Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Lipase catalyzed desymmetrization of roof shape *cis*-11,12-bis(hydroxymethyl)-9,10-dihydro-9,10-ethanoanthracene

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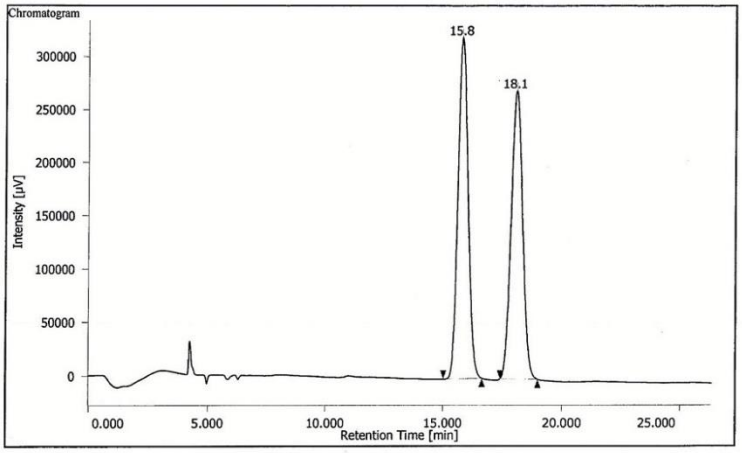
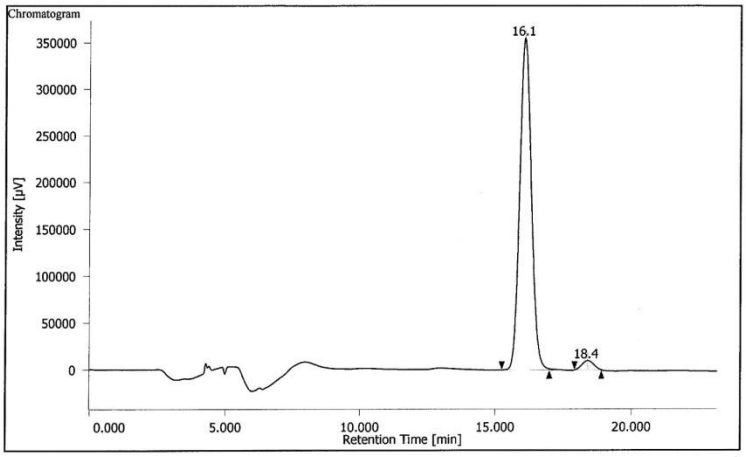
HPLC Report



Peak Information									
#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	NTP	Resolution	Symmetry Factor
1	Unknown	1	15.800	9488023	320759	51.179	6482	2.704	1.123
2	Unknown	1	18.058	9050729	270924	48.821	6588	N/A	1.105

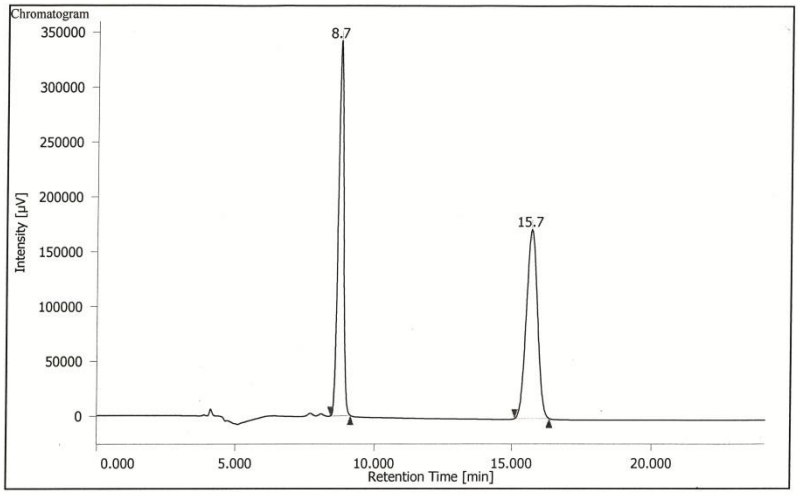
Figure: HPLC graph of Racemic 5



Peak Information									
#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	NTP	Resolution	Symmetry Factor
1	Unknown	1	16.075	10709431	355340	97.325	6528	2.882	1.131
2	Unknown	1	18.400	294359	9934	2.675	8014	N/A	1.011

Figure: HPLC graph of Chiral 5

HPLC REPORT

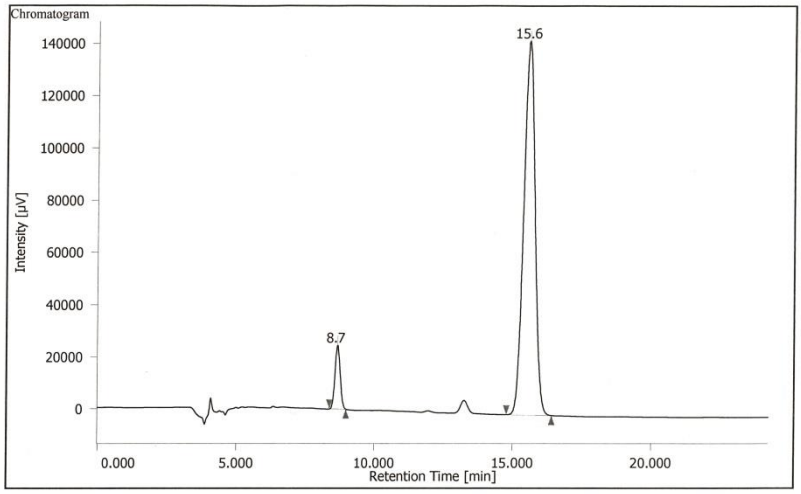


Peak Information

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	NTP	Resolution	Symmetry Factor
1	Unknown	1	8.742	4698441	341851	49.370	9203	12.533	1.063
2	Unknown	1	15.683	4818351	172688	50.630	7067	N/A	1.063

Figure: HPLC graph of Racemic 9

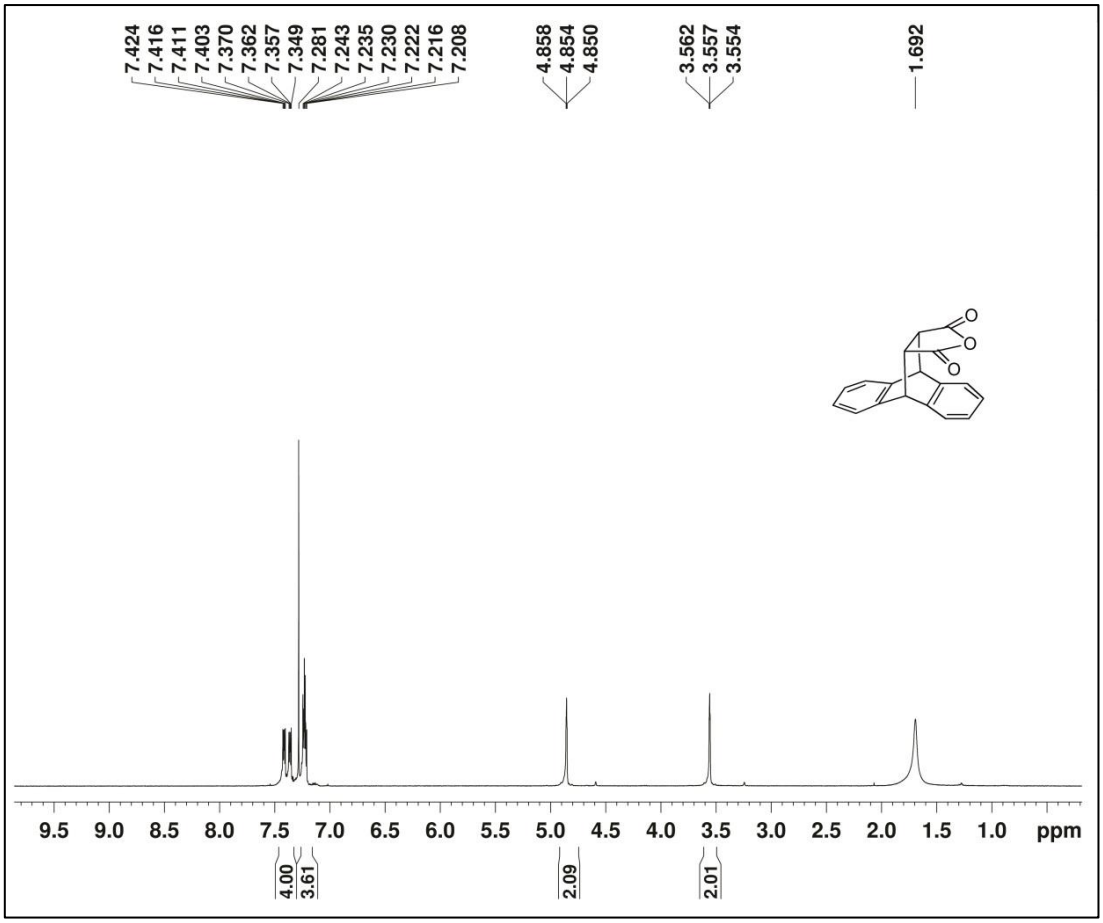
HPLC REPORT



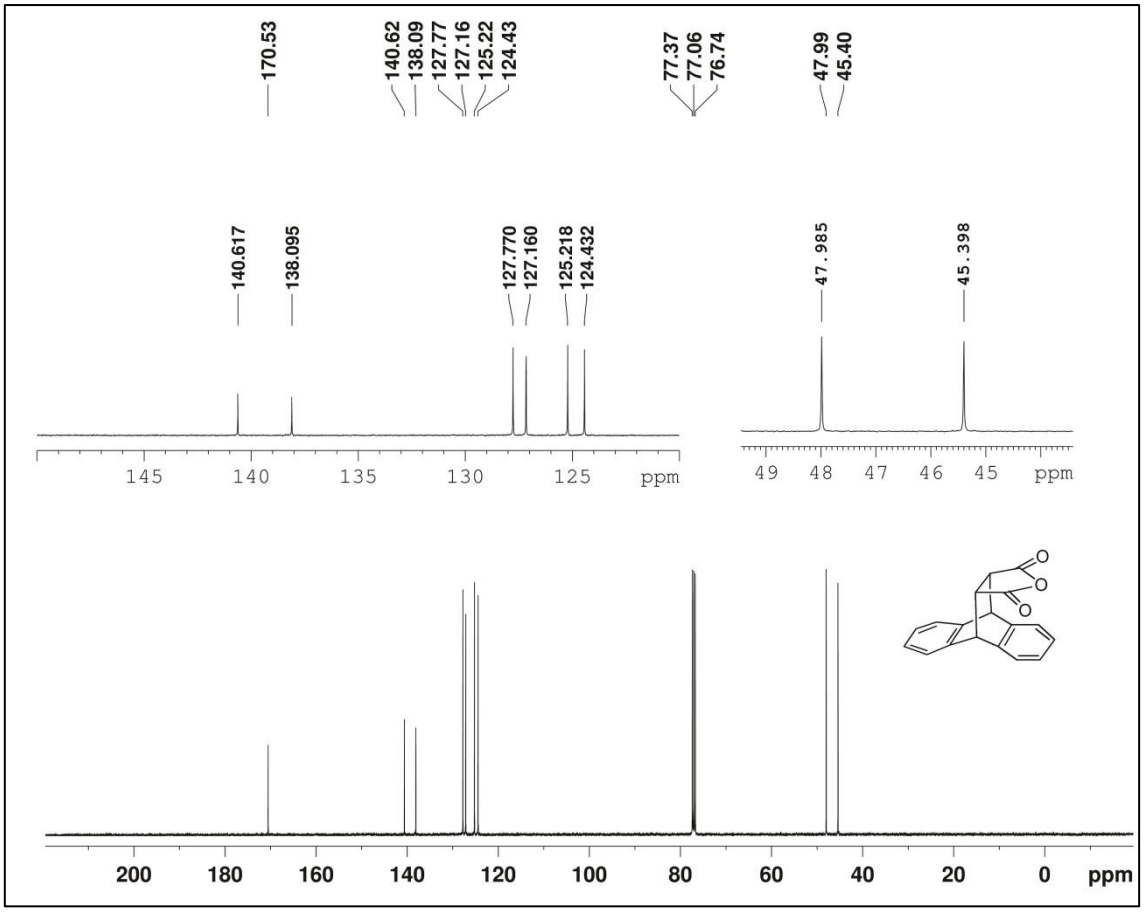
Peak Information

#	Peak Name	CH	tR [min]	Area [µV·sec]	Height [µV]	Area%	NTP	Resolution	Symmetry Factor
1	Unknown	1	8.650	326690	24528	7.502	9502	12.560	1.040
2	Unknown	1	15.550	4027862	143439	92.498	6939	N/A	1.047

