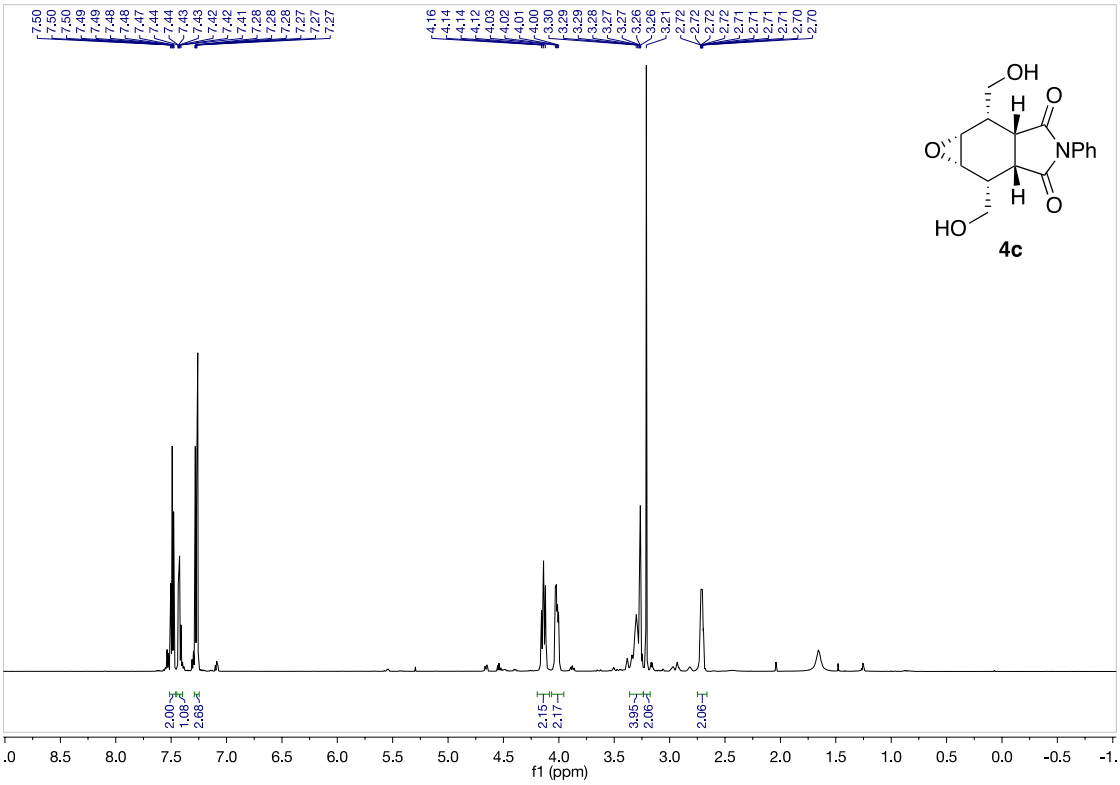
5b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NPh
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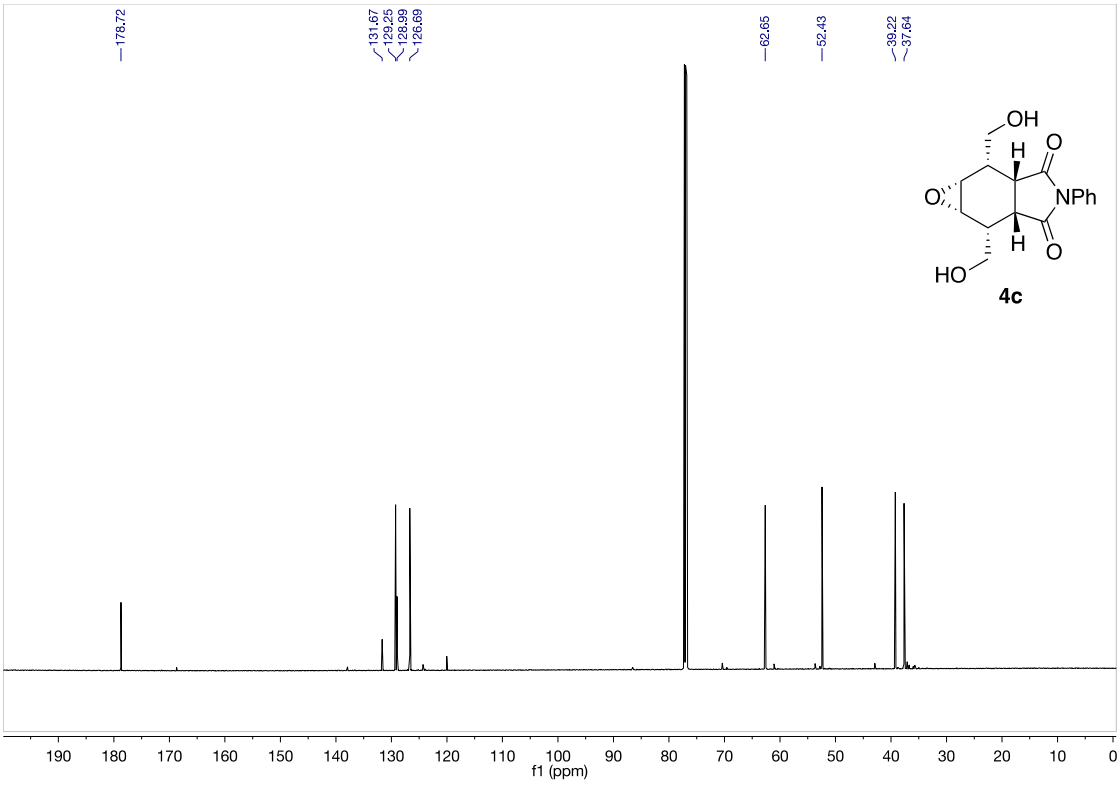
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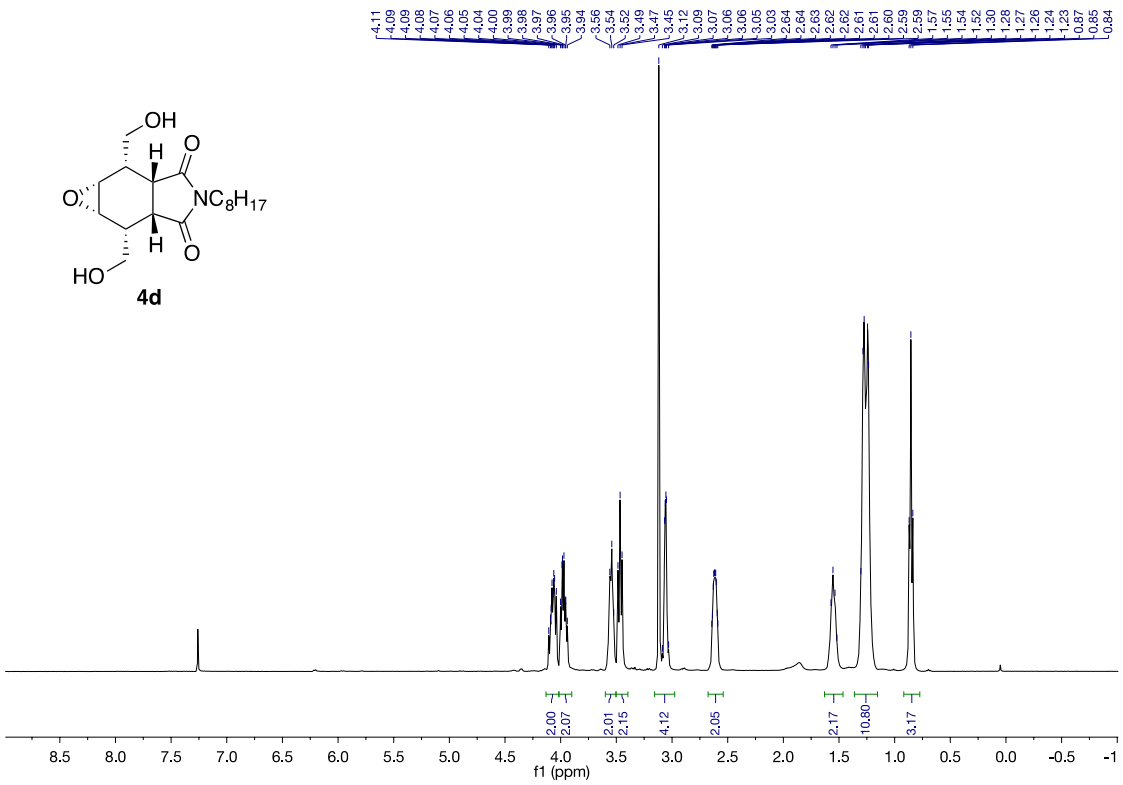
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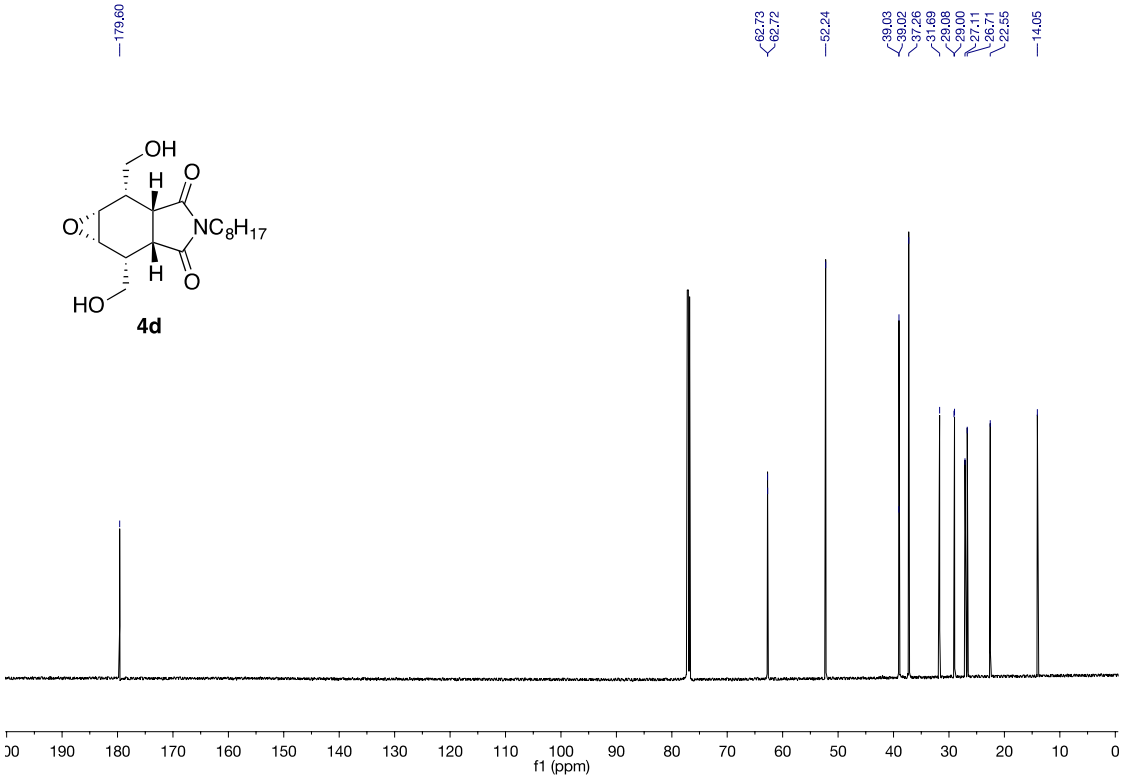
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O
HO
4d
190
180
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100
