Supporting Information

Osmium(0) Catalyzed C–C Coupling of Ethylene and α-Olefins with Diols, Ketols or Hydroxy Esters via Transfer Hydrogenation

Boyoung Y. Park[†], Tom Luong[†], Hiroki Sato and Michael J. Krische*

University of Texas at Austin, Department of Chemistry,
Austin, TX 78712, USA

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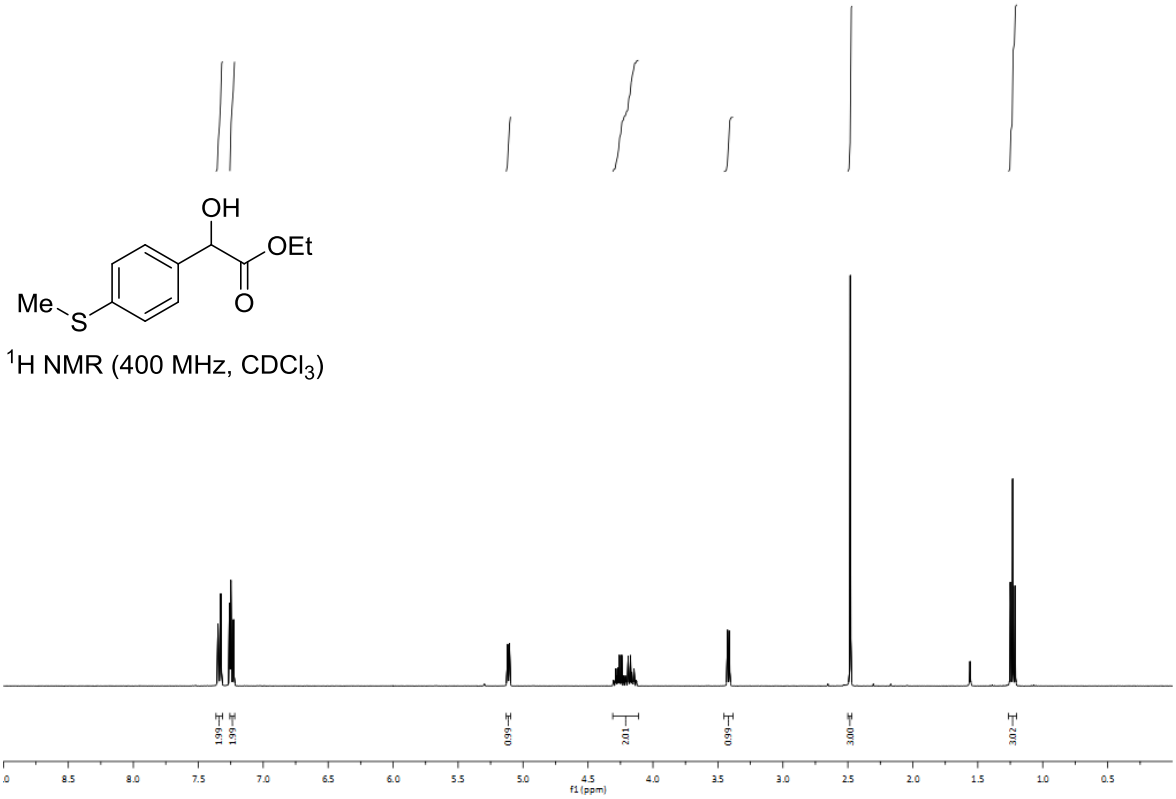
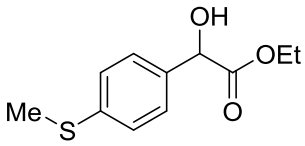
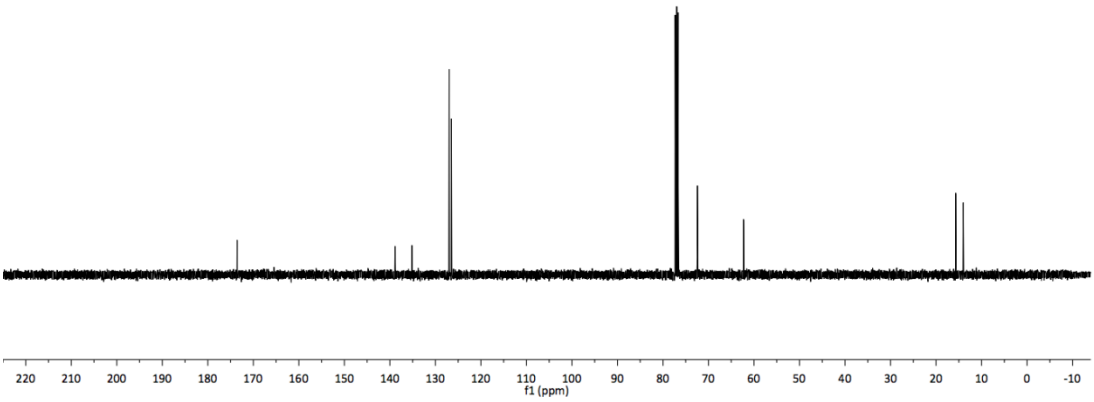
Ethyl 2-hydroxy-2-(4-(methylthio)phenyl)acetate (1g).