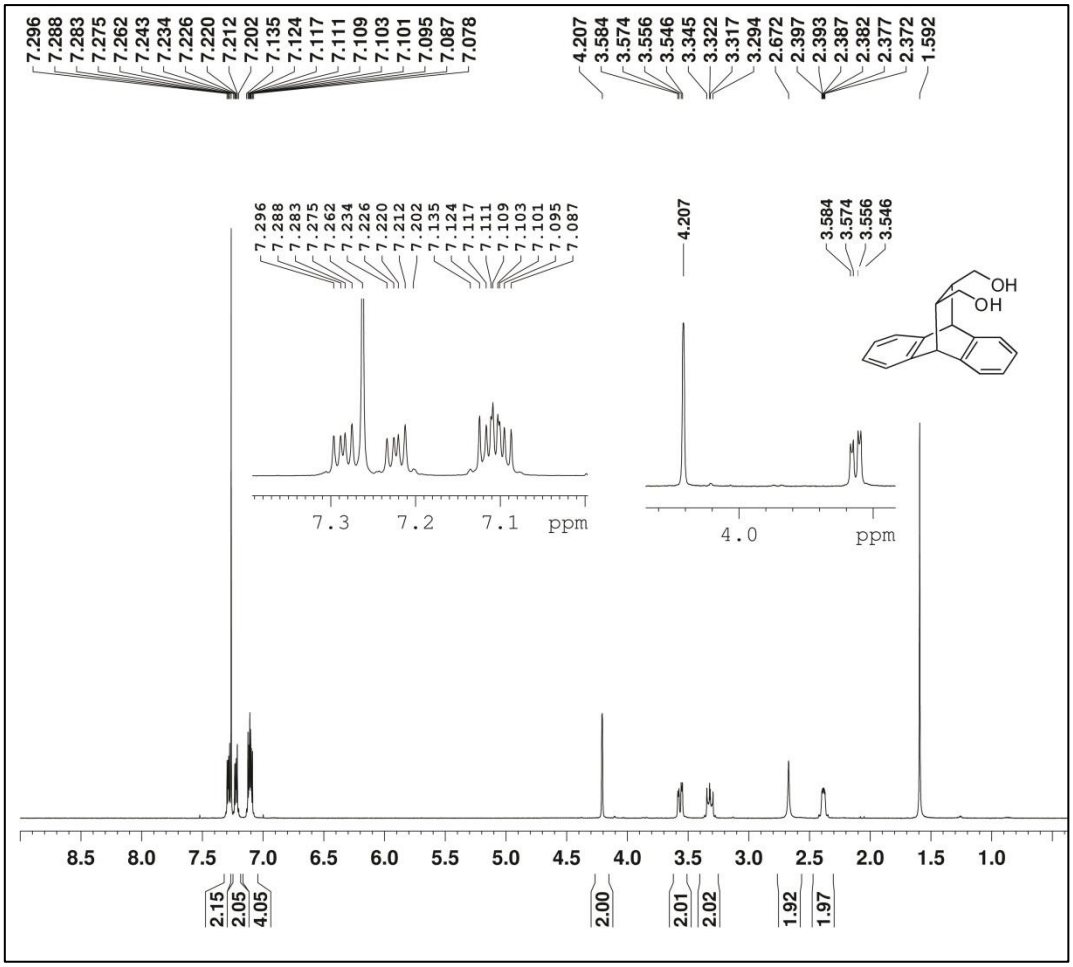
Figure: HPLC graph of Chiral 9



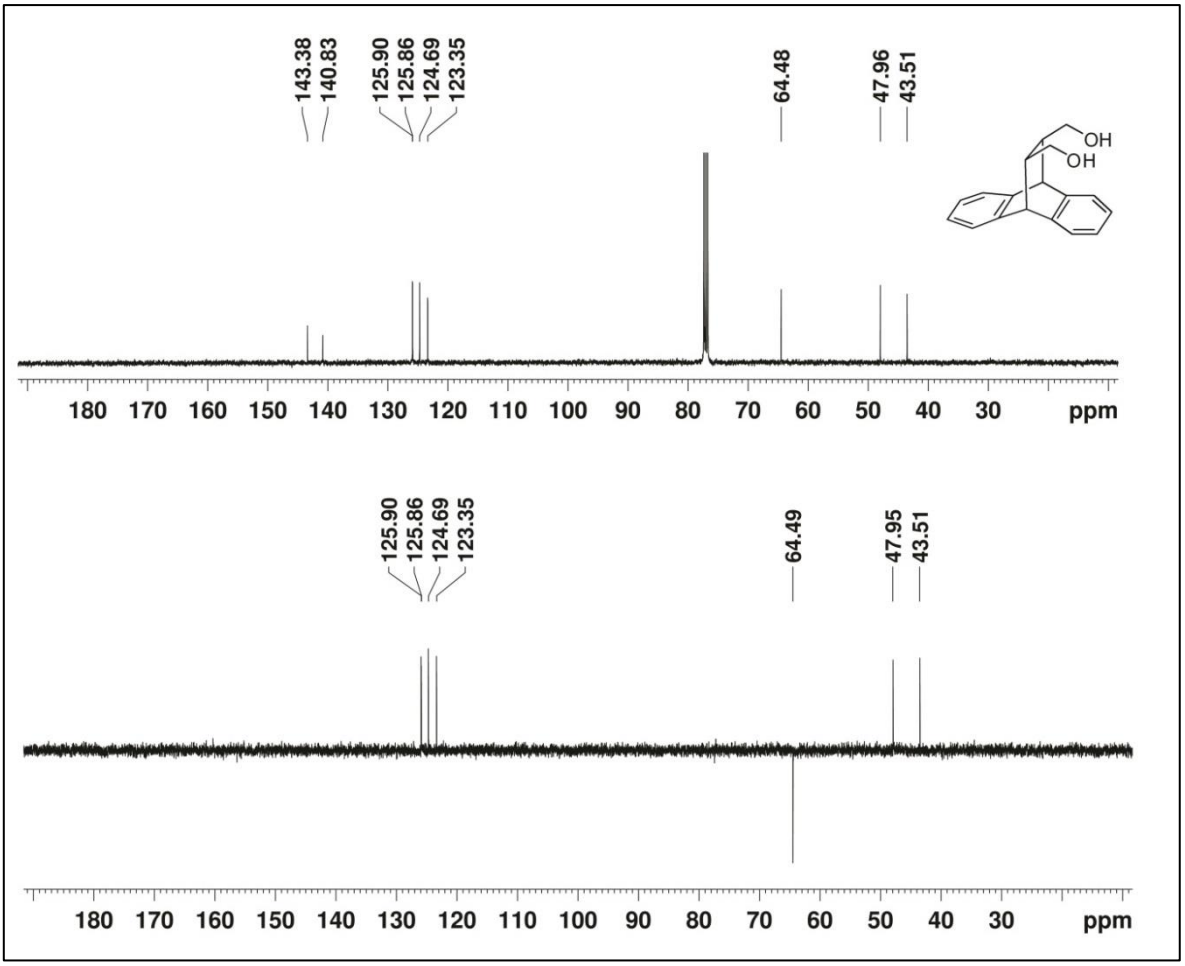
^{1}H NMR spectra of compound 4



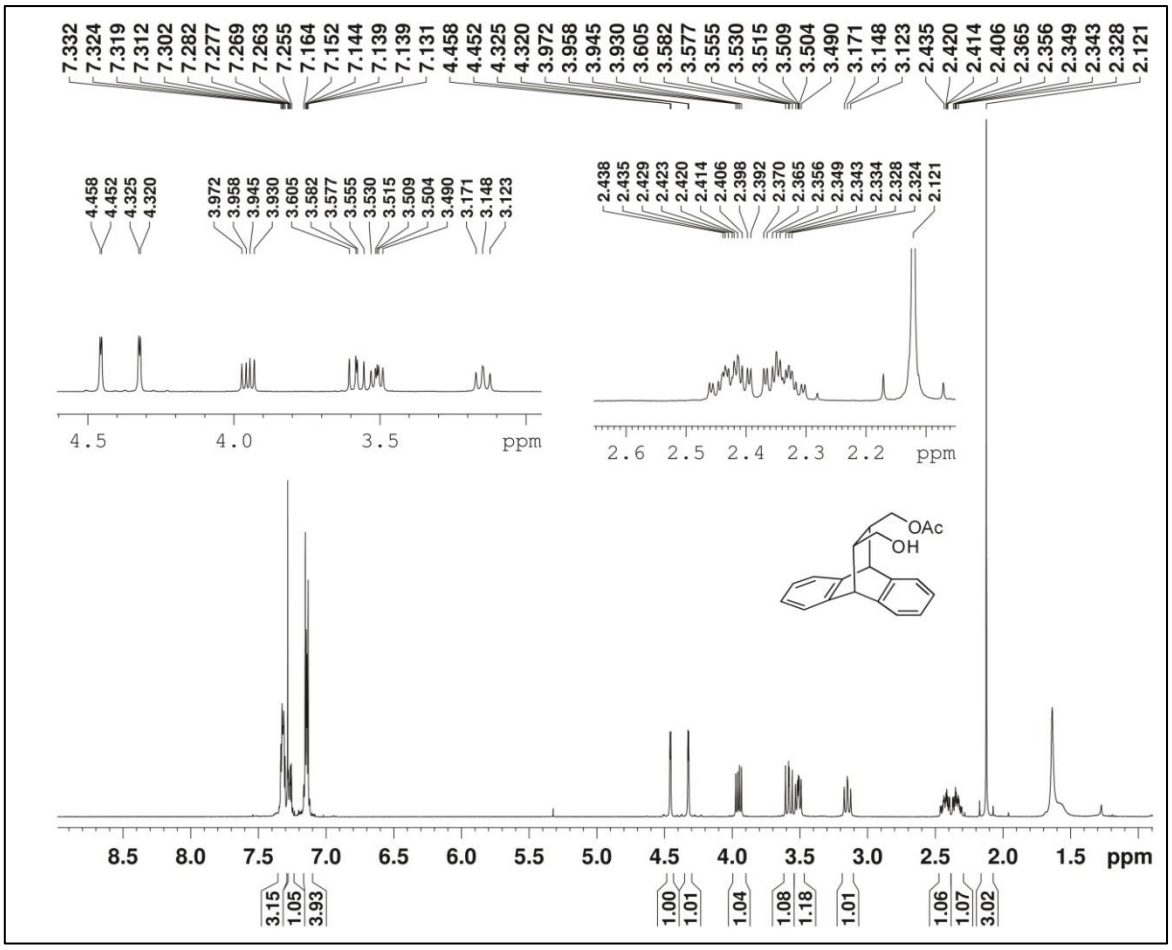