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0
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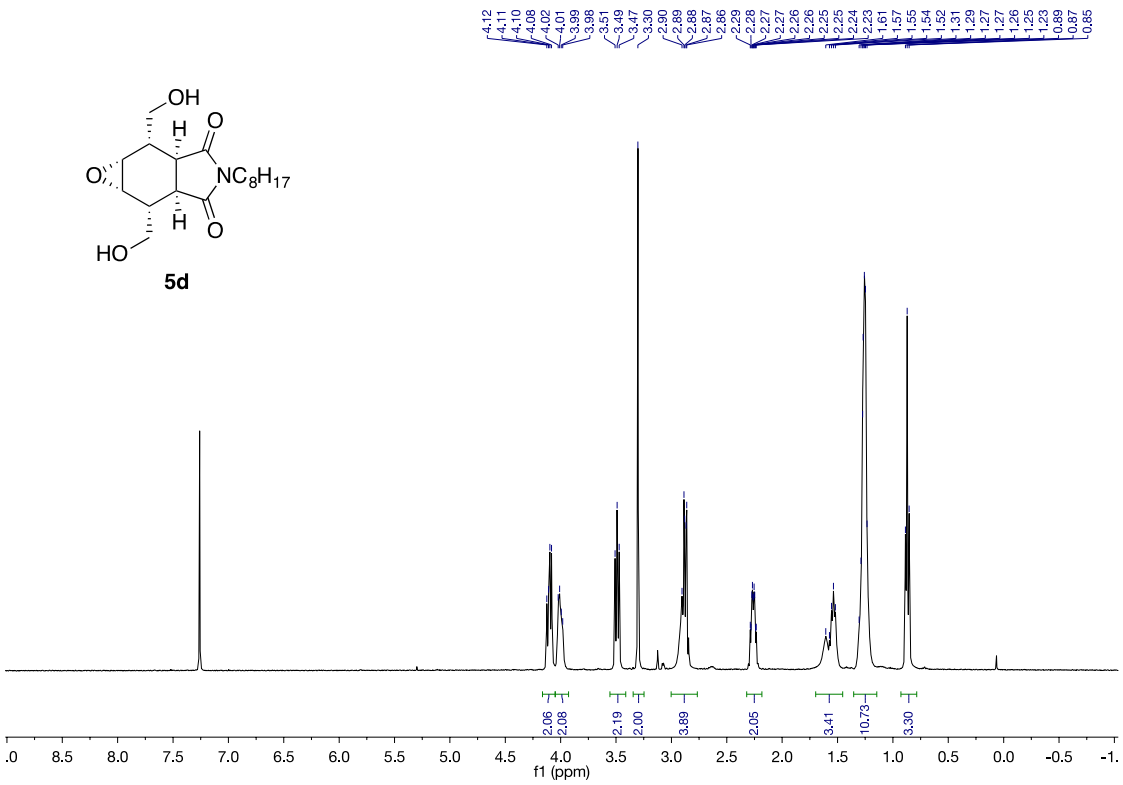
OH
H
O
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H
O
HO
5d
4.12
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3.51
3.49
3.47
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1.57
1.55
1.54
1.52
1.31
1.29
1.27
1.27
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1.23
0.89
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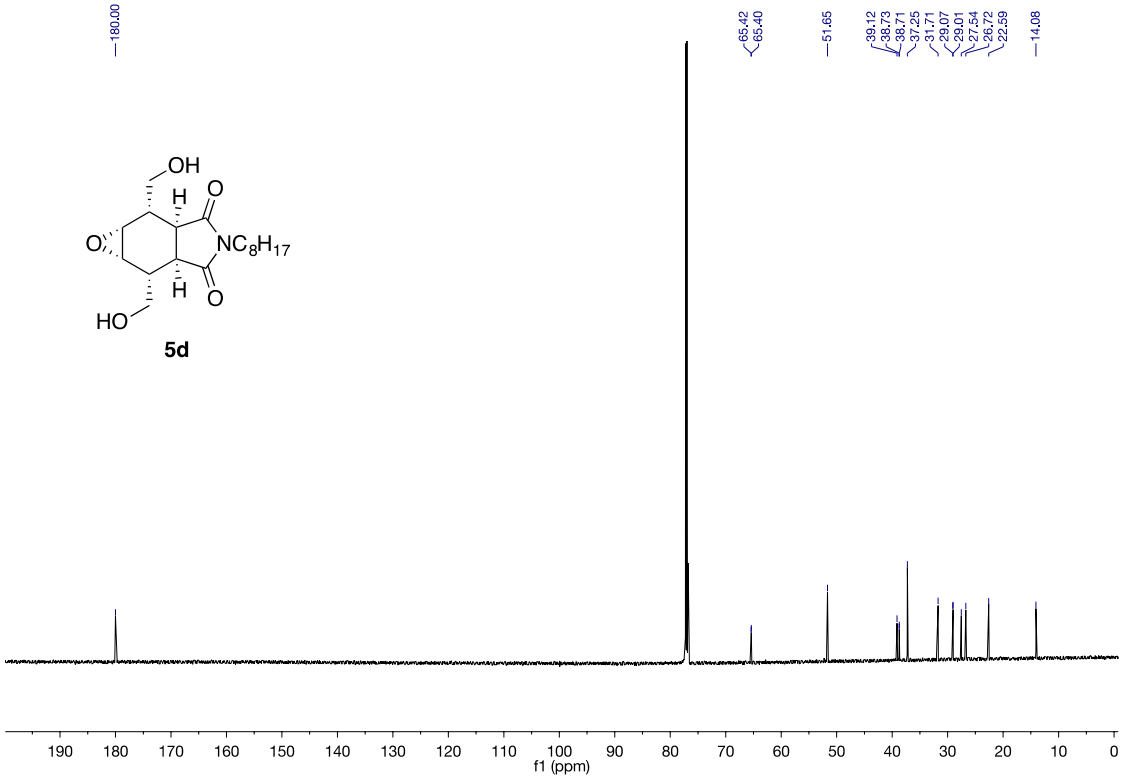