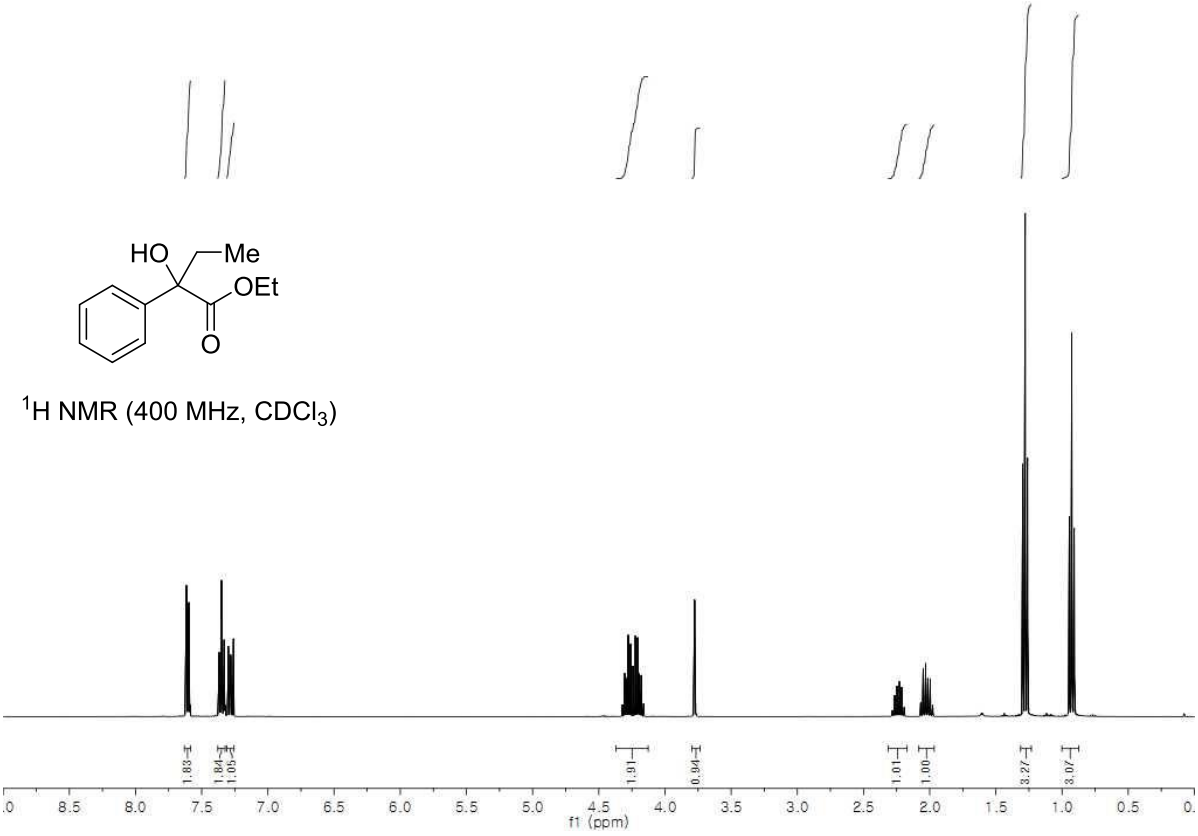
^{1}H NMR (400 MHz, $CDCl_3$)