^{13}C NMR spectra of compound 4



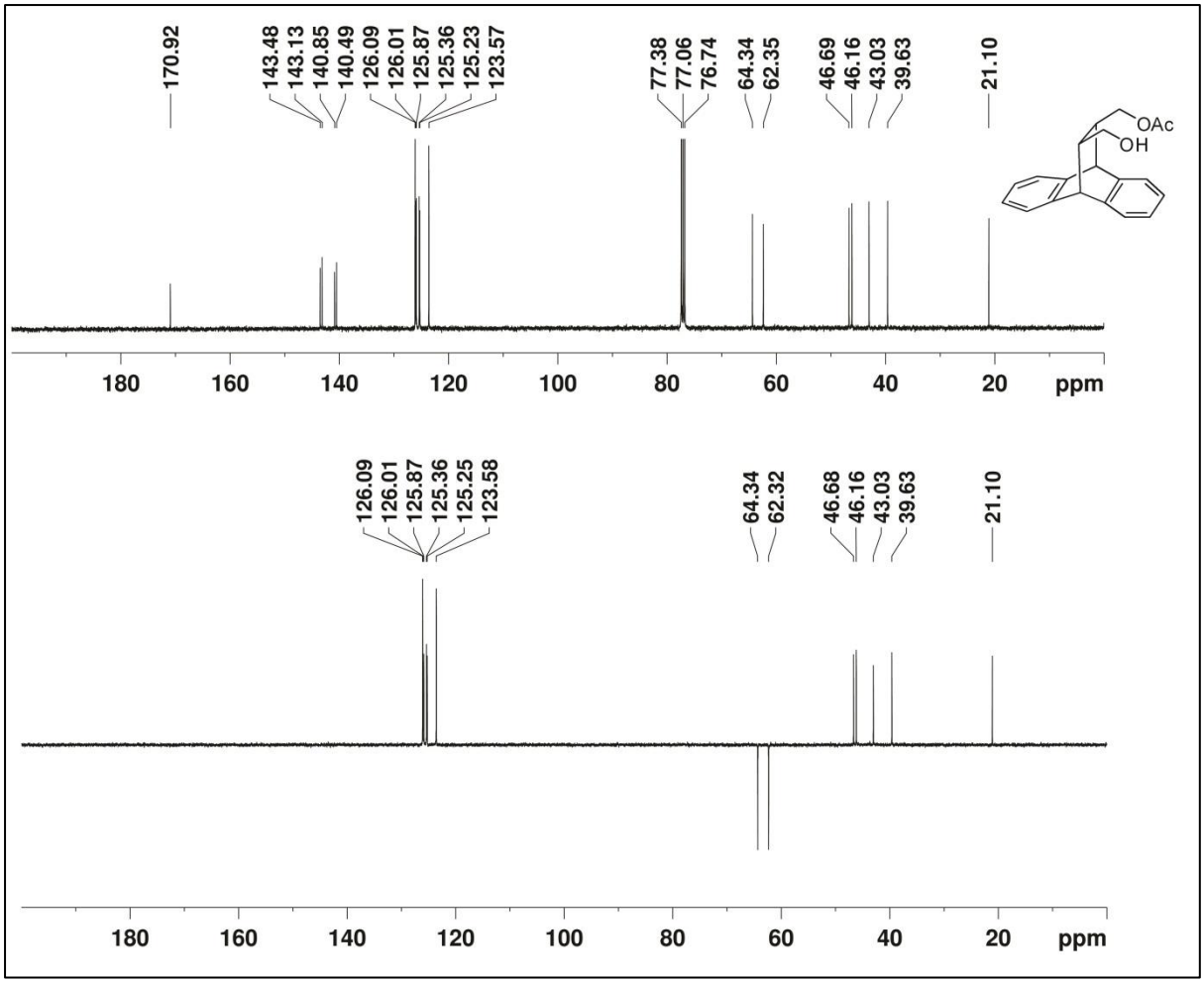
^{1}H NMR spectra of compound 3



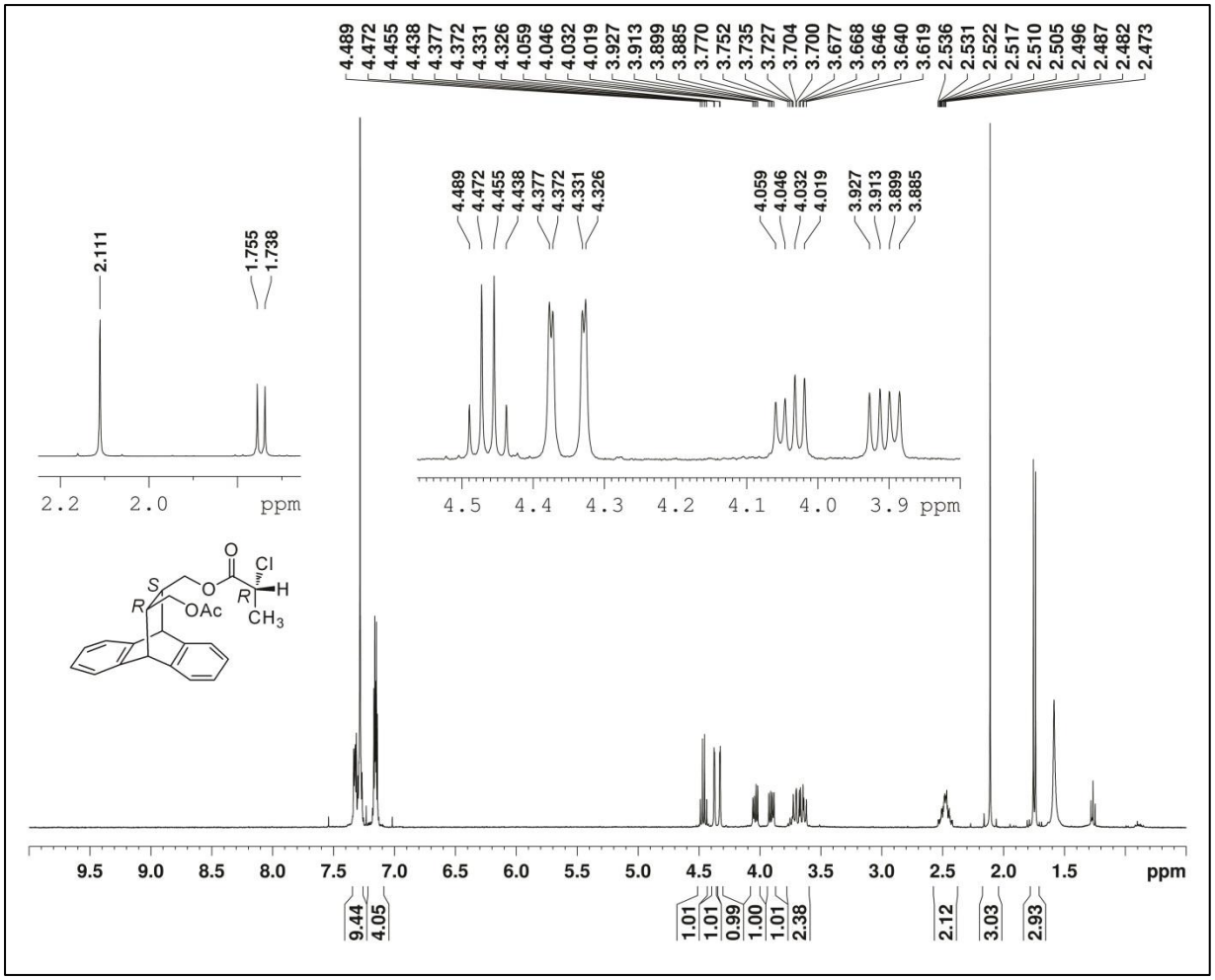
^{13}C NMR & DEPT-135 spectra of compound 3