f1 (ppm)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38.73
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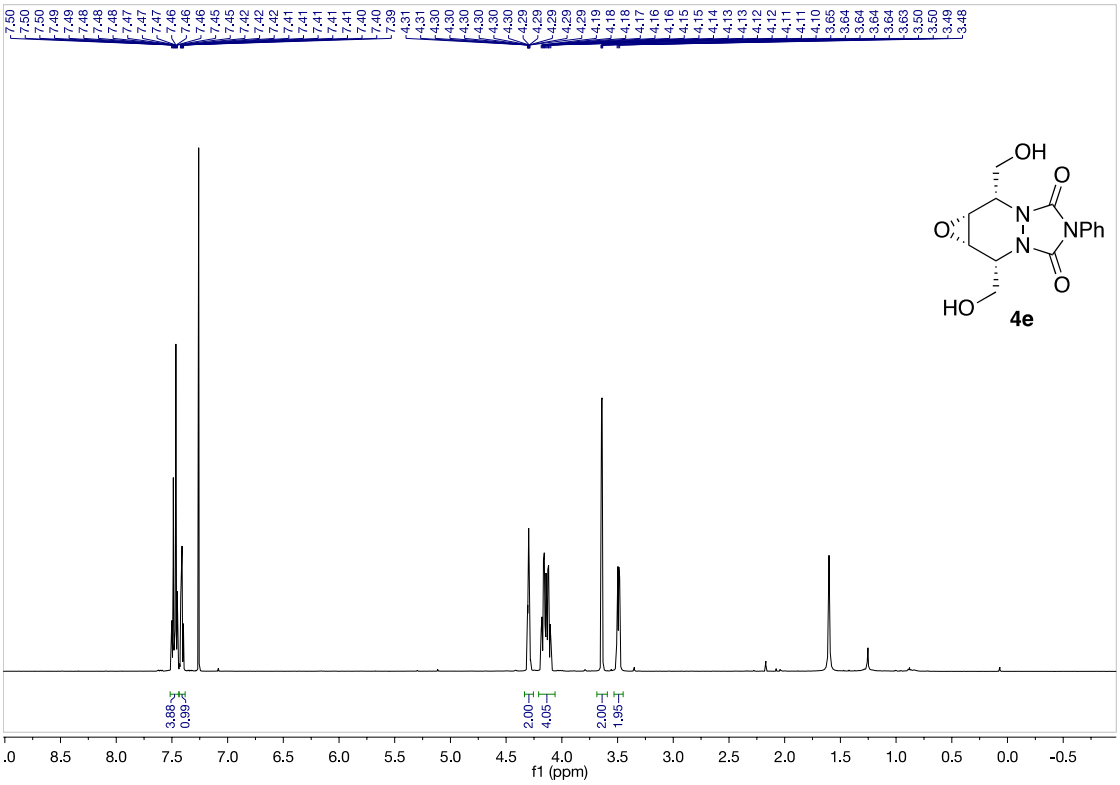
NC8H17
H
O
HO
5d
f1 (ppm)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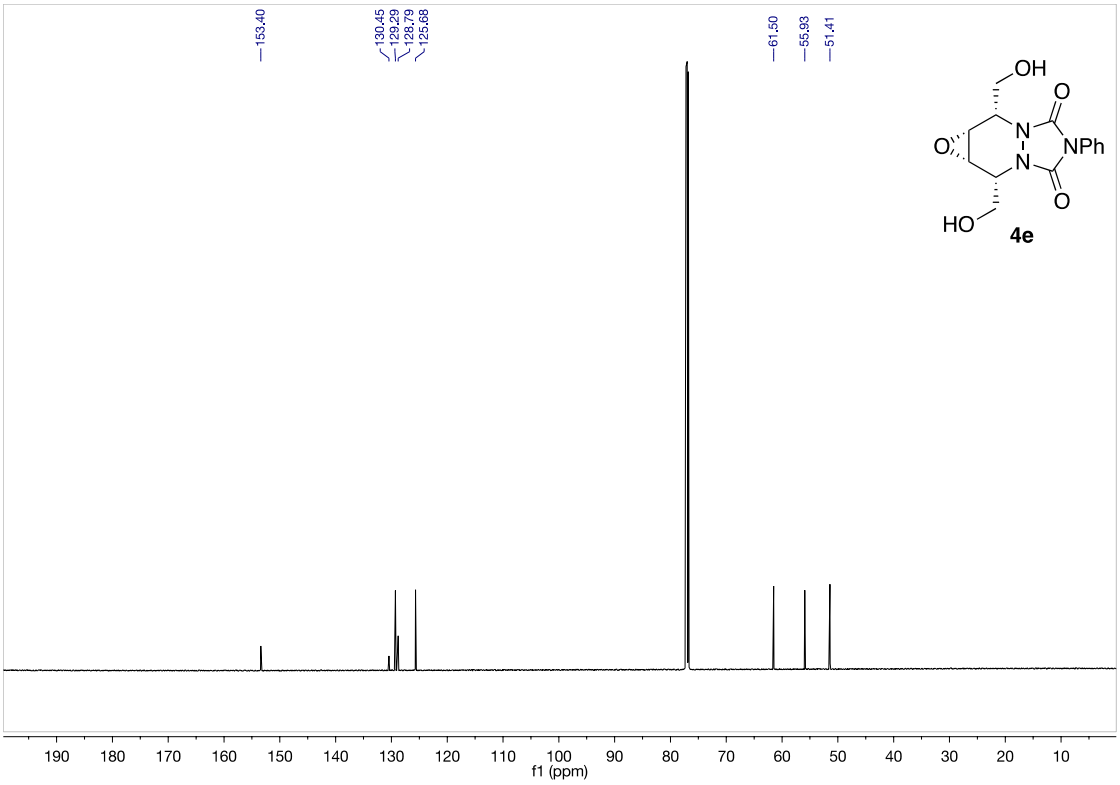
f1 (ppm)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55.93
51.41
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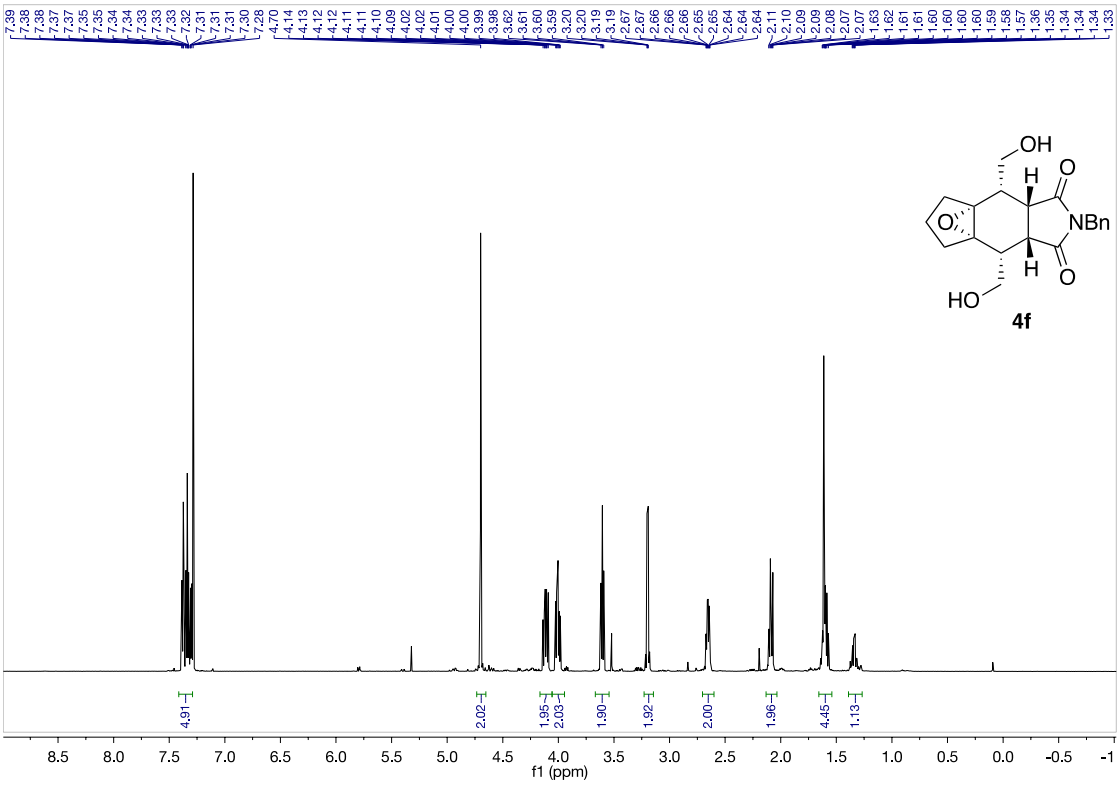
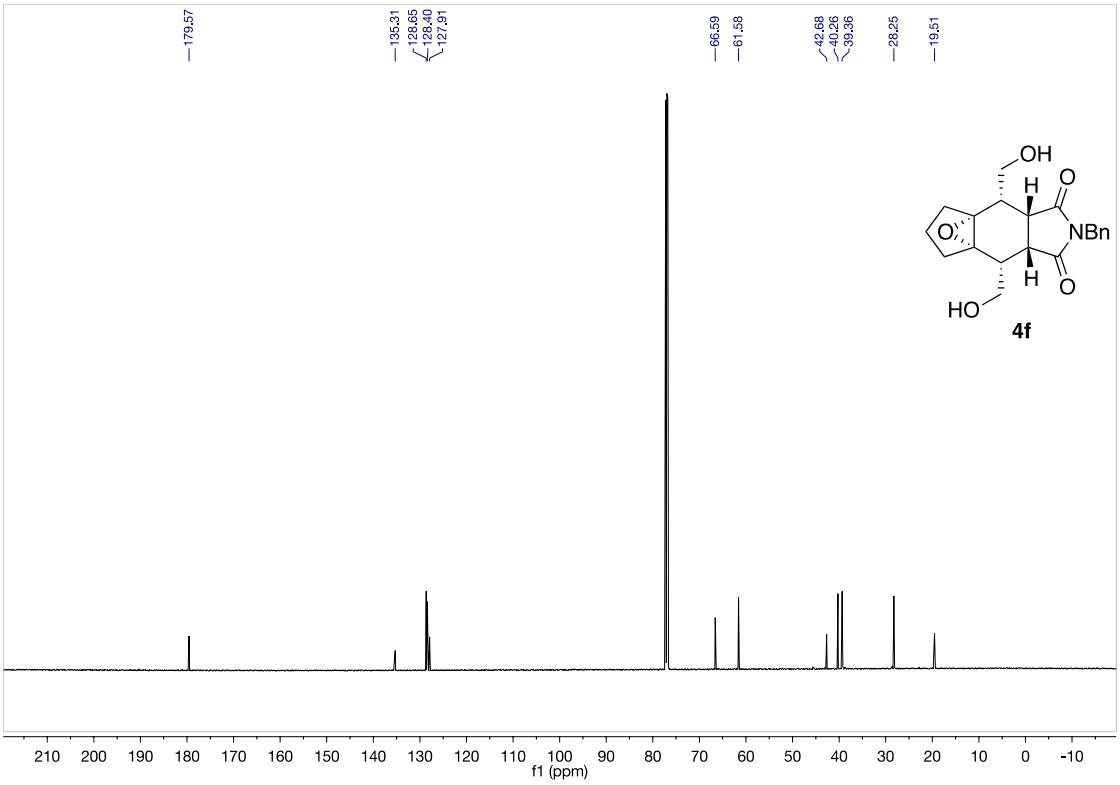
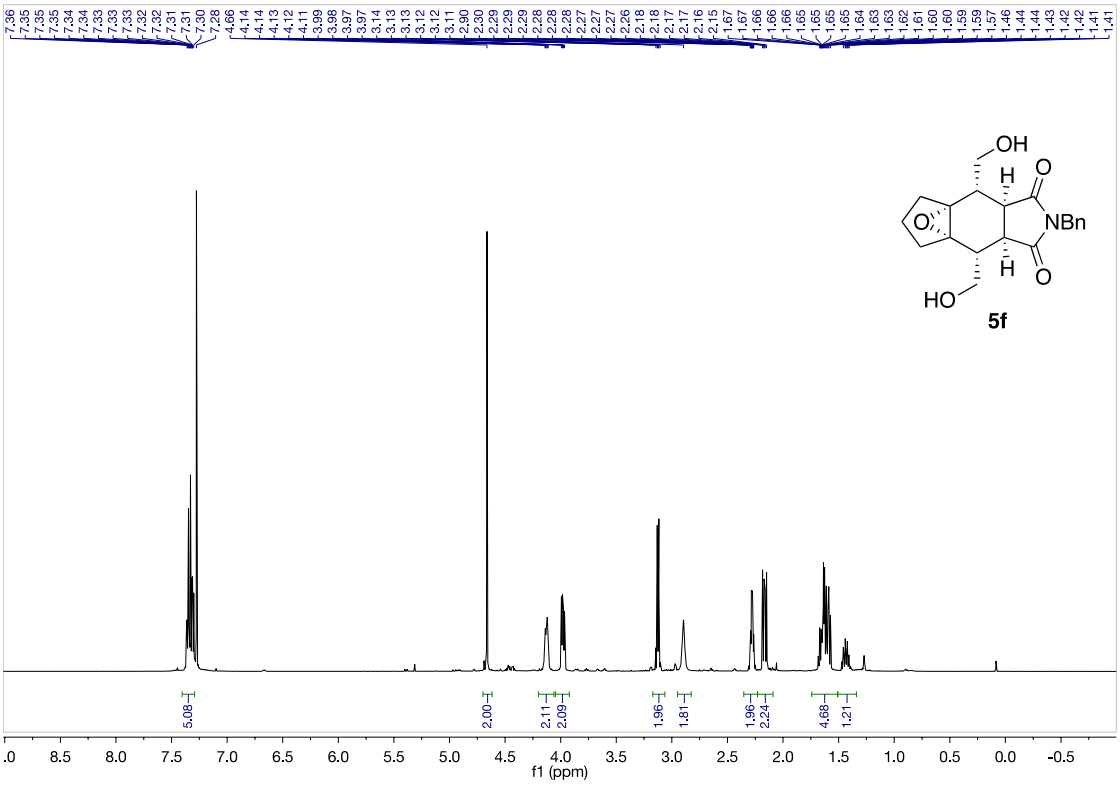
N
NPh
O
O
HO
4e
f1 (ppm)