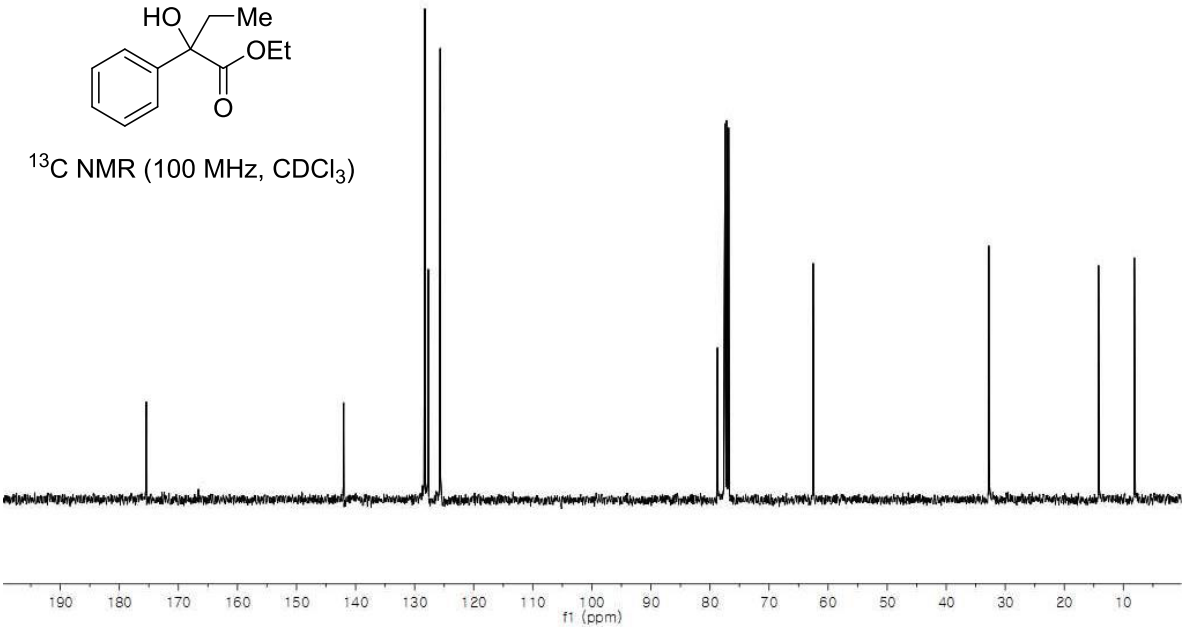
^{13}C NMR (100 MHz, $CDCl_3$)



Ethyl 2-hydroxy-2-phenylbutanoate (3a).