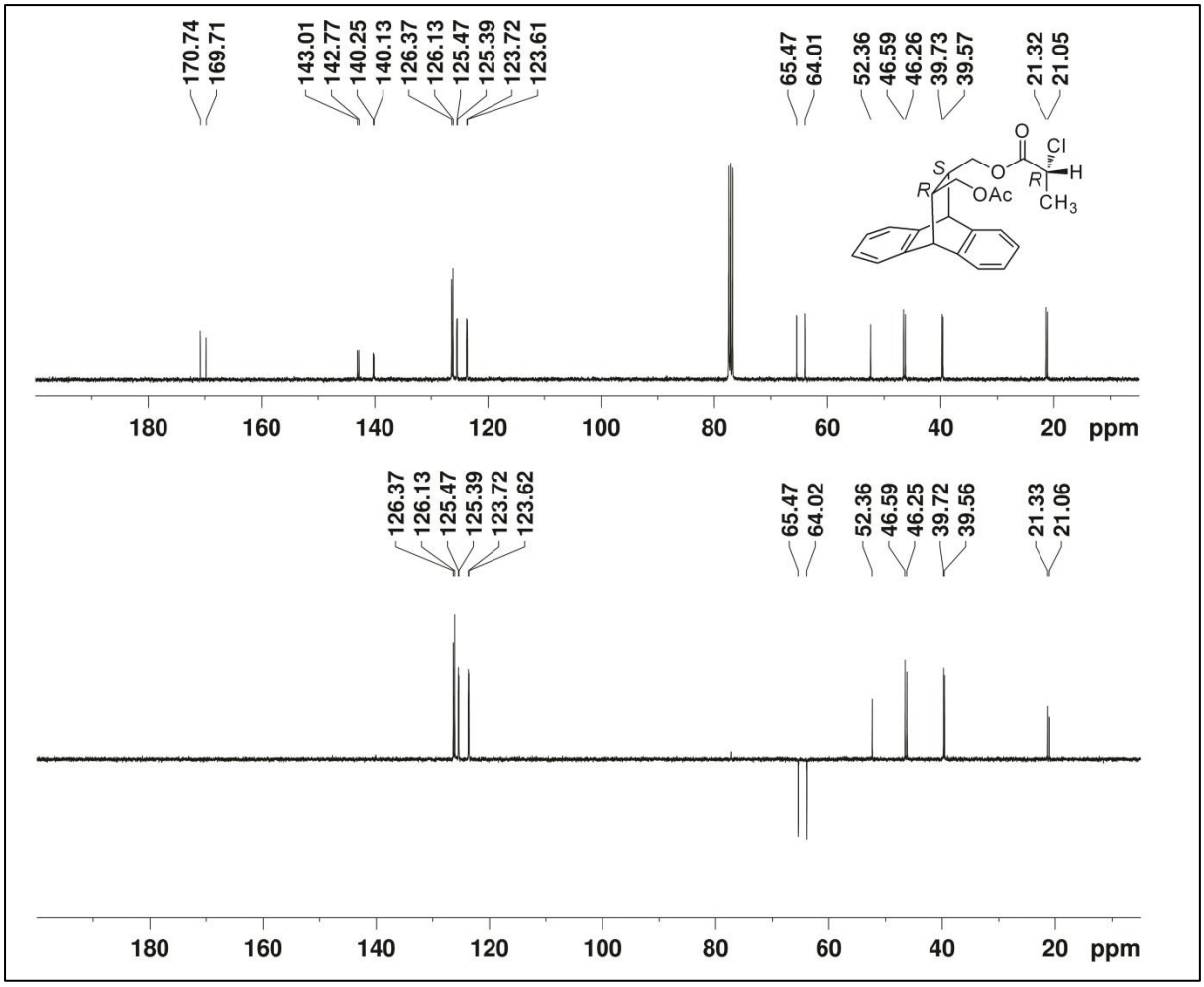
^{1}H NMR spectra of compound 5



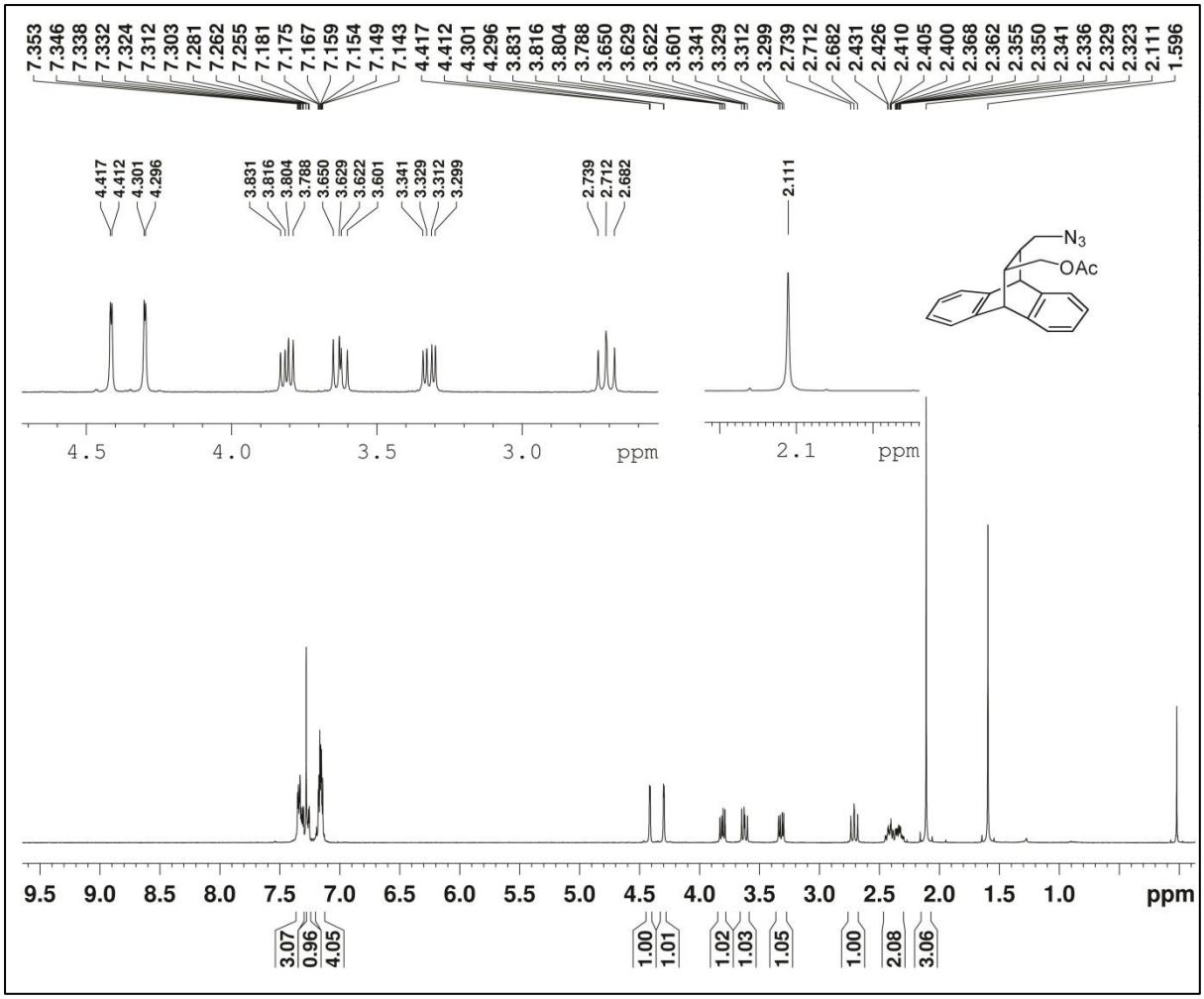
^{13}C NMR & DEPT-135 spectra of compound 5



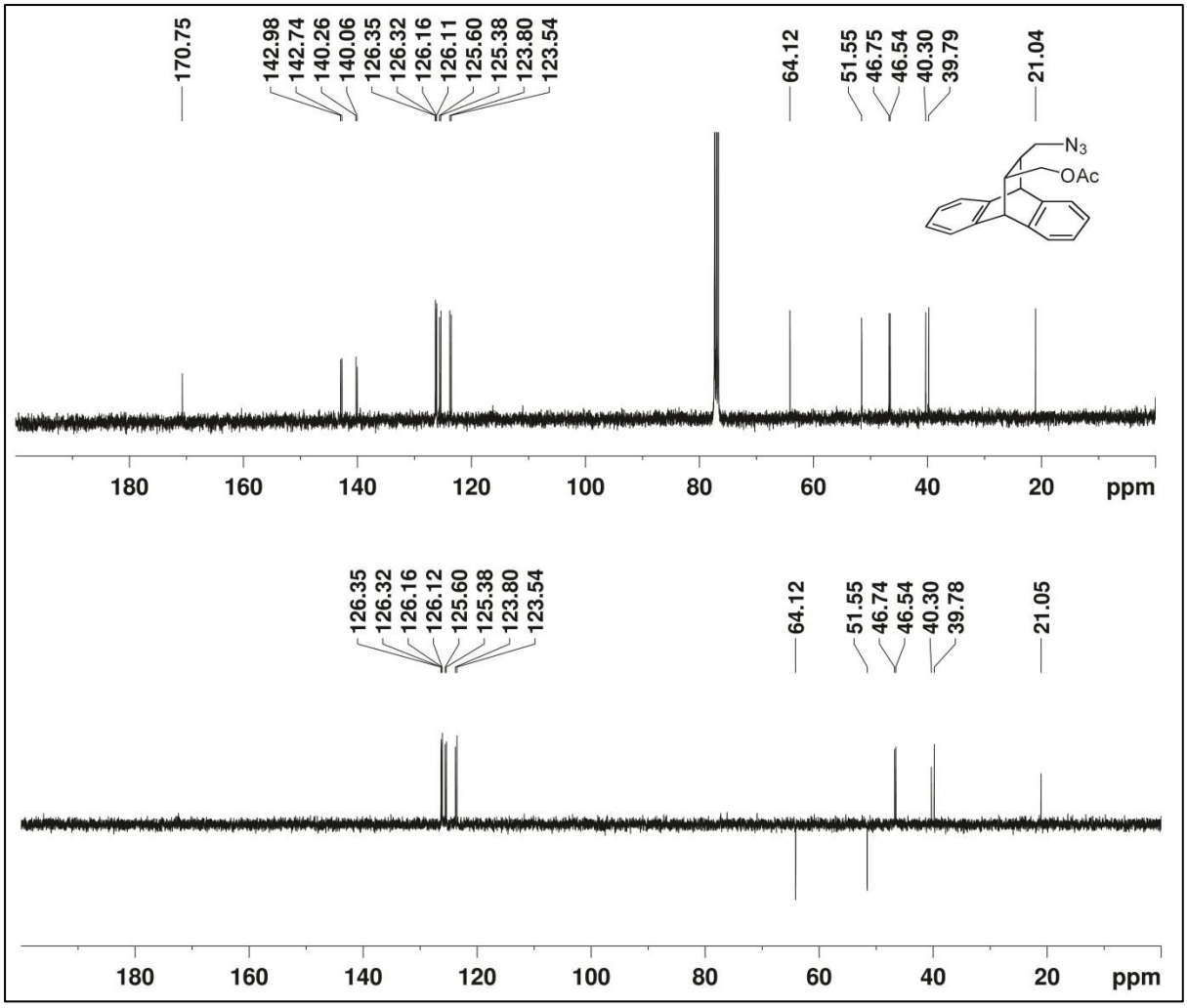
^{1}H NMR spectra of compound 7



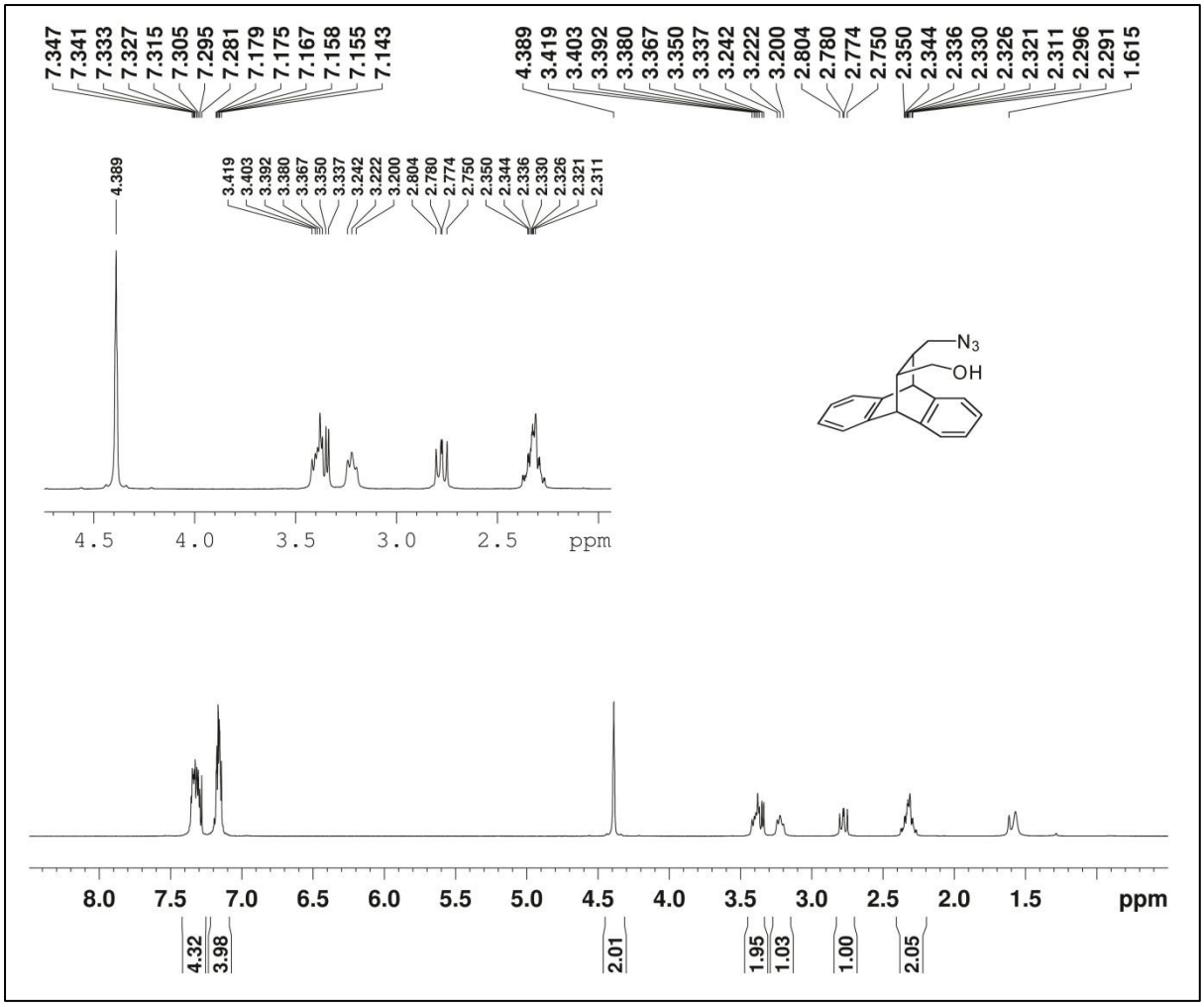
^{13}C NMR & DEPT-135 spectra of compound 7