OH
H
O
NBn
O
H
O
HO
4f
4.91
2.02
1.95
2.03
1.90
1.92
2.00
1.96
4.45
1.13
f1 (ppm)


179.57
135.31
128.65
128.40
127.91
66.59
61.58
42.68
40.26
39.36
28.25
19.51
OH
H
O
NBn
O
H
O
HO
4f
f1 (ppm)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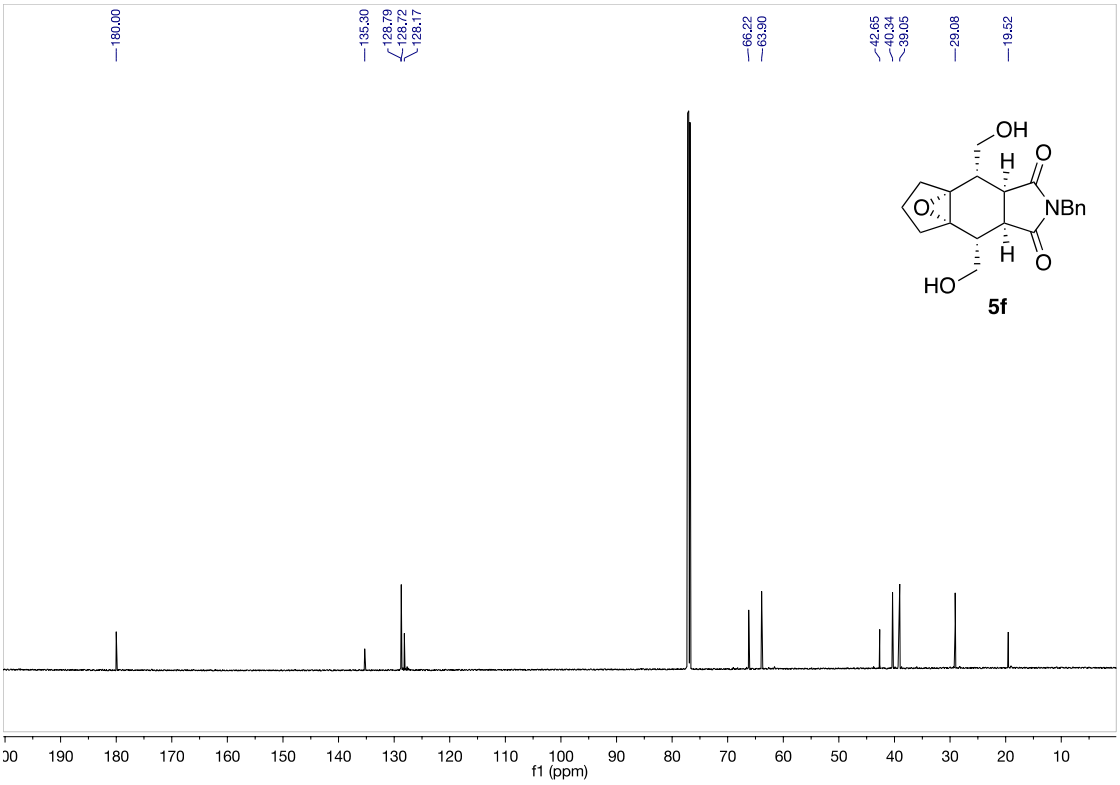
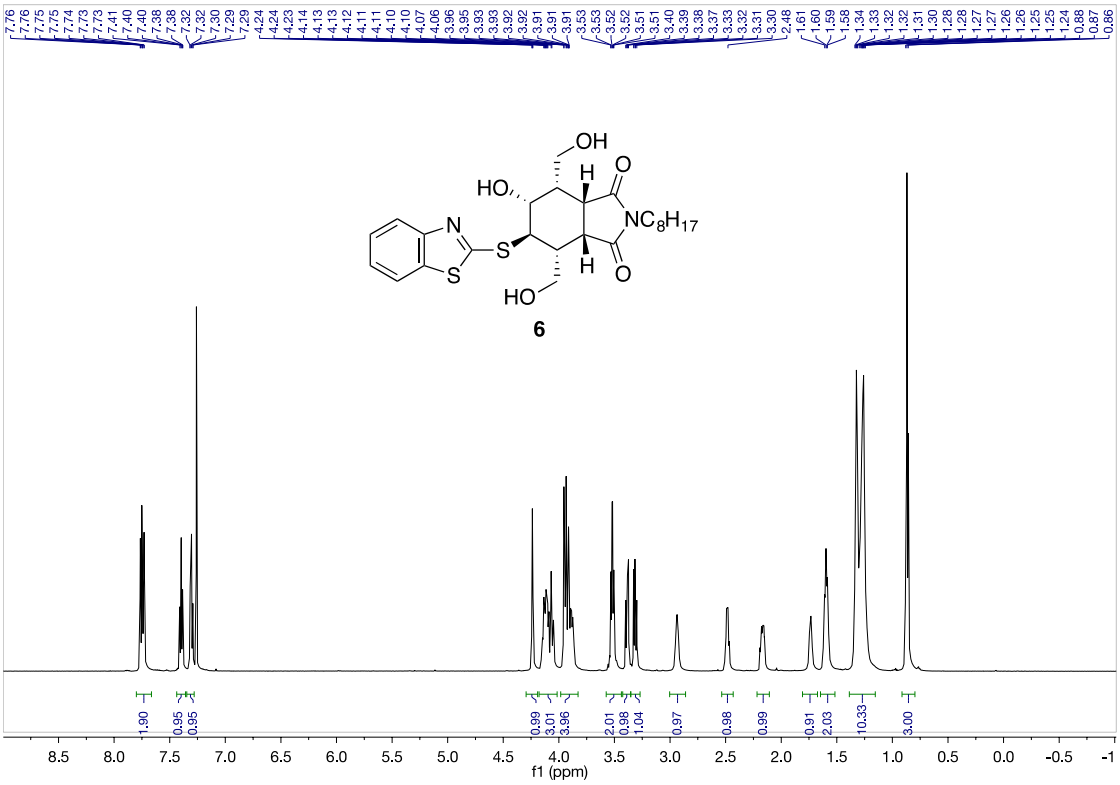
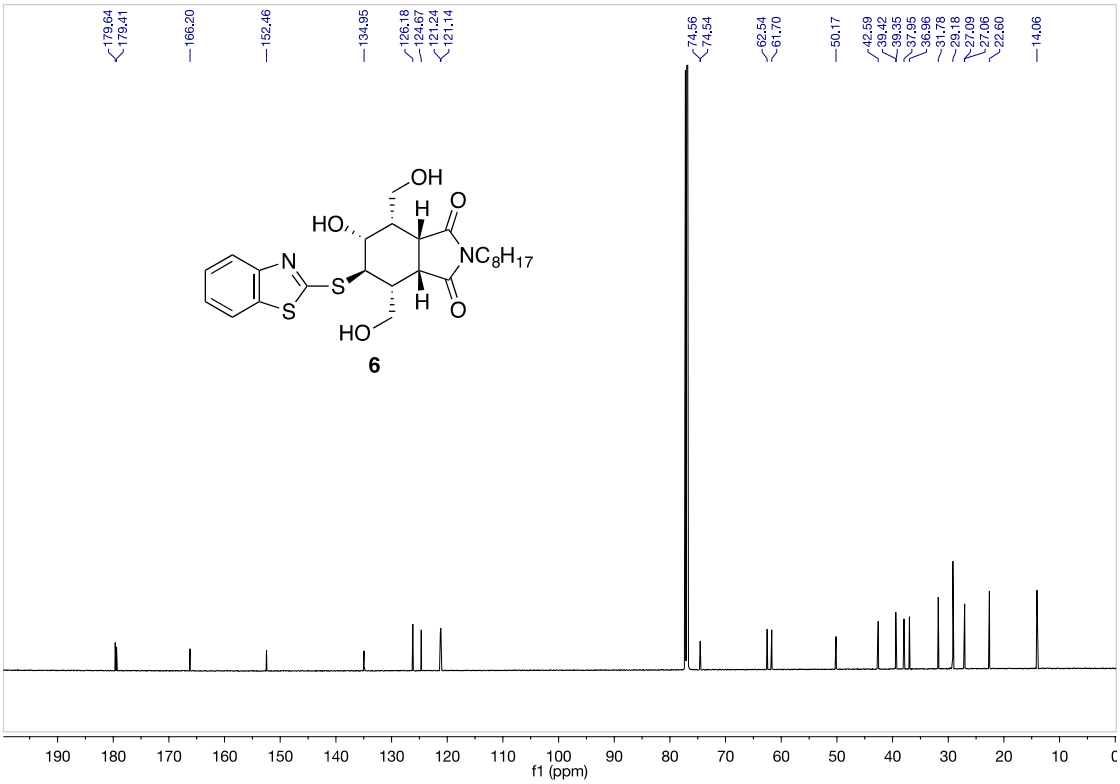
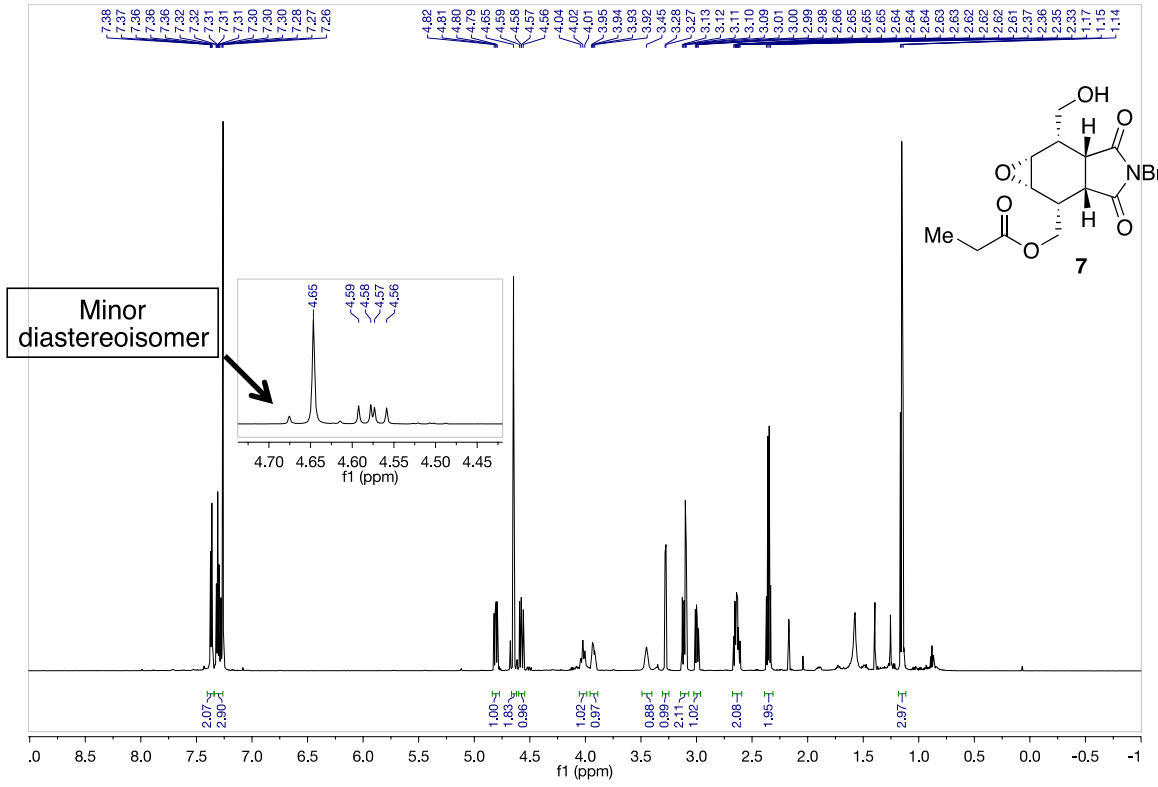
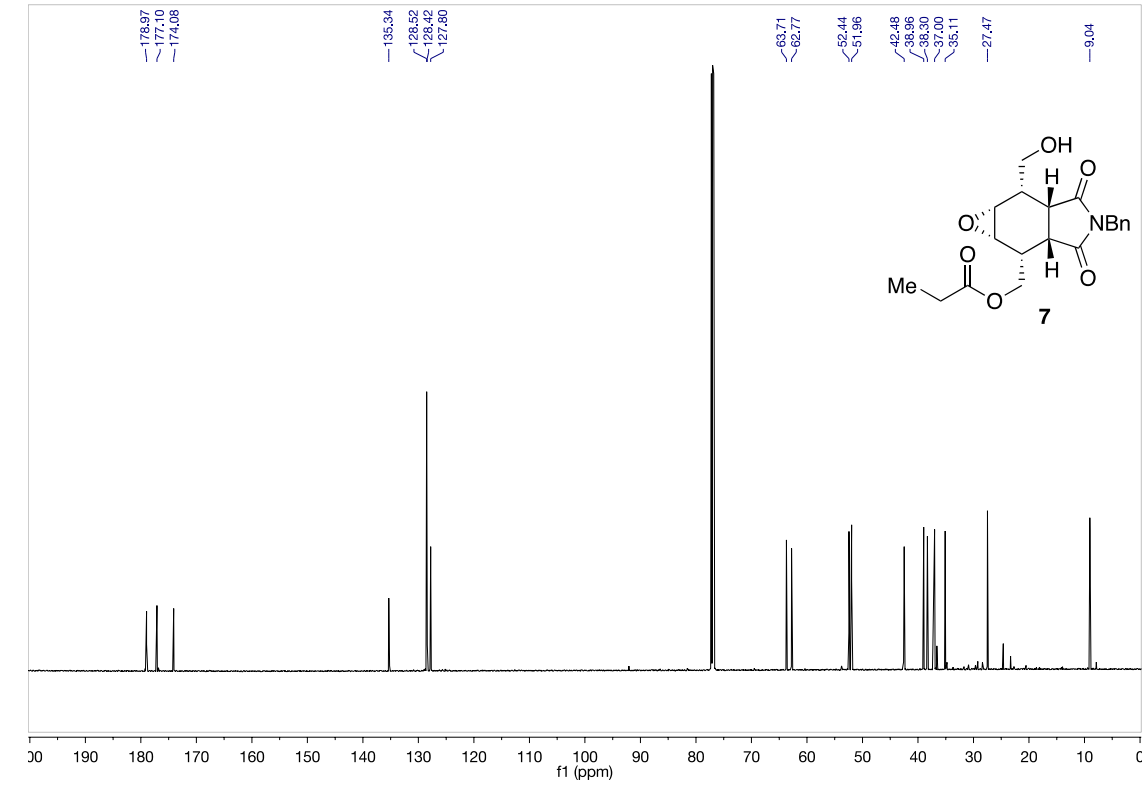
H
O
O
NBn
O
H
HO
5f
f1 (ppm)