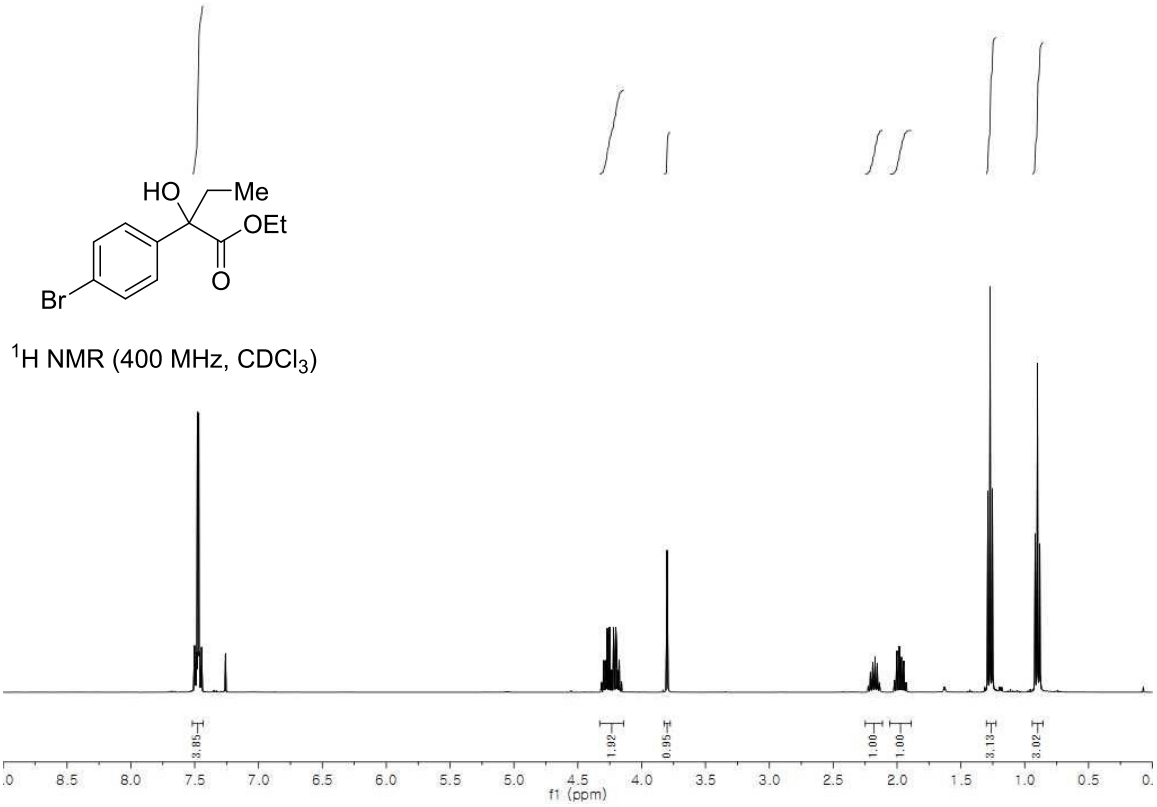


^{1}H NMR (400 MHz, $CDCl_3$)