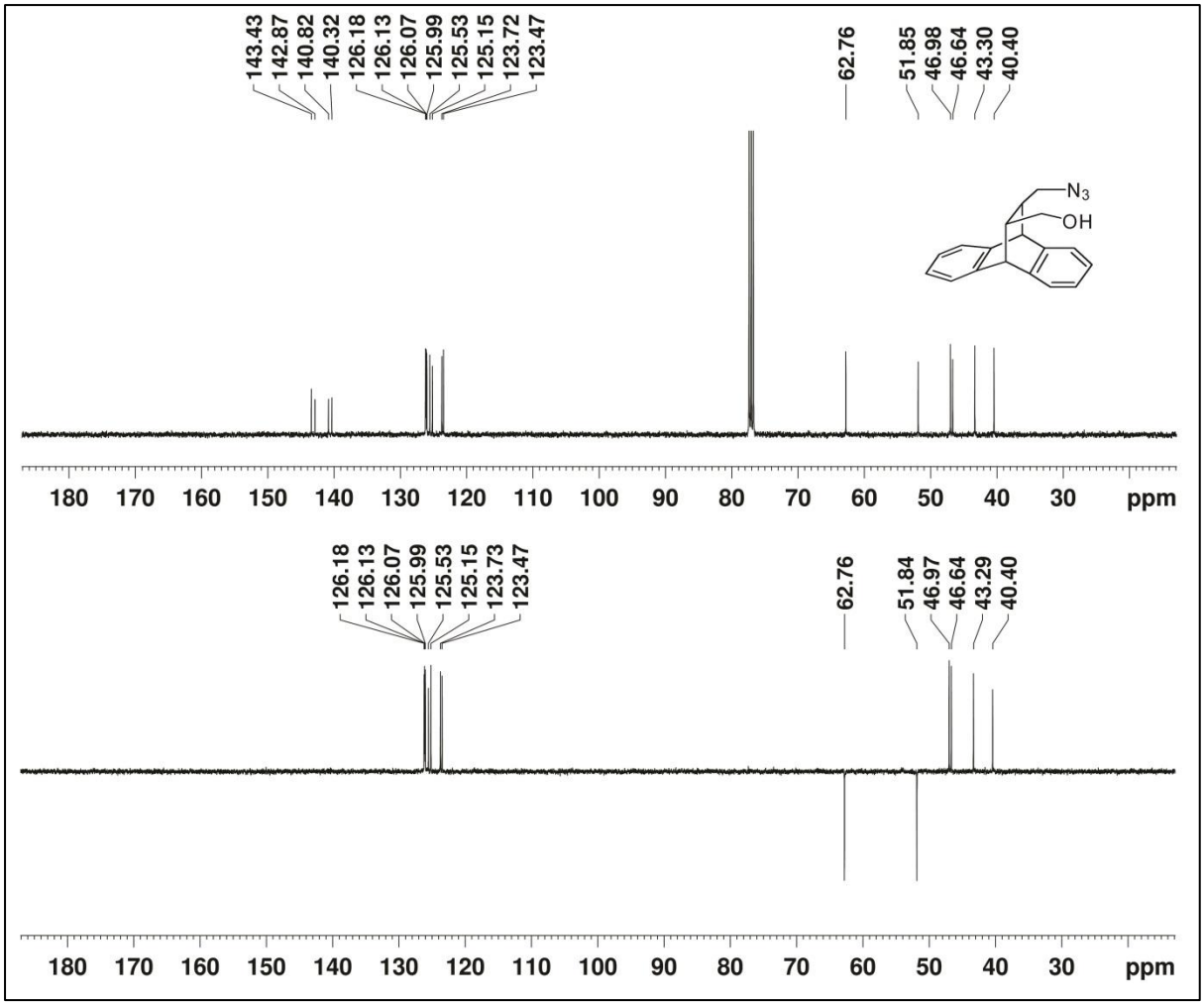
^{1}H NMR spectra of compound 8



^{13}C NMR & DEPT-135 spectra of compound 8



^{1}H NMR spectra of compound 9



^{13}C NMR & DEPT-135 spectra of compound 9

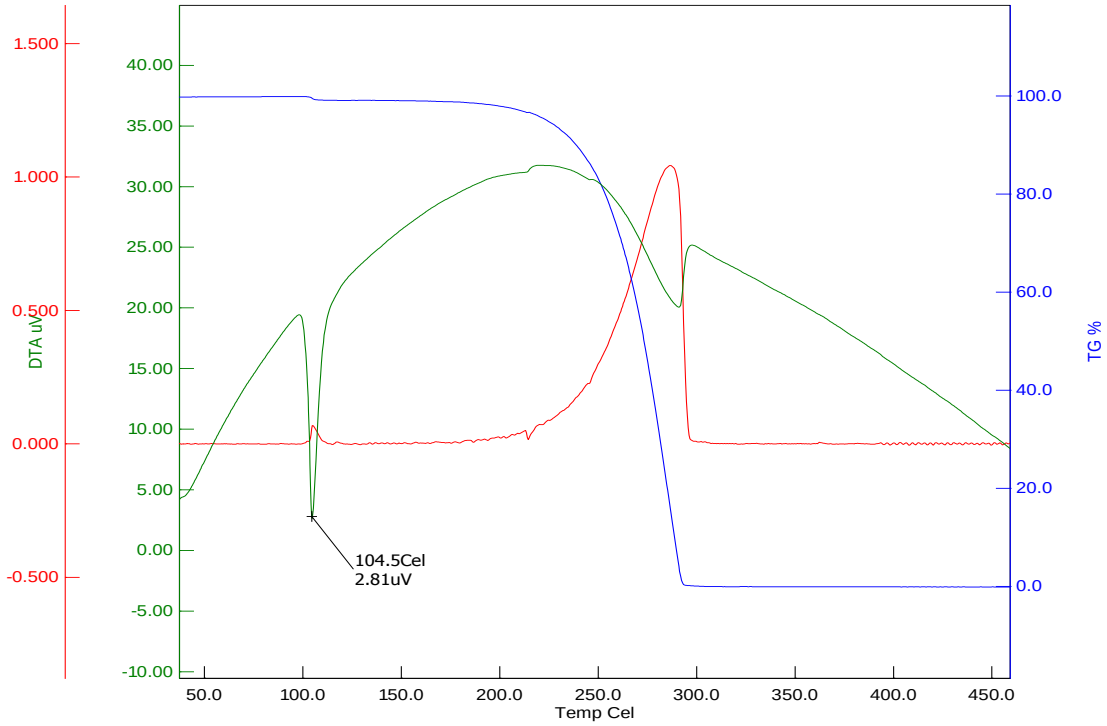


Figure TG-DTA analysis of Chiral crystal 5

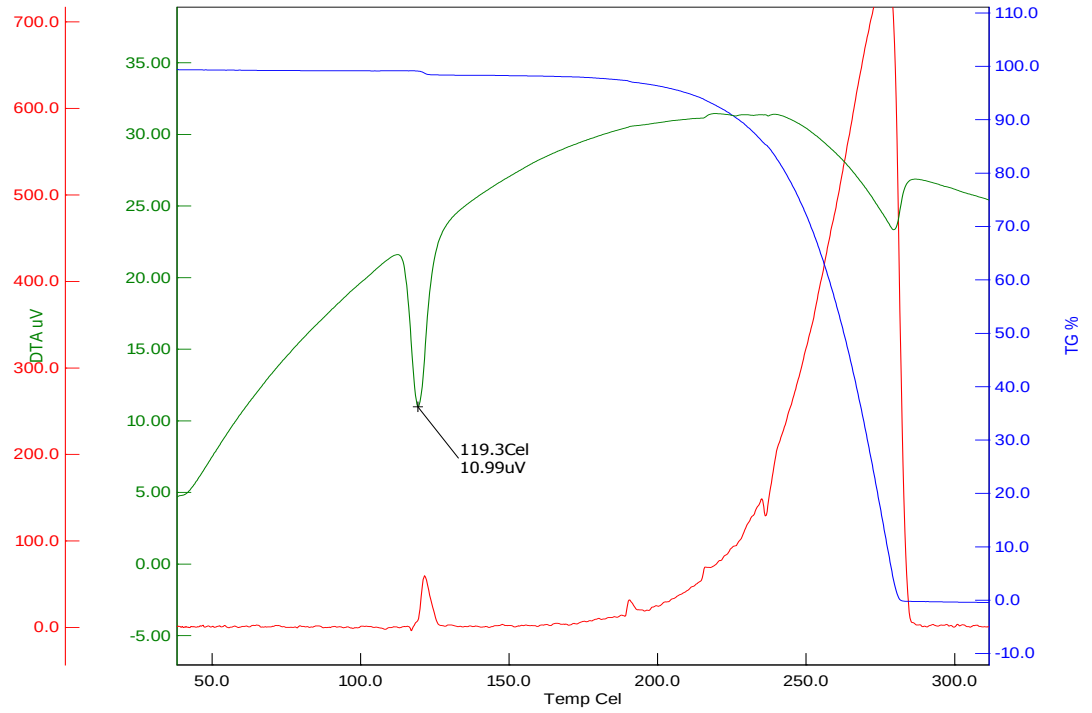


Figure TG-DTA analysis of Racemic Crystal 5

Crystal Data for Chiral **5 CCDC number = 1020854**

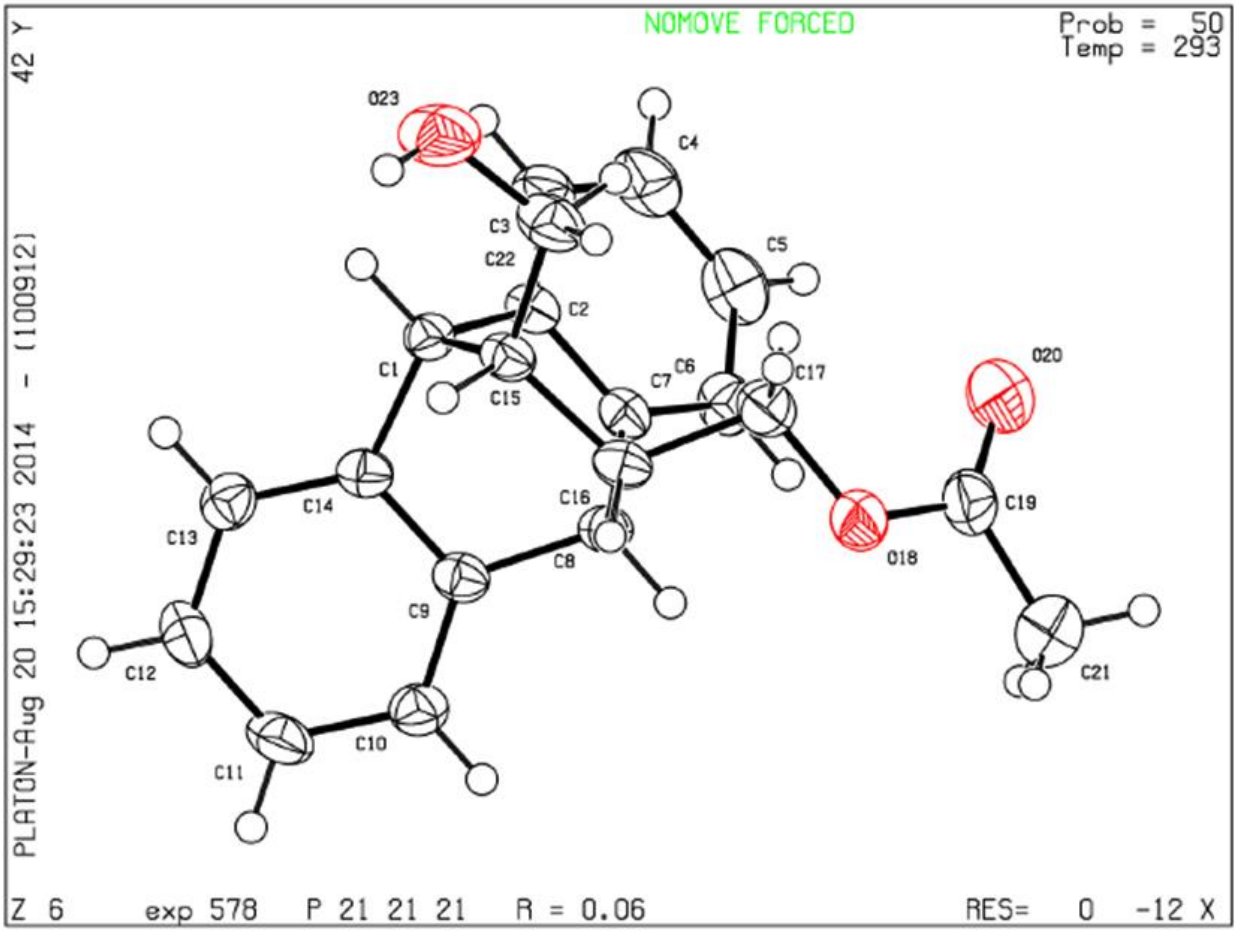


Table 1 Crystal data and structure refinement for 5

Identification code	exp_578
Empirical formula	$C_{20}H_{20}O_3$
Formula weight	308.36
Temperature/K	293(2)
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a/Å	7.35270(18)
b/Å	13.6016(4)
c/Å	15.7824(4)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	1578.37(7)
Z	4
ρ_{calc}mg/mm^3	1.298
μ/mm^{-1}	0.690
F(000)	656.0
2Θ range for data collection	8.58 to 150.82°
Index ranges	$-9 \leq h \leq 9$, $-17 \leq k \leq 11$, $-19 \leq l \leq 18$
Reflections collected	8107
Independent reflections	3085[R(int) = 0.0552]
Data/restraints/parameters	3085/0/210
Goodness-of-fit on F^2	1.075
Final R indexes [I>=2σ (I)]	R_1 = 0.0698, wR_2 = 0.1900
Final R indexes [all data]	R_1 = 0.0708, wR_2 = 0.1924
Largest diff. peak/hole / e Å^{-3}	0.29/-0.53
Flack parameter	0.0(4)