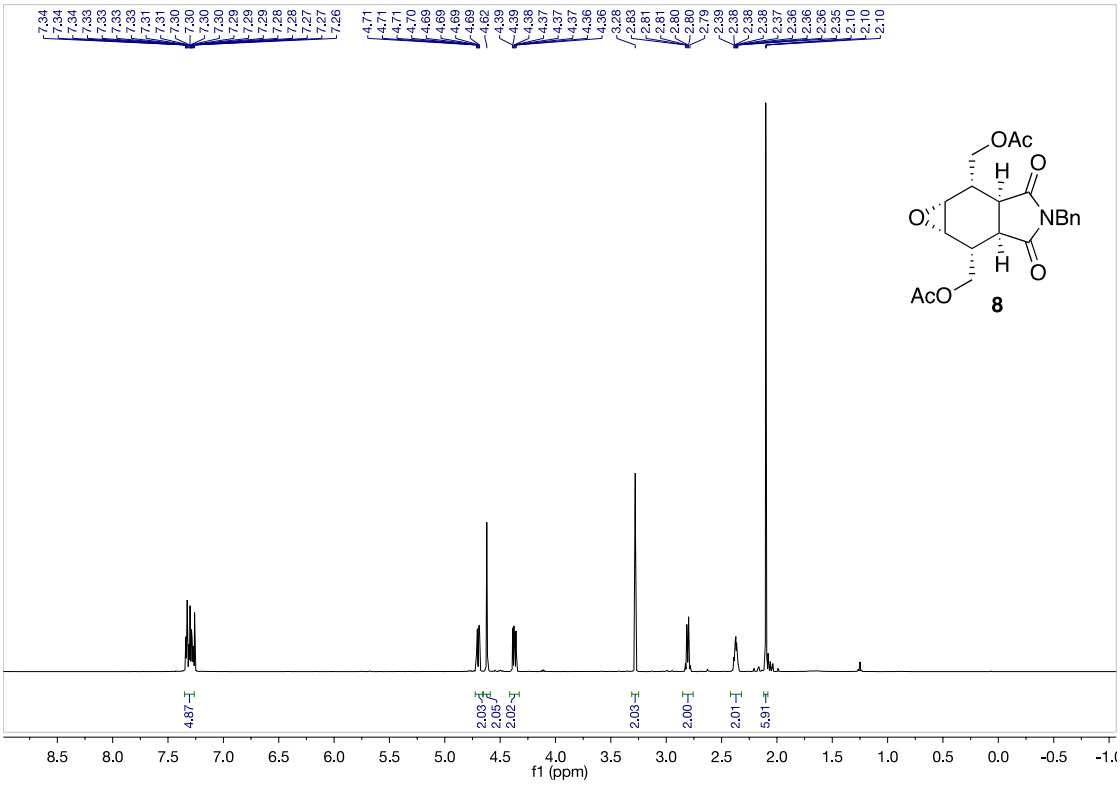

OH
H
O
O
NBn
O
H
HO
5f
f1 (ppm)

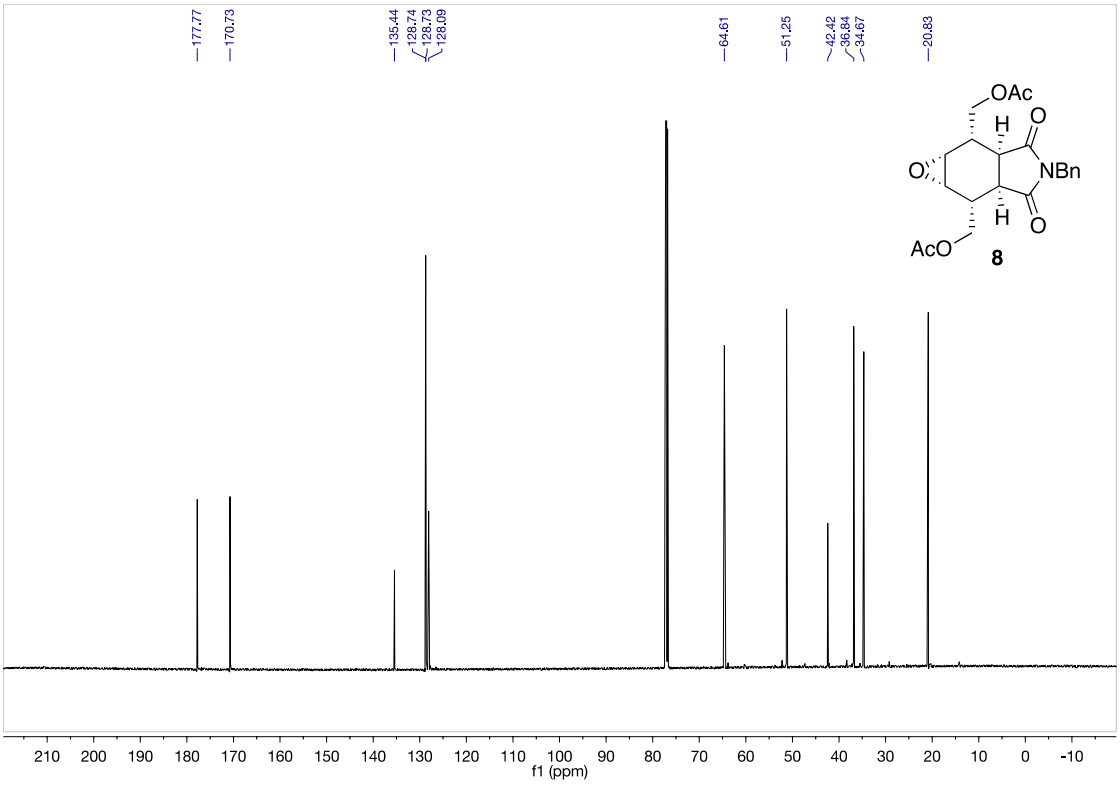

OH
HO
H
O
N
S
NC8H17
S
H
O
HO
6
f1 (ppm)

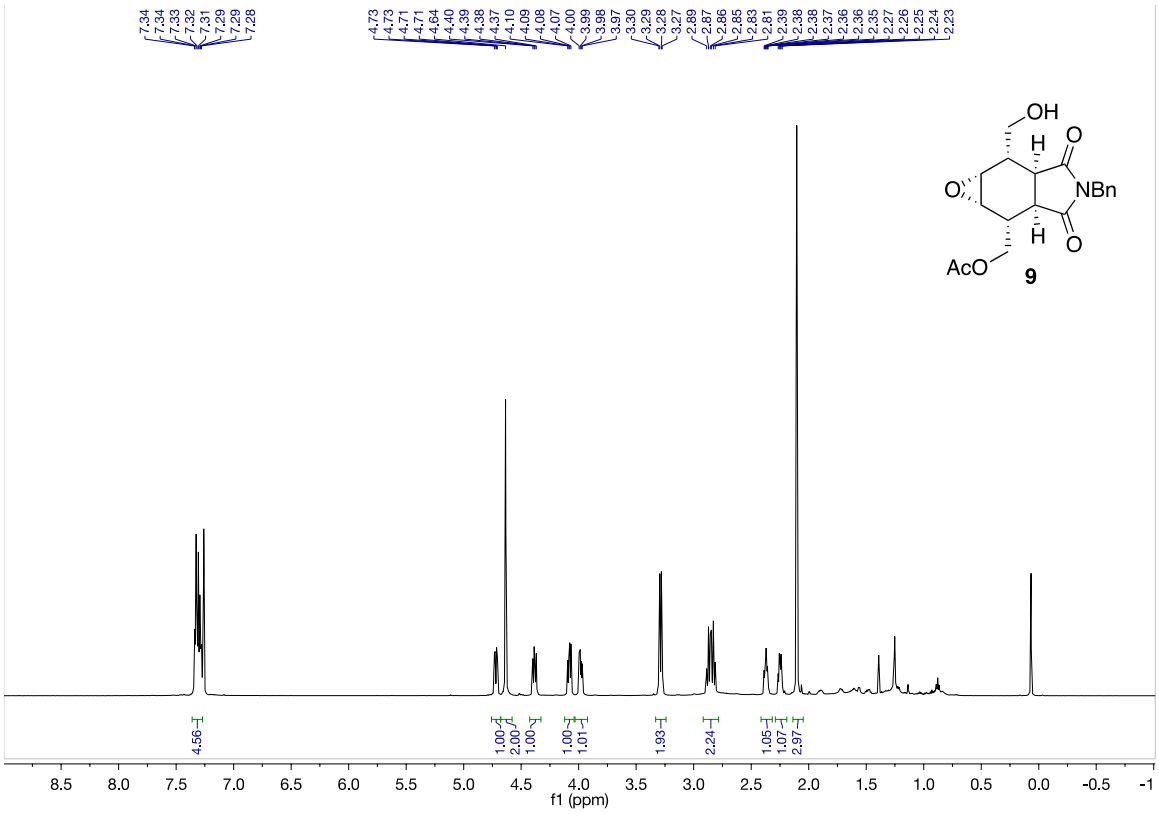

179.64
179.41
166.20
152.46
134.95
126.18
124.67
121.24
121.14
74.56
74.54
62.54
61.70
50.17
42.59
39.42
39.35
37.95
36.96
31.78
29.18
27.09
27.06
22.60
14.06
OH
HO
H
O
N
S
NC8H17
S
H
O
HO
6
f1 (ppm)