Table 2 Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for exp_578. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*X*	*y*	*z*	U(eq)
O18	8730(3)	3257.2(14)	7295.0(12)	40.9(5)
O23	5148(4)	6213.4(15)	9254.3(13)	54.1(6)
O20	8841(4)	3981.8(18)	6023.9(13)	55.6(6)
C9	3485(3)	2758.6(17)	8330.0(14)	27.0(5)
C12	638(4)	2384(2)	9439.9(16)	37.3(6)
C2	3271(3)	4594.8(16)	7498.2(14)	27.1(5)
C14	2518(3)	3536.9(16)	8702.8(14)	25.5(5)
C1	3274(3)	4532.8(16)	8455.9(14)	26.4(5)
C10	3043(4)	1799.5(17)	8530.8(15)	31.8(5)
C13	1107(3)	3346.8(18)	9252.3(14)	30.3(5)
C3	2552(3)	5349.5(19)	7009.8(17)	35.1(6)
C8	4959(3)	3090.2(16)	7732.5(13)	26.5(5)
C7	4135(3)	3802.4(17)	7110.3(14)	26.0(5)
C19	9124(4)	3284(2)	6473.1(18)	39.3(6)
C15	5299(3)	4521.1(16)	8759.4(14)	26.9(5)
C22	6146(4)	5542.6(19)	8753.0(16)	39.7(6)
C17	7858(3)	4098.6(19)	7682.2(17)	35.1(6)
C11	1602(4)	1607.9(19)	9085.9(17)	39.2(6)
C16	6356(3)	3705.2(16)	8260.2(14)	27.8(5)
C6	4250(4)	3753(2)	6234.3(15)	34.7(5)
C4	2690(4)	5293(2)	6132.9(19)	45.0(7)
C5	3516(4)	4507(2)	5754.3(16)	44.6(7)
C21	9959(6)	2334(3)	6195(3)	67.8(10)

Table 3 Anisotropic Displacement Parameters (Å²×10³) for exp_578. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka\times b\times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O18	41.1(9)	40.1(10)	41.6(10)	10.8(8)	9.8(8)	7.5(8)
O23	84.8(16)	33.9(10)	43.6(10)	-6.9(8)	-1.0(12)	-5.6(11)
O20	68.9(14)	56.8(13)	41.2(11)	9.5(10)	12.0(11)	3.8(13)
C9	33(1)	24(1)	24.0(9)	1.6(8)	-3.3(9)	-1.5(9)
C12	37.9(12)	42.4(14)	31.7(11)	1.9(11)	9.2(10)	-9.7(11)
C2	30.8(9)	23.3(10)	27.3(11)	3.6(8)	-0.4(9)	-2.9(9)
C14	31.3(10)	23(1)	22.1(10)	0.5(8)	-3.5(8)	-0.6(9)
C1	34.6(11)	21.2(10)	23.3(10)	-0.8(8)	3.5(9)	1.1(8)
C10	39.3(11)	25.0(11)	31.3(11)	-3.0(9)	1.1(10)	-1.5(9)
C13	34.8(11)	32.0(11)	24(1)	-2.5(9)	-0.2(9)	2.7(10)

C3	35.5(11)	29.1(11)	40.7(13)	9.5(10)	-0.9(11)	2.4(10)
C8	32.9(10)	21(1)	25.5(10)	0.4(8)	-0.3(9)	0.0(9)
C7	29.9(10)	26.2(10)	21.9(10)	3.3(8)	0.3(9)	-3.1(9)
C19	35.2(11)	41.3(14)	41.4(14)	-0.1(11)	9.6(11)	-5.0(11)
C15	36.9(11)	22.6(10)	21.3(9)	1.5(8)	-0.3(9)	-3.2(9)
C22	53.4(14)	31.2(12)	34.7(12)	-0.5(10)	-0.7(12)	-12.9(12)
C17	33.1(11)	33.4(12)	38.9(13)	4.7(10)	1.2(11)	-1.5(10)
C11	54.8(15)	24.5(11)	38.2(13)	2.3(10)	2.5(12)	-10.4(11)
C16	32.6(10)	23.9(10)	27(1)	5.5(9)	-4.6(9)	0.0(9)
C6	41.8(12)	38.9(13)	23.5(11)	-1.1(10)	1.8(10)	-4.0(11)
C4	44.8(13)	53.6(17)	36.5(13)	22.1(12)	-7.1(12)	2.6(14)
C5	50.3(14)	60.0(17)	23.4(11)	7.4(12)	-4.9(11)	-4.9(14)
C21	77(2)	51.0(19)	75(2)	-1.5(17)	31(2)	5(2)

Table 4 Bond Lengths for exp_578.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O18	C19	1.330(3)	C14	C13	1.377(3)
O18	C17	1.447(3)	C1	C15	1.564(3)
O23	C22	1.413(4)	C10	C11	1.399(4)
O20	C19	1.203(4)	C3	C4	1.390(4)
C9	C14	1.405(3)	C8	C7	1.507(3)
C9	C10	1.381(3)	C8	C16	1.564(3)
C9	C8	1.506(3)	C7	C6	1.387(3)
C12	C13	1.386(4)	C19	C21	1.497(4)
C12	C11	1.389(4)	C15	C22	1.523(3)
C2	C1	1.514(3)	C15	C16	1.567(3)
C2	C3	1.388(3)	C17	C16	1.529(3)
C2	C7	1.393(3)	C6	C5	1.385(4)
C14	C1	1.515(3)	C4	C5	1.367(5)

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom
C19	O18	C17	119.1(2)	C7	C8	C16
C14	C9	C8	113.65(19)	C2	C7	C8
C10	C9	C14	119.8(2)	C6	C7	C2
C10	C9	C8	126.6(2)	C6	C7	C8
C13	C12	C11	120.3(2)	O18	C19	C21
C3	C2	C1	126.6(2)	O20	C19	O18
C3	C2	C7	120.1(2)	O20	C19	C21
C7	C2	C1	113.27(19)	C1	C15	C16
C9	C14	C1	112.34(19)	C22	C15	C1
C13	C14	C9	120.3(2)	C22	C15	C16
C13	C14	C1	127.3(2)	O23	C22	C15
C2	C1	C14	107.81(18)	O18	C17	C16
C2	C1	C15	107.95(17)	C12	C11	C10
C14	C1	C15	105.16(18)	C8	C16	C15
C9	C10	C11	119.9(2)	C17	C16	C8
C14	C13	C12	120.0(2)	C17	C16	C15
C2	C3	C4	119.0(3)	C5	C6	C7
C9	C8	C7	108.12(19)	C5	C4	C3
C9	C8	C16	107.41(18)	C4	C5	C6

Table 6 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for exp_578.

Atom	*x*	*y*	*z*	U(eq)
H23	5157	6030	9749	81
H12	-328	2257	9804	45
H1	2585	5072	8714	32
H10	3702	1282	8298	38
H13	469	3864	9498	36
H3	1987	5884	7265	42
H8	5542	2534	7446	32
H15	5278	4310	9353	32
H22A	7382	5502	8965	48
H22B	6197	5783	8175	48
H17A	7344	4523	7250	42
H17B	8733	4474	8009	42
H11	1292	963	9218	47
H16	6920	3265	8675	33
H6	4812	3221	5973	42
H4	2215	5795	5800	54
H5	3586	4479	5166	53
H21A	10589	2039	6663	102
H21B	10803	2457	5743	102
H21C	9022	1898	6000	102

Single crystals of $C_{20}H_{20}O_3$. A suitable crystal was selected and on a Gemini diffractometer. The crystal was kept at 293(2) K during data collection. Using Olex2 [1], the structure was solved with the Superflip structure solution program using Charge Flipping and refined with the ShelXL refinement package using Least Squares minimisation. **Crystal Data** for $C_{20}H_{20}O_3$ (M =308.36): orthorhombic, space group $P2_12_12_1$ (no. 19), a = 7.35270(18) Å, b = 13.6016(4) Å, c = 15.7824(4) Å, V = 1578.37(7) Å^3,Z = 4, T = 293(2) K, μ(Cu Kα) = 0.690 mm^{-1}, *Dcalc* = 1.298 g/mm^3, 8107 reflections measured ($8.58 \leq 2\Theta \leq 150.82$), 3085 unique ($R_{int}$ = 0.0552) which were used in all calculations. The final R_1 was 0.0698 (>2sigma(I)) and wR_2 was 0.1924 (all data).

Crysral data of racemic 5 **CCDC No.1020855**

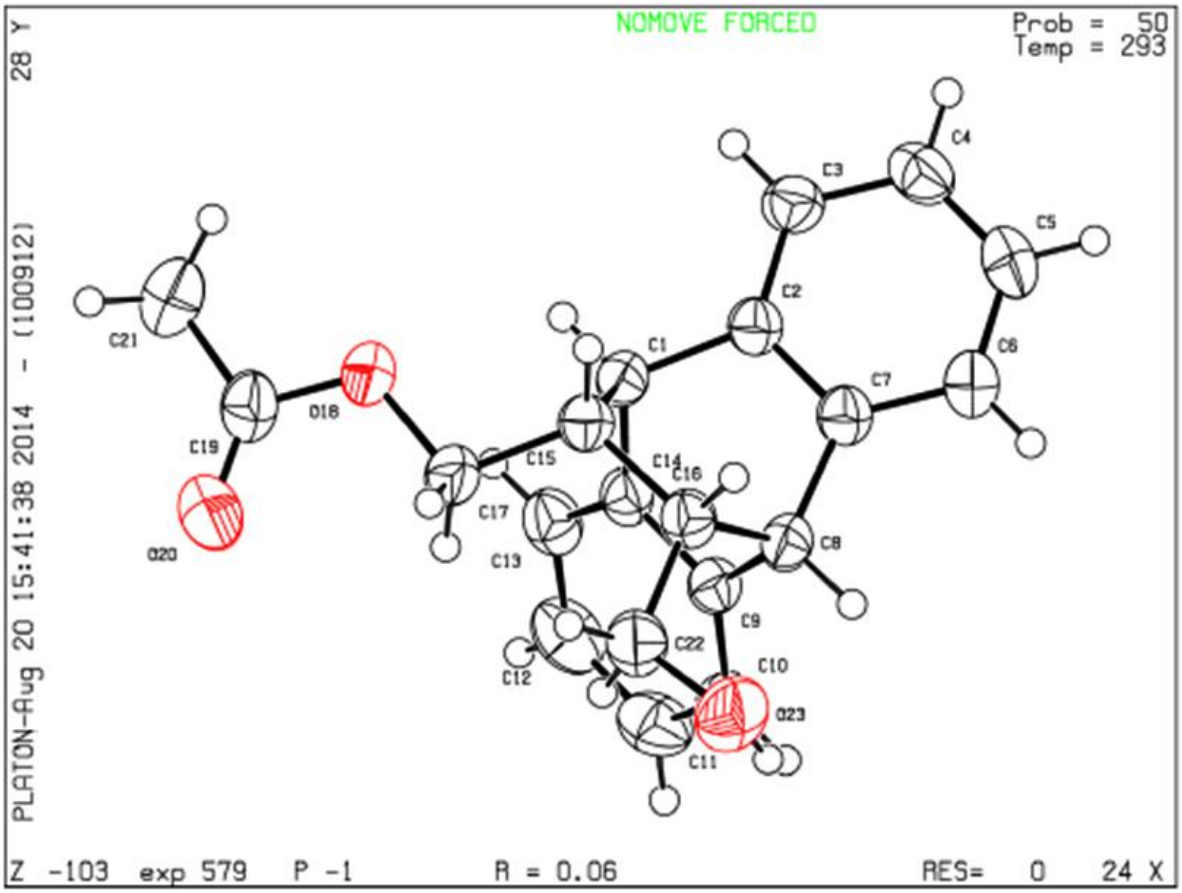


Table 1 Crystal data and structure refinement for exp_579

Identification code	exp_579
Empirical formula	$C_{20}H_{20}O_3$
Formula weight	308.36
Temperature/K	293(2)
Crystal system	Triclinic
Space group	P-1
a/Å	8.2328(5)
b/Å	8.8177(4)
c/Å	11.1343(7)
α/°	95.635(4)
β/°	97.037(5)
γ/°	95.431(4)
Volume/Å^3	793.65(8)
Z	2
ρ_{calc}mg/mm^3	1.290
μ/mm^{-1}	0.686
F(000)	328.0
Crystal size/mm^3	? × ? × ?
2Θ range for data collection	10.14 to 150.68°
Index ranges	$-10 \leq h \leq 9$, $-6 \leq k \leq 11$, $-13 \leq l \leq 13$
Reflections collected	4910
Independent reflections	3114[R(int) = 0.0212]
Data/restraints/parameters	3114/0/210
Goodness-of-fit on F^2	1.114
Final R indexes [I>=2σ (I)]	R_1 = 0.0549, wR_2 = 0.1525
Final R indexes [all data]	R_1 = 0.0590, wR_2 = 0.1568
Largest diff. peak/hole / e Å^{-3}	0.23/-0.19