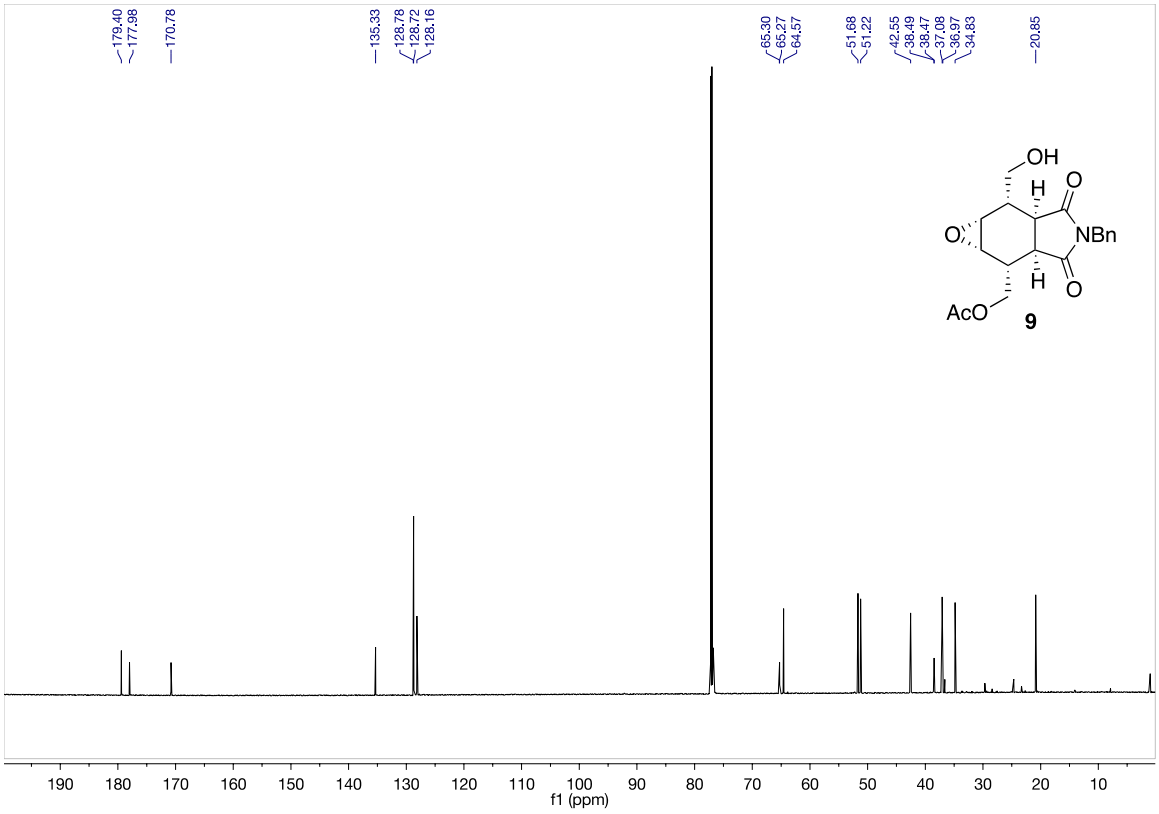

Minor diastereoisomer
OH
H
O
O
NBn
O
H
O
Me
O
O
7
4.70 4.65 4.60 4.55 4.50 4.45
f1 (ppm)
f1 (ppm)


OH
H
O
O
NBn
O
H
O
Me
O
O
7
f1 (ppm)


OAc
H
O
O
NBn
H
O
AcO
8
f1 (ppm)

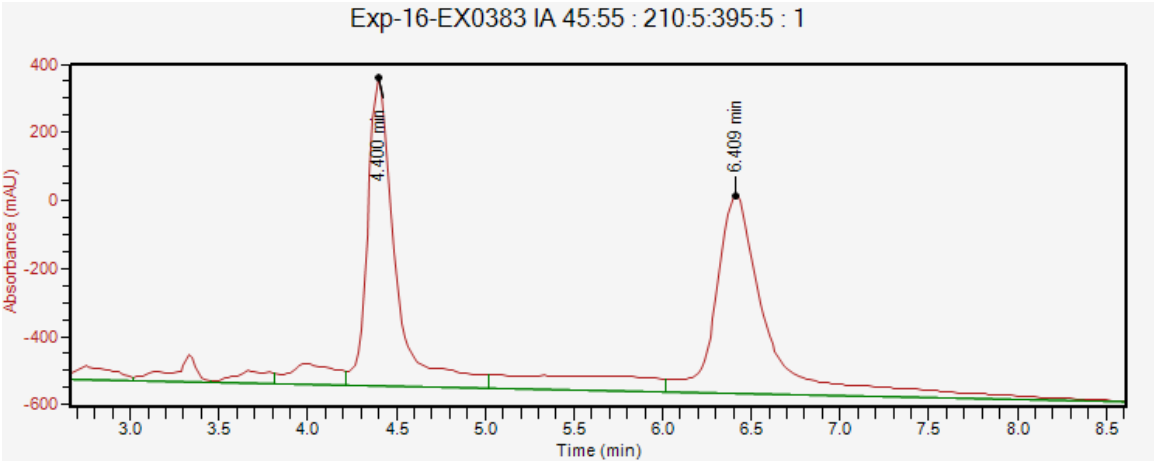

OAc
H
O
O
NBn
H
O
AcO
8
f1 (ppm)


OH
H
O
O
NBn
H
O
AcO
9
f1 (ppm)

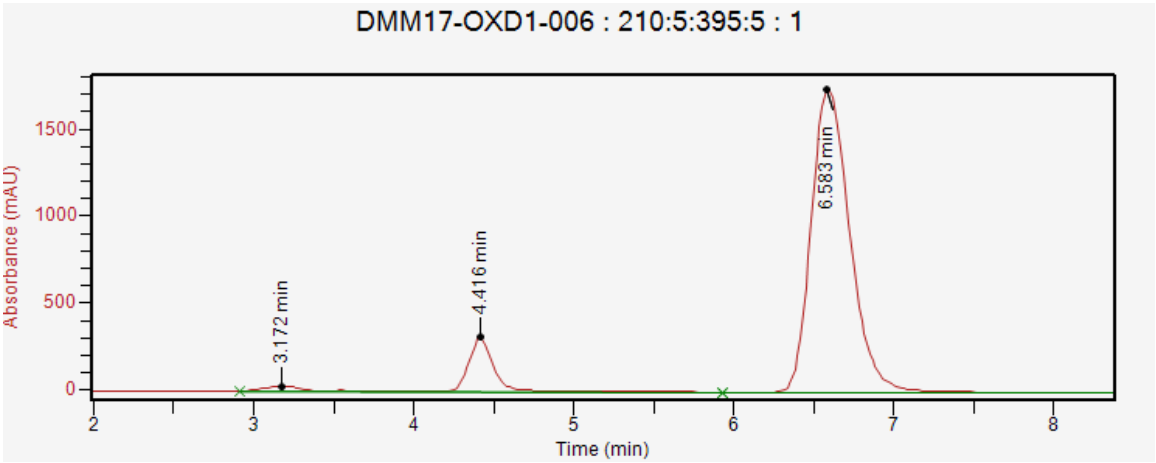

OH
H
O
O
NBn
H
O
AcO
9
f1 (ppm)

HPLC Traces of Racemic and Optically Active Desymmetrization Products

Compound 6

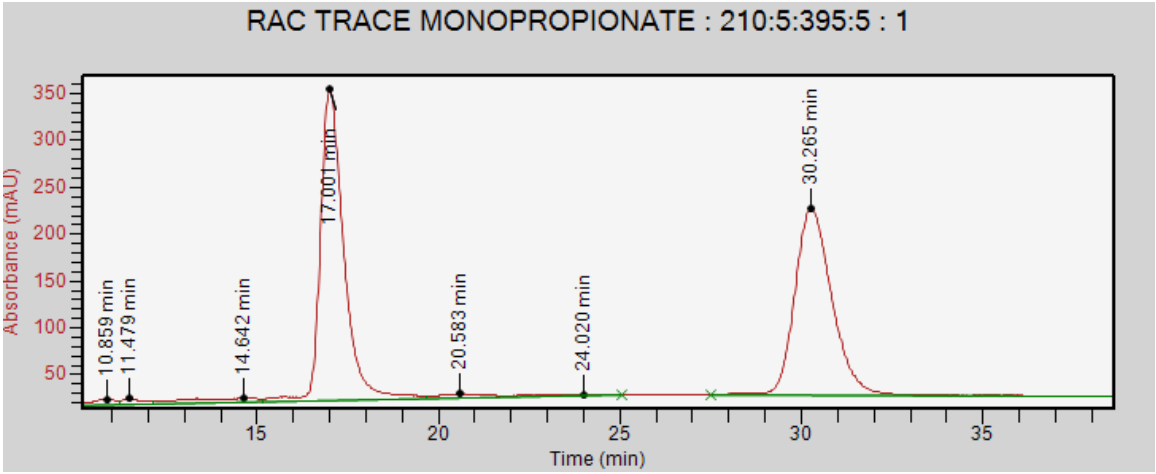


Channel	Ret. Time	Area	Height
210:5:395:5	4.400 min	9900522.28	905661.77
210:5:395:5	6.409 min	11956289.52	584583.69

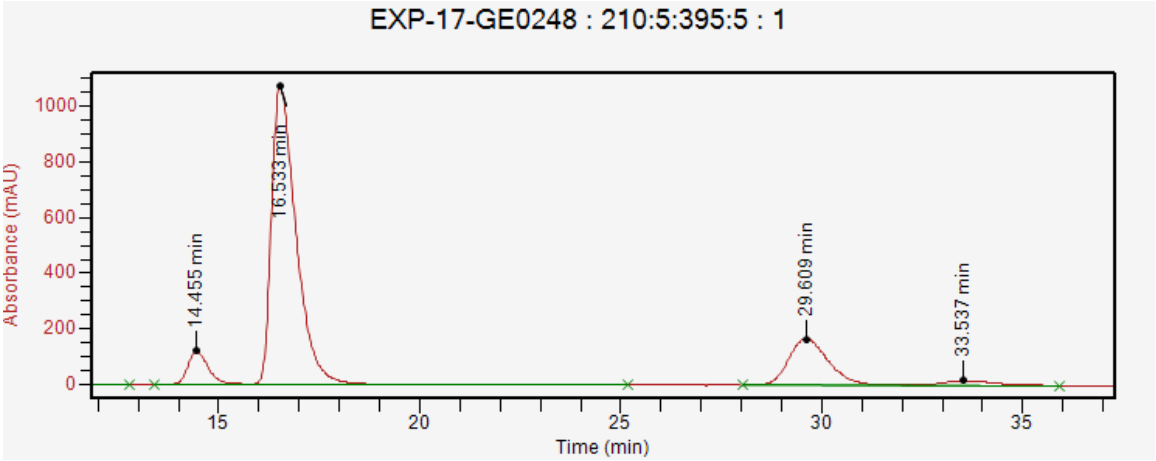


Channel	Ret. Time	Area	Height
210:5:395:5	4.416 min	3342624.28	313171.62
210:5:395:5	6.583 min	29164163.62	1738356.90