Table 2 Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for exp_579. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O18	4058.8(16)	6241.2(13)	8524.3(12)	51.9(3)
C7	1247.2(19)	899.8(17)	6986.7(14)	38.6(3)
C2	2249.9(19)	1979.3(17)	6489.7(14)	38.5(3)
O23	501(2)	1698.9(17)	10746.0(13)	65.7(4)
C9	-508.8(19)	2853.5(16)	7547.5(13)	37.7(3)
C3	3090(2)	1518.2(19)	5539.8(15)	44.5(4)
C8	430.6(19)	1596.2(16)	8022.0(14)	38.5(3)
C16	1851(2)	2372.5(16)	9021.1(14)	39.4(3)
C15	2916.2(19)	3629.7(17)	8462.5(14)	40.0(3)
C1	2244.0(19)	3596.9(16)	7082.1(13)	38.4(3)
C14	471(2)	3927.2(16)	7027.2(13)	37.7(3)
C6	1068(2)	-627.8(17)	6518.1(15)	44.9(4)
O20	3243(2)	8262.1(17)	9563.2(17)	84.8(6)
C10	-2159(2)	3010(2)	7580.4(15)	46.3(4)
C13	-207(2)	5150.3(18)	6551.3(15)	47.7(4)
C11	-2831(2)	4229(2)	7086.4(17)	56.1(5)
C4	2889(2)	-17(2)	5061.7(16)	49.5(4)
C5	1887(2)	-1080.0(19)	5543.1(16)	49.7(4)
C17	2966(2)	5241.1(18)	9108.9(16)	46.7(4)
C22	1184(2)	2938.4(19)	10181.5(15)	49.0(4)
C12	-1861(3)	5290(2)	6574.6(17)	57.4(5)
C19	4061(2)	7749.2(19)	8825.1(17)	51.7(4)
C21	5176(3)	8679(2)	8155(2)	62.2(5)

Table 3 Anisotropic Displacement Parameters (Å²×10³) for exp_579. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka\times b\times U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O18	55.2(7)	39.8(6)	57.9(7)	-5.4(5)	15.6(6)	-8.7(5)
C7	42.9(8)	35.5(7)	36.0(7)	1.1(6)	3.3(6)	2.1(6)
C2	39.4(7)	37.0(7)	37.3(7)	-0.3(6)	4.0(6)	1.4(6)
O23	86(1)	62.1(8)	51.8(8)	17.1(6)	20.6(7)	-3.9(7)
C9	41.5(8)	36.0(7)	33.9(7)	-1.6(6)	6.1(6)	-0.3(6)
C3	43.0(8)	48.9(9)	40.3(8)	-0.9(7)	8.2(6)	1.2(7)
C8	43.7(8)	32.4(7)	38.9(8)	2.9(6)	8.8(6)	-1.5(6)
C16	46.1(8)	35.4(7)	36.5(7)	3.1(6)	5.8(6)	3.4(6)
C15	39.8(8)	39.8(8)	38.4(8)	-1.6(6)	5.0(6)	-0.3(6)

C1	43.5(8)	34.0(7)	36.7(7)	0.8(6)	9.9(6)	-3.1(6)
C14	46.2(8)	33.1(7)	32.5(7)	-0.5(5)	5.2(6)	1.4(6)
C6	51.2(9)	35.4(7)	45.5(9)	1.1(6)	3.0(7)	0.1(6)
O20	109.1(14)	49.9(8)	98.1(13)	-12.0(8)	49.0(11)	-1.9(8)
C10	43.2(8)	52.4(9)	41.5(8)	-2.6(7)	8.4(7)	0.9(7)
C13	64.5(11)	38.7(8)	40.8(8)	4.1(6)	9.0(7)	7.5(7)
C11	49(1)	67.8(11)	51.4(10)	-1.7(8)	4.0(8)	17.9(8)
C4	51.0(9)	53.8(9)	42.0(8)	-8.1(7)	6.1(7)	10.1(7)
C5	59.5(10)	38.4(8)	47.6(9)	-7.1(7)	1.5(8)	6.6(7)
C17	50.0(9)	41.9(8)	44.7(8)	-6.1(6)	10.8(7)	-8.4(7)
C22	64.0(11)	44.3(8)	38.9(8)	3.5(7)	12.4(7)	0.7(7)
C12	70.5(12)	54.8(10)	48.9(10)	5.6(8)	1.3(8)	25.4(9)
C19	57.5(10)	41.1(8)	52.1(10)	-2.6(7)	2.8(8)	-4.2(7)
C21	66.3(12)	50.8(10)	66.3(12)	9.4(9)	5.5(10)	-8.5(9)

Table 4 Bond Lengths for exp_579.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O18	C17	1.4550(19)	C16	C15	1.572(2)
O18	C19	1.339(2)	C16	C22	1.523(2)
C7	C2	1.400(2)	C15	C1	1.565(2)
C7	C8	1.512(2)	C15	C17	1.523(2)
C7	C6	1.383(2)	C1	C14	1.511(2)
C2	C3	1.382(2)	C14	C13	1.382(2)
C2	C1	1.512(2)	C6	C5	1.392(3)
O23	C22	1.418(2)	O20	C19	1.206(2)
C9	C8	1.511(2)	C10	C11	1.385(3)
C9	C14	1.400(2)	C13	C12	1.382(3)
C9	C10	1.383(2)	C11	C12	1.383(3)
C3	C4	1.392(2)	C4	C5	1.381(3)
C8	C16	1.566(2)	C19	C21	1.489(3)

Table 5 Bond Angles for exp_579.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C19	O18	C17	116.27(13)	C17	C15	C1	110.19(13)
C2	C7	C8	113.15(13)	C2	C1	C15	107.63(12)
C6	C7	C2	120.33(14)	C14	C1	C2	107.50(12)
C6	C7	C8	126.51(14)	C14	C1	C15	106.45(12)
C7	C2	C1	112.97(13)	C9	C14	C1	113.19(13)
C3	C2	C7	120.06(14)	C13	C14	C9	120.08(15)

C3	C2	C1	126.96(14)	C13	C14	C1	126.71(14)
C14	C9	C8	112.99(13)	C7	C6	C5	119.35(15)
C10	C9	C8	126.92(14)	C9	C10	C11	119.37(16)
C10	C9	C14	120.09(14)	C14	C13	C12	119.53(16)
C2	C3	C4	119.36(16)	C12	C11	C10	120.45(17)
C7	C8	C16	106.54(12)	C5	C4	C3	120.59(16)
C9	C8	C7	107.29(12)	C4	C5	C6	120.28(15)
C9	C8	C16	107.46(11)	O18	C17	C15	107.04(13)
C8	C16	C15	109.05(11)	O23	C22	C16	111.30(14)
C22	C16	C8	111.30(13)	C13	C12	C11	120.47(16)
C22	C16	C15	114.87(12)	O18	C19	C21	112.40(16)
C1	C15	C16	108.67(11)	O20	C19	O18	122.41(17)
C17	C15	C16	114.04(12)	O20	C19	C21	125.19(17)

Table 6 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for exp_579.

Atom	*x*	*y*	*z*	U(eq)
H23	-477	1497	10478	99
H3	3782	2227	5223	53
H8	-288	826	8341	46
H16	2564	1581	9227	47
H15	4047	3355	8519	48
H1	2895	4342	6687	46
H6	407	-1346	6851	54
H10	-2811	2303	7931	56
H13	446	5875	6218	57
H11	-3942	4335	7099	67
H4	3435	-330	4412	59
H5	1758	-2102	5214	60
H17A	1871	5573	9039	56
H17B	3376	5263	9966	56
H22A	2068	3525	10743	59
H22B	346	3611	9988	59
H12	-2324	6103	6244	69
H21A	6015	8079	7907	93
H21B	5678	9575	8676	93
H21C	4552	8983	7449	93

Single crystals of $C_{20}H_{20}O_3$ [exp_579]. A Suitable crystal was selected and on a Xcalibur, Eos, Gemini diffractometer. The crystal was kept at 293(2) K during data collection. Using Olex 2 the structure was solved with the ShelXS structure solution program using Direct Methods and refined with the ShelXL [3] refinement package using Least Squares minimisation.

Crystal Data for $C_{20}H_{20}O_3$ (M =308.36): triclinic, space group P-1 (no. 2), a = 8.2328(5) Å, b = 8.8177(4) Å, c = 11.1343(7) Å, α = 95.635(4)°, β = 97.037(5)°, γ = 95.431(4)°, V = 793.65(8) Å^3, Z = 2, T = 293(2) K, μ(Cu Kα) = 0.686 mm^{-1}, *Dcalc* = 1.290 g/mm^3, 4910 reflections measured (10.14 ≤ 2Θ ≤ 150.68), 3114 unique (R_{int} = 0.0212) which were used in all calculations. The final R_1 was 0.0549 (>2sigma(I)) and wR_2 was 0.1568 (all data)

Crystal data of compound **7 CCDC number 1054141**

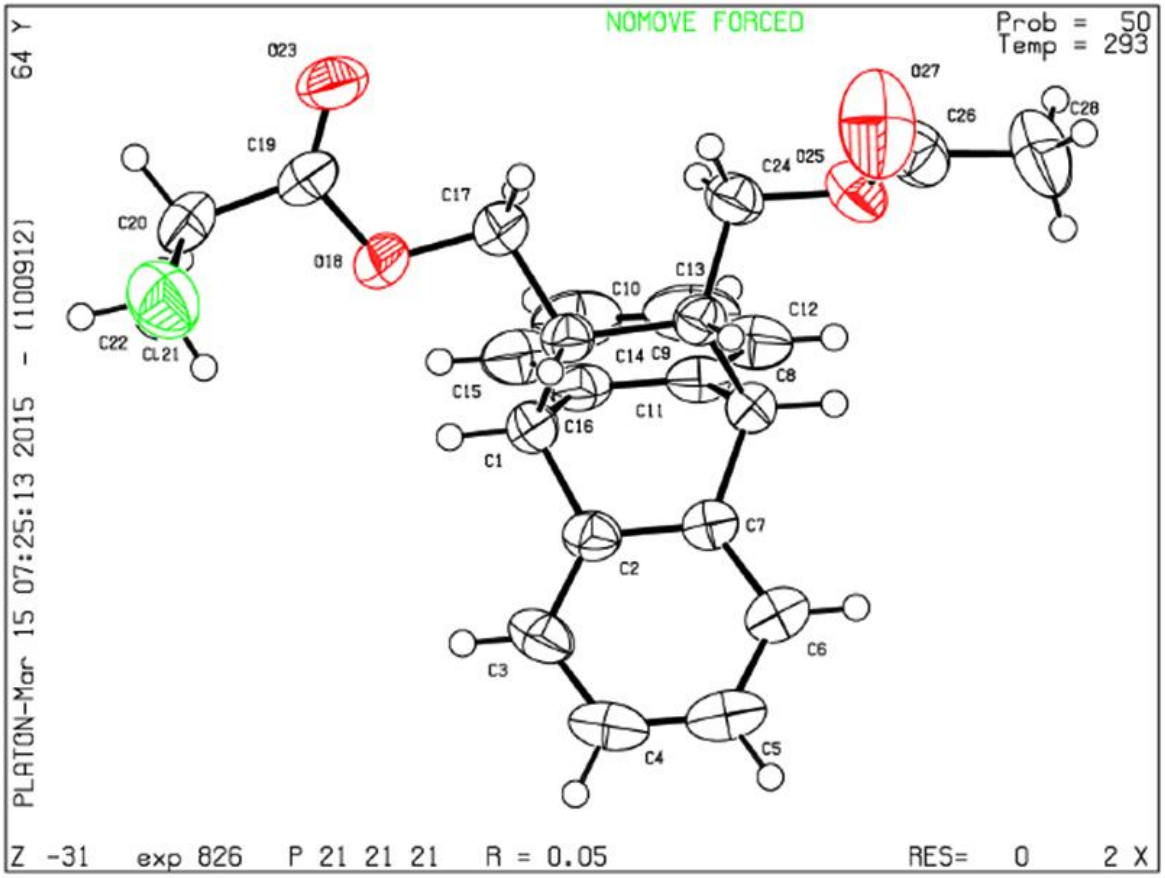


Table 1 Crystal data and structure refinement for exp_826.

Identification code	exp_826
Empirical formula	$C_{23}H_{23}ClO_4$
Formula weight	398.86
Temperature/K	293(2)
Space group	$P2_12_12_1$
a/Å	10.5625(3)
b/Å	12.0642(4)
c/Å	15.9997(5)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	2038.82(11)
Z	4
ρ_{calc}g/cm^3	1.299
μ/mm^{-1}	1.871
F(000)	840.0
Crystal size/mm^3	0.31 × 0.28 × 0.27
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	9.18 to 143.34
Index ranges	$-12 \leq h \leq 12$, $-14 \leq k \leq 13$, $-14 \leq l \leq 19$
Reflections collected	4965
Independent reflections	3323 [R_{int} = 0.0229,
Data/restraints/parameters	3323/0/255
Goodness-of-fit on F^2	1.046
Final R indexes [I>=2σ (I)]	R_1 = 0.0477, wR_2 = 0.1316
Final R indexes [all data]	R_1 = 0.0504, wR_2 = 0.1363
Largest diff. peak/hole / e Å^{-3}	0.24/-0.38
Flack parameter	-0.01(2)

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for exp_826. U_{eq} is defined as 1/3 of of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*Y*	*z*	U(eq)
Cl21	3378.4(11)	8066.1(9)	9648.7(5)	83.4(3)
O25	4639.5(18)	5764.9(18)	4648.8(12)	52.8(5)
O18	2911(2)	7397.5(16)	7771.2(13)	63.2(6)
O23	3586(2)	9137.7(16)	7644.0(13)	63.7(6)
O27	6639(2)	6217(3)	4889(2)	104.6(11)
C19	3034(3)	8446(2)	8015.3(18)	48.7(6)
C2	2544(2)	4074(2)	7222.0(16)	42.9(6)
C7	3100(2)	3612(2)	6511.0(15)	40.5(5)
C9	4085(2)	5399.5(19)	6099.6(15)	38.0(5)
C16	1411(2)	5419(2)	6346.4(18)	45.6(6)
C26	5864(3)	5796(3)	4467(2)	60.9(8)
C24	4232(3)	6278(2)	5420.0(17)	46.9(6)
C11	1927(3)	4933(2)	5634.6(16)	44.6(6)
C8	3214(2)	4425.1(19)	5790.1(14)	39.1(5)
C6	3515(3)	2523(2)	6518.0(18)	49.6(6)
C1	2222(2)	5285(2)	7114.7(16)	43.7(6)
C10	3525(2)	5866.0(19)	6936.6(15)	39.3(5)
C15	269(3)	5984(3)	6297(2)	60.6(8)
C5	3363(3)	1899(2)	7236(2)	59.7(8)
C3	2411(3)	3451(3)	7938.3(18)	53.6(7)
C12	1307(3)	4998(3)	4878.4(19)	58.0(7)
C17	3427(3)	7118(2)	6955.6(18)	50.6(6)
C4	2822(3)	2360(3)	7941(2)	61.9(8)
C20	2379(3)	8633(2)	8845(2)	62.1(8)
C13	147(4)	5562(3)	4838(3)	75.7(11)
C14	-351(3)	6044(3)	5531(3)	73.3(10)
C22	1062(4)	8127(3)	8903(3)	76.8(10)
C28	6116(4)	5215(4)	3655(2)	89.6(13)

Table 3 Anisotropic Displacement Parameters (Å^2×10^3) for exp_826. The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+\ldots]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cl21	94.8(7)	95.7(7)	59.7(4)	3.0(4)	1.1(5)	-1.0(6)
O25	47.2(10)	66.6(12)	44.6(9)	4.8(9)	6.4(8)	-9.1(10)
O18	83.5(15)	37.5(9)	68.5(13)	-12.0(9)	27.1(11)	-13.8(10)
O23	86.2(16)	39.9(9)	65.0(12)	4.2(9)	-4.9(12)	-11.3(11)
O27	44.8(13)	157(3)	112(2)	-42(2)	-0.6(14)	-4.8(16)
C19	57.1(15)	32.6(11)	56.4(14)	-2.8(11)	-8.2(13)	2.8(12)
C2	42.4(12)	40.0(12)	46.2(13)	3.0(11)	0.6(11)	-7.7(11)
C7	41.9(12)	36.2(11)	43.4(12)	2.5(10)	-2.7(10)	-1.3(10)

C9	36.0(11)	37.1(11)	40.9(11)	2.8(10)	0.5(10)	3.2(9)
C16	37.8(12)	34.7(11)	64.3(15)	8.0(11)	5.4(11)	-4.1(10)
C26	48.9(15)	75(2)	58.7(17)	9.3(16)	6.4(14)	5.5(15)
C24	45.5(13)	45.7(13)	49.5(13)	8.1(12)	8.2(12)	-0.5(11)
C11	42.2(12)	37.6(11)	54.2(13)	10.1(11)	-3.5(11)	-7.1(11)
C8	43.0(12)	36.6(11)	37.8(11)	-0.1(9)	1.6(10)	0.7(10)
C6	48.8(14)	39.0(12)	60.8(15)	0.6(12)	-6.8(12)	0.0(11)
C1	43.7(13)	39.3(12)	48.2(13)	-1.0(11)	10.8(11)	-1(1)
C10	39.3(12)	35.5(11)	43.0(12)	1.4(10)	2.7(10)	-1.9(10)
C15	41.8(14)	48.9(15)	91(2)	16.6(15)	5.8(15)	2.1(13)
C5	56.5(17)	40.6(13)	82(2)	16.1(14)	-16.9(16)	-7.4(13)
C3	55.4(15)	60.4(16)	44.9(13)	9.9(13)	2.8(12)	-17.3(14)
C12	59.1(17)	53.3(15)	61.6(16)	16.1(14)	-12.6(14)	-10.0(14)
C17	59.3(16)	39.6(12)	52.9(14)	-3.8(11)	11.0(13)	-4.4(12)
C4	64.9(18)	56.3(16)	64.5(17)	25.8(15)	-8.2(15)	-17.6(15)
C20	81(2)	38.5(13)	66.2(18)	-8.7(13)	6.2(17)	10.9(15)
C13	64.2(19)	69(2)	94(3)	36(2)	-32(2)	-13.6(17)
C14	45.2(16)	58.7(18)	116(3)	28(2)	-9.1(19)	4.3(15)
C22	62.6(19)	86(2)	82(2)	-13(2)	14.7(18)	15.6(19)
C28	82(3)	122(3)	64.8(19)	-10(2)	29.1(19)	-1(3)

Table 4 Bond Lengths for exp_826.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Cl21	C20	1.799(4)	C16	C15	1.388(4)
O25	C26	1.326(4)	C16	C11	1.392(4)
O25	C24	1.446(3)	C16	C1	1.506(4)
O18	C19	1.330(3)	C26	C28	1.501(5)
O18	C17	1.454(3)	C11	C12	1.378(4)
O23	C19	1.179(3)	C11	C8	1.511(4)
O27	C26	1.177(4)	C6	C5	1.383(4)
C19	C20	1.515(4)	C1	C10	1.571(3)
C2	C3	1.378(4)	C10	C17	1.514(3)
C2	C7	1.396(4)	C15	C14	1.392(5)
C2	C1	1.510(3)	C5	C4	1.381(5)
C7	C6	1.385(4)	C3	C4	1.385(4)
C7	C8	1.519(3)	C12	C13	1.403(5)
C9	C24	1.526(3)	C20	C22	1.521(5)
C9	C10	1.569(3)	C13	C14	1.358(6)
C9	C8	1.573(3)			

Table 5 Bond Angles for exp_826.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C26	O25	C24	117.7(2)	C16	C11	C8	112.8(2)
C19	O18	C17	116.6(2)	C11	C8	C7	108.4(2)
O23	C19	O18	125.0(3)	C11	C8	C9	105.95(19)
O23	C19	C20	124.2(2)	C7	C8	C9	106.84(19)
O18	C19	C20	110.8(2)	C5	C6	C7	119.1(3)
C3	C2	C7	120.2(3)	C16	C1	C2	108.9(2)
C3	C2	C1	126.8(3)	C16	C1	C10	107.6(2)
C7	C2	C1	112.9(2)	C2	C1	C10	104.8(2)
C6	C7	C2	120.3(2)	C17	C10	C9	113.6(2)
C6	C7	C8	126.4(2)	C17	C10	C1	112.4(2)
C2	C7	C8	113.2(2)	C9	C10	C1	109.0(19)
C24	C9	C10	113.4(2)	C16	C15	C14	119.0(3)
C24	C9	C8	110.7(2)	C4	C5	C6	120.5(3)
C10	C9	C8	108.43(19)	C2	C3	C4	119.3(3)
C15	C16	C11	120.0(3)	C11	C12	C13	118.9(3)
C15	C16	C1	126.4(3)	O18	C17	C10	106.0(2)
C11	C16	C1	113.6(2)	C5	C4	C3	120.6(3)
O27	C26	O25	124.4(3)	C19	C20	C22	114.3(3)
O27	C26	C28	125.1(3)	C19	C20	Cl21	107.6(2)
O25	C26	C28	110.5(3)	C22	C20	Cl21	109.9(2)
O25	C24	C9	110.0(2)	C14	C13	C12	120.6(3)
C12	C11	C16	120.6(3)	C13	C14	C15	120.9(3)
C12	C11	C8	126.5(3)				

Table 6 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3) for exp_826.

Atom	*x*	*y*	*z*	U(eq)
H9	4925	5093	6220	46
H24A	3430	6652	5333	56
H24B	4851	6825	5594	56
H8	3553	4070	5287	47
H6	3891	2216	6046	59
H1	1809	5590	7613	52
H10	4104	5649	7386	47
H15	-78	6316	6769	73
H5	3626	1164	7245	72
H3	2049	3759	8415	64
H12	1651	4673	4403	70
H17A	4254	7453	6881	61
H17B	2872	7379	6515	61
H4	2732	1935	8422	74
H20	2305	9434	8937	74

H13	-284	5605	4333	91
H14	-1117	6421	5493	88
H22A	1126	7334	8883	115
H22B	557	8382	8443	115
H22C	673	8347	9419	115
H28A	5775	4478	3677	134
H28B	7013	5178	3560	134
H28C	5722	5618	3207	134

Single crystals of $C_{23}H_{23}ClO_4$. A suitable crystal was selected and on a diffractometer. The crystal was kept at 293(2) K during data collection. Using Olex2 the structure was solved with the Superflip structure solution program using Charge Flipping and refined with the ShelXL refinement package using Least Squares minimisation.

Crystal Data for $C_{23}H_{23}ClO_4$ (M =398.86 g/mol): , space group $P2_12_12_1$ (no. 19), a = 10.5625(3) Å, b = 12.0642(4) Å, c = 15.9997(5) Å, V = 2038.82(11) Å^3, Z = 4, T = 293(2) K, μ(CuKα) = 1.871 mm^{-1}, *Dcalc* = 1.299 g/cm^3, 4965 reflections measured (9.18° ≤ 2Θ ≤ 143.34°), 3323 unique (R_{int} = 0.0229, which were used in all calculations. The final R_1 was 0.0477 (>2sigma(I)) and wR_2 was 0.1363 (all data).