Compound 7

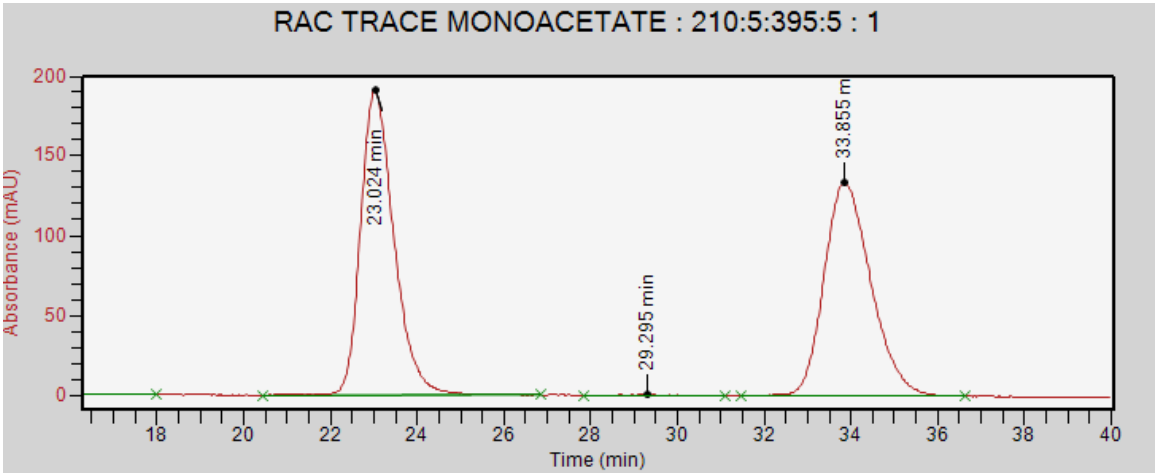


Channel	Ret. Time	Area	Height
210:5:395:5	17.001 min	14950419.41	333050.22
210:5:395:5	30.265 min	14339241.57	198885.42

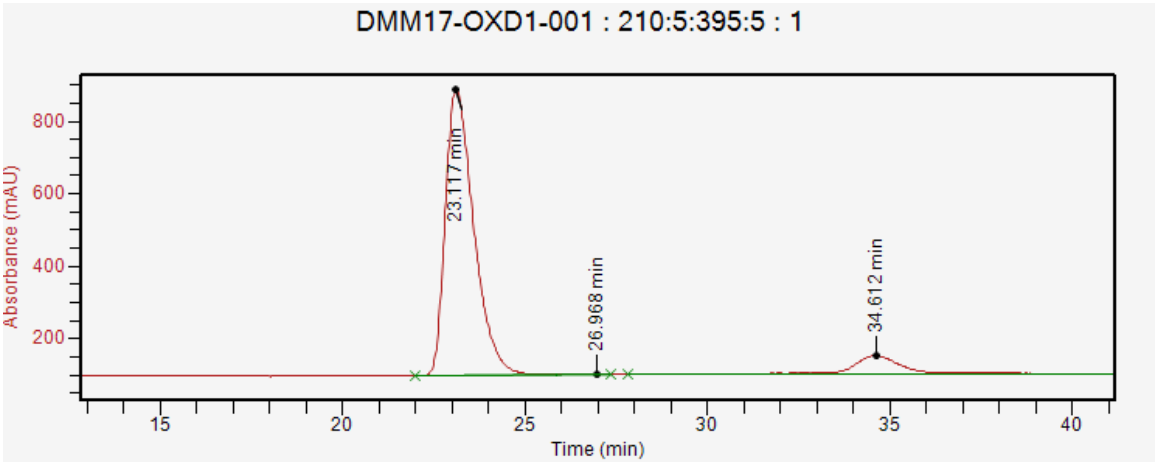


Channel	Ret. Time	Area	Height
210:5:395:5	16.533 min	45878443.13	1074573.49
210:5:395:5	29.609 min	11510821.50	167790.40

Compound 9

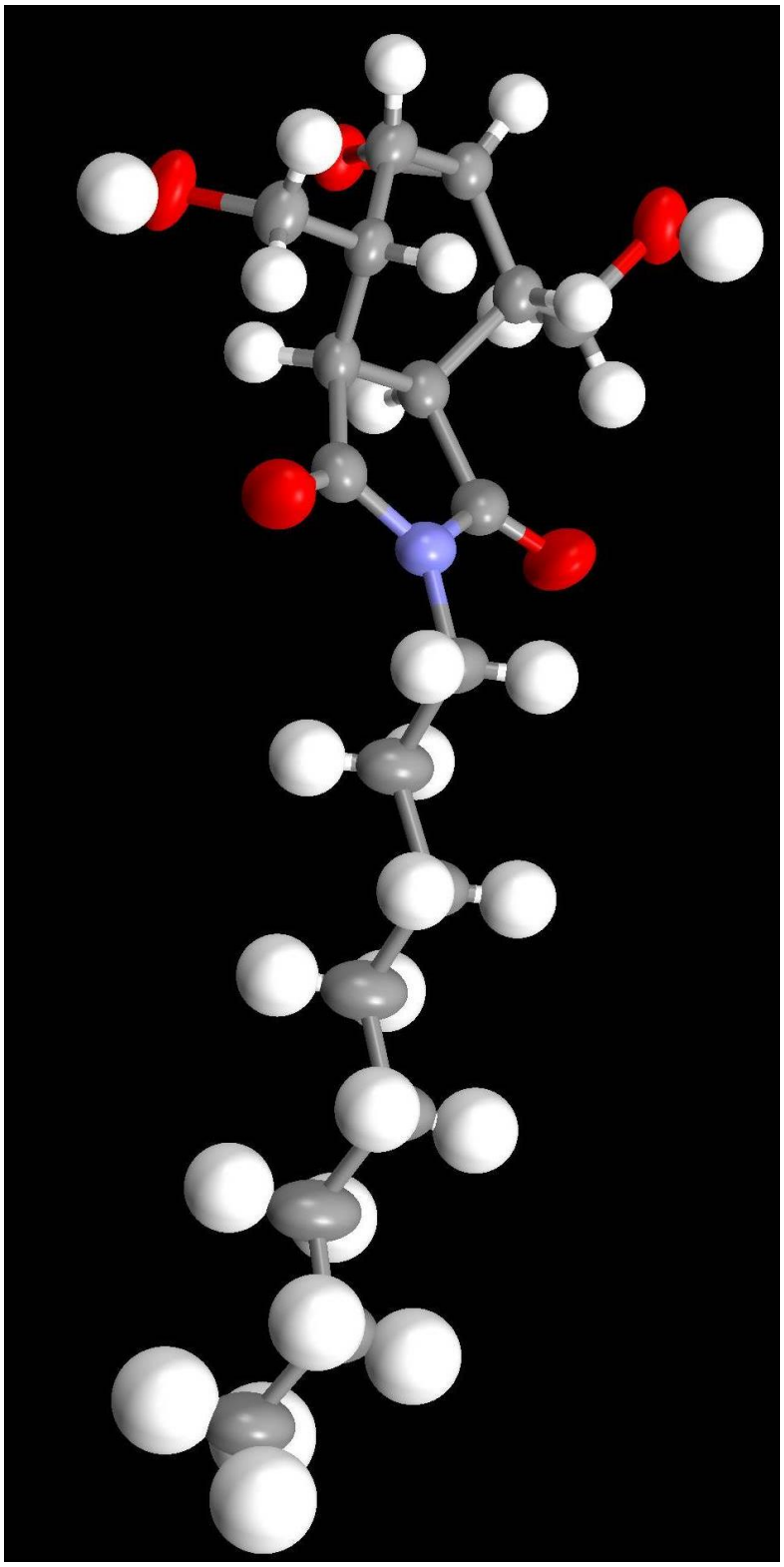


Channel	Ret. Time	Area	Height
210:5:395:5	23.024 min	10248305.99	191246.18
210:5:395:5	33.855 min	10075514.22	133769.43



Channel	Ret. Time	Area	Height
210:5:395:5	23.117 min	43053094.08	792074.24
210:5:395:5	34.612 min	6096713.01	52065.35

ORTEP diagram for succinimide **5d.** Ellipsoid contours at the 50% probability level.



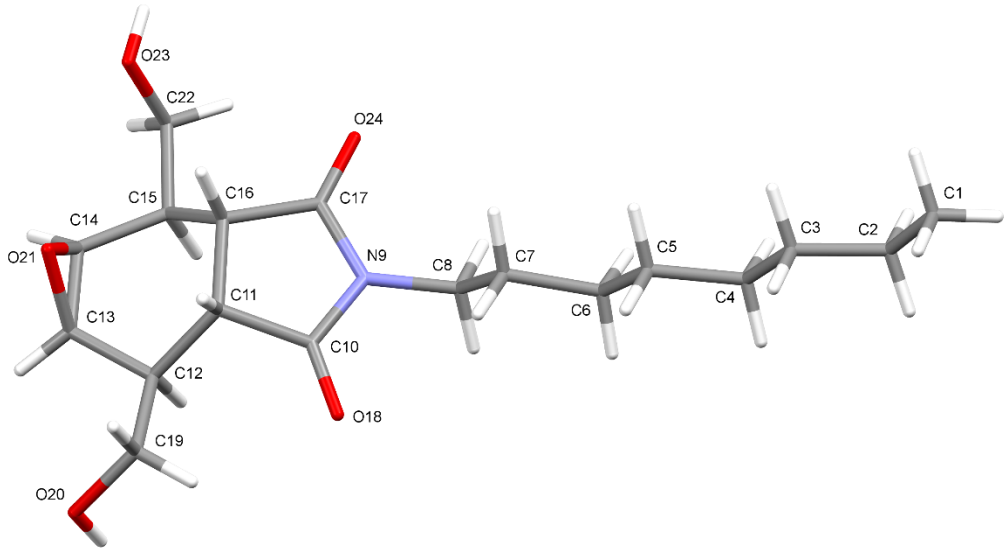


Table S2. Crystal data and structure refinement for x1602003.

Identification code	x1602003
Empirical formula	$C_{18}H_{29}NO_5$
Formula weight	339.42
Temperature/K	100
Crystal system	orthorhombic
Space group	Pbca
a/Å	5.79580(6)
b/Å	14.35143(15)
c/Å	43.7743(5)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	3641.06(7)
Z	8
ρ_{calc}g/cm^3	1.238
μ/mm^{-1}	0.732
F(000)	1472.0
Crystal size/mm^3	0.262 × 0.189 × 0.036
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	4.036 to 133.198

Index ranges	$-6 \leq h \leq 6, -15 \leq k \leq 17, -51 \leq l \leq 52$
Reflections collected	40425
Independent reflections	3201 [$R_{int} = 0.0298$, $R_{sigma} = 0.0173$]
Data/restraints/parameters	3201/0/226
Goodness-of-fit on F^2	1.057
Final R indexes [$I>=2\sigma(I)$]	$R_1 = 0.0491$, $wR_2 = 0.1242$
Final R indexes [all data]	$R_1 = 0.0638$, $wR_2 = 0.1336$
Largest diff. peak/hole / e $Å^{-3}$	0.20/-0.16

Table S3. Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for x1602003. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
C1	2417(7)	11906(3)	5845.3(6)	103.0(11)
C2	3441(6)	11979(3)	5533.3(7)	104.4(12)
C3	2446(6)	11324(2)	5305.3(6)	90.2(9)
C4	3359(6)	11442(2)	4980.1(5)	81.9(9)
C5	2164(5)	10833.6(19)	4750.7(5)	75.6(8)
C6	2886(5)	11007.3(19)	4420.3(5)	67.3(7)
C7	1509(5)	10431.7(18)	4193.6(5)	66.1(7)
C8	2100(4)	10661.4(17)	3866.4(5)	60.7(6)
N9	892(3)	10053.5(13)	3650.1(4)	51.6(5)
C10	1588(4)	9154.7(17)	3591.5(5)	52.4(6)
C11	-139(3)	8696.5(14)	3378.1(4)	42.9(5)
C12	1071(3)	8193.1(13)	3113.1(4)	39.3(4)
C13	-539(3)	8079.8(13)	2846.6(4)	40.1(4)
C14	-2003(3)	8864.4(13)	2765.3(4)	41.5(5)
C15	-1796(3)	9742.4(14)	2951.4(4)	40.9(5)
C16	-1813(3)	9495.6(13)	3293.6(4)	41.7(5)
C17	-1094(4)	10301.4(16)	3495.0(5)	50.4(5)
O18	3315(3)	8812.3(14)	3698.5(4)	75.5(5)
C19	2127(4)	7272.7(14)	3215.7(5)	49.6(5)
O20	3221(3)	6797.6(11)	2967.7(4)	56.4(4)
O21	-2994(2)	8052(1)	2913.3(3)	49.8(4)
C22	-3538(3)	10473.0(14)	2859.2(5)	48.0(5)
O23	-5805(3)	10141.3(11)	2919.6(4)	60.4(5)
O24	-2032(3)	11049.7(12)	3522.3(4)	72.0(5)

Table S4. Anisotropic Displacement Parameters ($Å^2 \times 10^3$) for x1602003. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	117(3)	132(3)	60.1(16)	-11.0(17)	-8.0(18)	-15(2)
C2	122(3)	126(3)	64.5(17)	-10.1(17)	-3.2(18)	-33(2)
C3	118(3)	99(2)	53.3(14)	-2.6(14)	-7.3(16)	-25(2)
C4	109(2)	83.1(19)	53.8(14)	-4.1(13)	-7.8(14)	-18.5(17)
C5	100(2)	77.7(18)	48.8(13)	-1.0(12)	-5.5(14)	-14.6(16)
C6	78.8(18)	72.6(16)	50.4(13)	-6.7(11)	-4.3(12)	-10.3(14)
C7	79.5(18)	71.8(16)	46.9(12)	-6.8(11)	-3.1(11)	-15.4(14)
C8	60.2(15)	72.6(16)	49.2(12)	-9.1(11)	-3.8(11)	-14.1(13)
N9	49.4(11)	62.6(12)	42.8(9)	-6.8(8)	-1.1(8)	-4.6(9)
C10	44.7(13)	71.3(15)	41.2(11)	-0.3(10)	-1.2(9)	3.0(11)
C11	38.1(11)	49.4(12)	41.4(10)	4.0(9)	3.5(8)	-1.8(9)
C12	33.5(10)	39(1)	45.4(10)	3.1(8)	1.9(8)	-2.5(8)
C13	34.7(10)	40.8(10)	44.9(10)	0.9(8)	2.0(8)	-2.8(9)
C14	34.9(10)	44.5(11)	45(1)	1.8(8)	-1.6(8)	-5.0(9)
C15	29.5(10)	43.5(11)	49.7(11)	0.5(9)	-0.7(8)	-3.5(8)
C16	31.2(10)	47.0(11)	47.0(11)	-3.4(8)	3.4(8)	-0.9(9)
C17	44.8(12)	57.8(14)	48.6(12)	-4.4(10)	5.5(9)	-0.1(11)
O18	64.0(11)	97.1(13)	65.4(10)	-11.9(9)	-24.1(9)	19.5(10)
C19	46.6(12)	42.4(11)	59.8(12)	6.1(10)	-6.6(10)	-1.6(10)
O20	39.3(9)	40.6(8)	89.2(12)	-7.4(8)	3.9(8)	-1.3(7)
O21	38.7(8)	47.4(8)	63.3(9)	1.2(7)	1.1(7)	-8.9(7)
C22	39.7(12)	41.3(11)	62.9(13)	1.4(9)	-0.9(9)	-1.5(9)
O23	34.9(9)	42.4(9)	103.9(13)	-3.6(8)	-8.5(8)	2.1(7)
O24	73.1(12)	61.3(11)	81.6(12)	-21.9(9)	-7.9(9)	11.9(9)

Table S5. Bond Lengths for x1602003.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
C1	C2	1.493(4)	C11	C16	1.547(3)
C2	C3	1.487(4)	C12	C13	1.503(3)
C3	C4	1.528(4)	C12	C19	1.523(3)
C4	C5	1.500(3)	C13	C14	1.454(3)
C5	C6	1.526(3)	C13	O21	1.453(2)
C6	C7	1.518(3)	C14	C15	1.505(3)
C7	C8	1.509(3)	C14	O21	1.452(2)
C8	N9	1.466(3)	C15	C16	1.539(3)
N9	C10	1.376(3)	C15	C22	1.511(3)
N9	C17	1.383(3)	C16	C17	1.513(3)
C10	C11	1.519(3)	C17	O24	1.210(3)
C10	O18	1.210(3)	C19	O20	1.430(3)
C11	C12	1.536(3)	C22	O23	1.423(2)

Table S6. Bond Angles for x1602003.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C3	C2	C1	114.6(3)	C14	C13	C12	117.94(17)
C2	C3	C4	114.9(3)	O21	C13	C12	117.08(16)
C5	C4	C3	113.6(2)	O21	C13	C14	59.94(12)
C4	C5	C6	114.4(2)	C13	C14	C15	118.00(16)
C7	C6	C5	112.8(2)	O21	C14	C13	60.00(12)
C8	C7	C6	112.5(2)	O21	C14	C15	117.54(16)
N9	C8	C7	111.99(19)	C14	C15	C16	109.48(16)
C10	N9	C8	122.59(19)	C14	C15	C22	112.54(16)
C10	N9	C17	113.18(18)	C22	C15	C16	114.54(16)
C17	N9	C8	124.2(2)	C15	C16	C11	113.52(15)
N9	C10	C11	109.12(18)	C17	C16	C11	104.75(16)
O18	C10	N9	123.5(2)	C17	C16	C15	112.92(16)
O18	C10	C11	127.4(2)	N9	C17	C16	108.59(18)
C10	C11	C12	111.53(16)	O24	C17	N9	123.6(2)
C10	C11	C16	103.85(16)	O24	C17	C16	127.8(2)
C12	C11	C16	117.03(15)	O20	C19	C12	111.57(17)
C13	C12	C11	110.70(16)	C14	O21	C13	60.06(12)
C13	C12	C19	112.60(16)	O23	C22	C15	109.59(16)
C19	C12	C11	111.63(16)				

Table S7. Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for x1602003.

Atom	*x*	*y*	*z*	U(eq)
H1A	755	12027	5834	155
H1B	3145	12365	5980	155
H1C	2679	11278	5926	155
H2A	3228	12624	5458	125
H2B	5121	11863	5548	125
H3A	751	11407	5302	108
H3B	2765	10679	5373	108
H4A	3173	12102	4918	98
H4B	5029	11298	4978	98
H5A	479	10933	4768	91
H5B	2481	10174	4802	91
H6A	2681	11676	4372	81
H6B	4544	10858	4397	81
H7A	1814	9762	4231	79
H7B	-157	10543	4227	79
H8A	1682	11318	3824	73
H8B	3786	10596	3836	73
H11	-1022	8222	3498	52
H12	2368	8602	3044	47
H13	13	7679	2675	48
H14	-2353	8945	2543	50
H15	-238	10009	2906	49
H16	-3413	9304	3352	50
H19A	3276	7393	3378	60
H19B	903	6871	3302	60
H20	4460(60)	7100(20)	2927(6)	96(11)
H22A	-3257	11054	2975	58
H22B	-3374	10614	2639	58
H23	-6670(50)	10630(20)	2917(6)	89(10)

Experimental

Single crystals of $C_{18}H_{29}NO_5$ **[x1602003]** were grown from dichloromethane/hexane. A suitable crystal was selected and diffraction data was obtained on a 'Bruker APEX-II CCD' diffractometer. The crystal was kept at 100 K during data collection. Using Olex2 [1], the structure was solved with the olex2.solve [2] structure solution program using Charge Flipping and refined with the XL [3] refinement package using Least Squares minimisation.

1. Dolomanov, O.V., Bourhis, L.J., Gildea, R.J, Howard, J.A.K. & Puschmann, H. (2009), J. Appl. Cryst. 42, 339-341.
2. Bourhis, L.J., Dolomanov, O.V., Gildea, R.J., Howard, J.A.K., Puschmann, H. (2015). Acta Cryst. A71, 59-75.
3. Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

Crystal structure determination of [x1602003]

Crystal Data for $C_{18}H_{29}NO_5$ (M =339.42 g/mol): orthorhombic, space group Pbca (no. 61), a = 5.79580(6) Å, b = 14.35143(15) Å, c = 43.7743(5) Å, V = 3641.06(7) Å^3, Z = 8, T = 100 K, μ(CuKα) = 0.732 mm^{-1}, *Dcalc* = 1.238 g/cm^3, 40425 reflections measured (4.036° ≤ 2Θ ≤ 133.198°), 3201 unique (R_{int} = 0.0298, R_{sigma} = 0.0173) which were used in all calculations. The final R_1 was 0.0491 (I > 2σ(I)) and wR_2 was 0.1336 (all data).

Refinement model description

Number of restraints - 0, number of constraints - unknown.

Details:

```
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, All C(H,H) groups
 At 1.5 times of:
  All C(H,H,H) groups
2.a Ternary CH refined with riding coordinates:
 C11(H11), C12(H12), C13(H13), C14(H14), C15(H15), C16(H16)
2.b Secondary CH2 refined with riding coordinates:
 C2(H2A,H2B), C3(H3A,H3B), C4(H4A,H4B), C5(H5A,H5B), C6(H6A,H6B), C7(H7A,H7B),
 C8(H8A,H8B), C19(H19A,H19B), C22(H22A,H22B)
2.c Idealised Me refined as rotating group:
 C1(H1A,H1B,H1C)
```

This report has been created with Olex2, compiled on 2015.09.30 svn.r3233 for OlexSys. Please let us know if there are any errors or if you would like to have additional features.