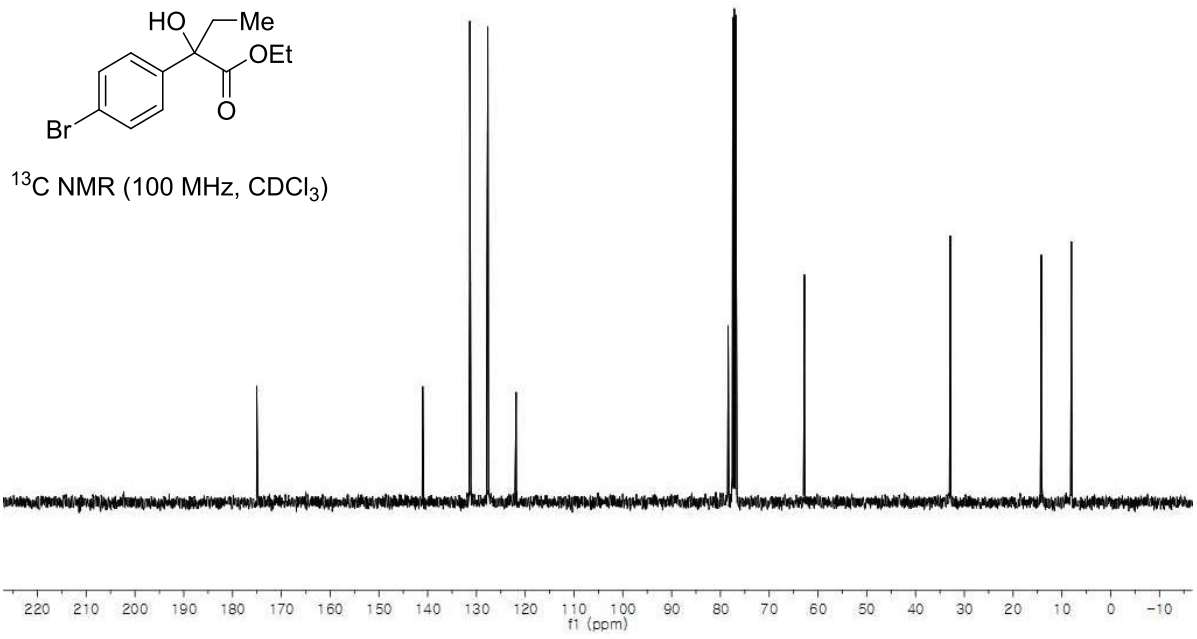


^{13}C NMR (100 MHz, $CDCl_3$)

Ethyl 2-(4-bromophenyl)-2-hydroxybutanoate (3b).

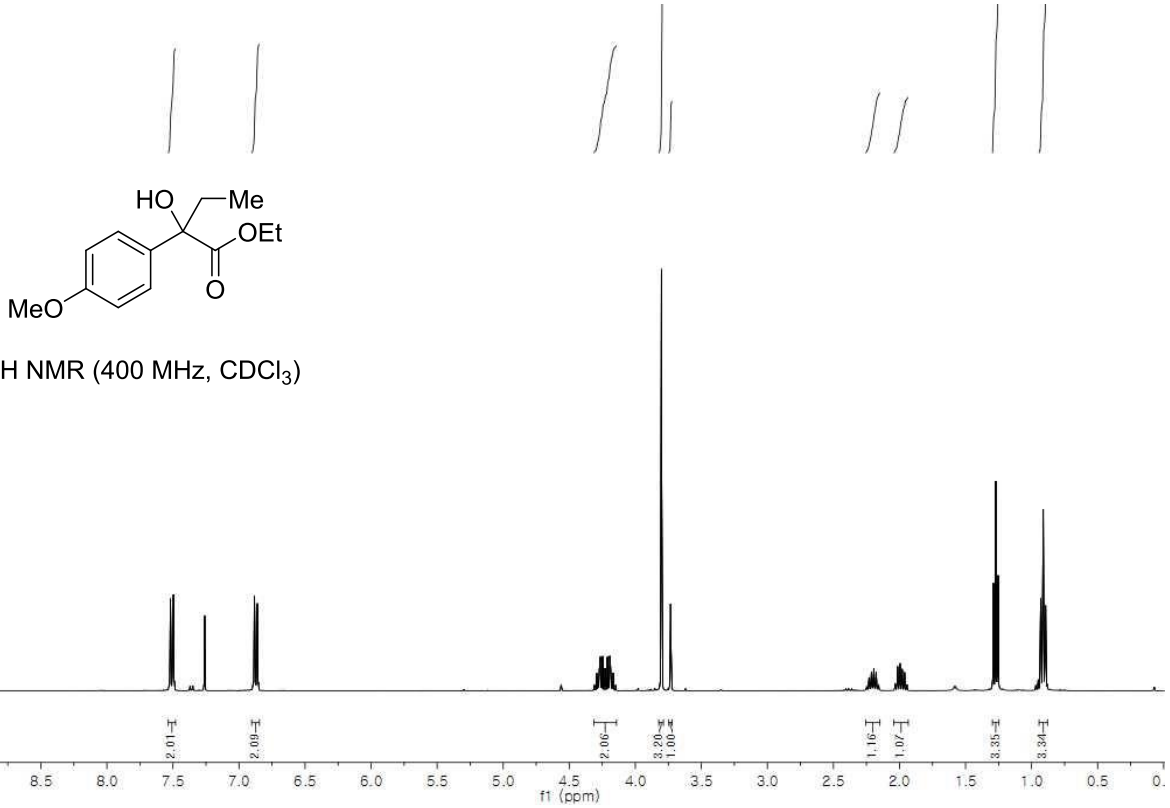


^{1}H NMR (400 MHz, $CDCl_3$)

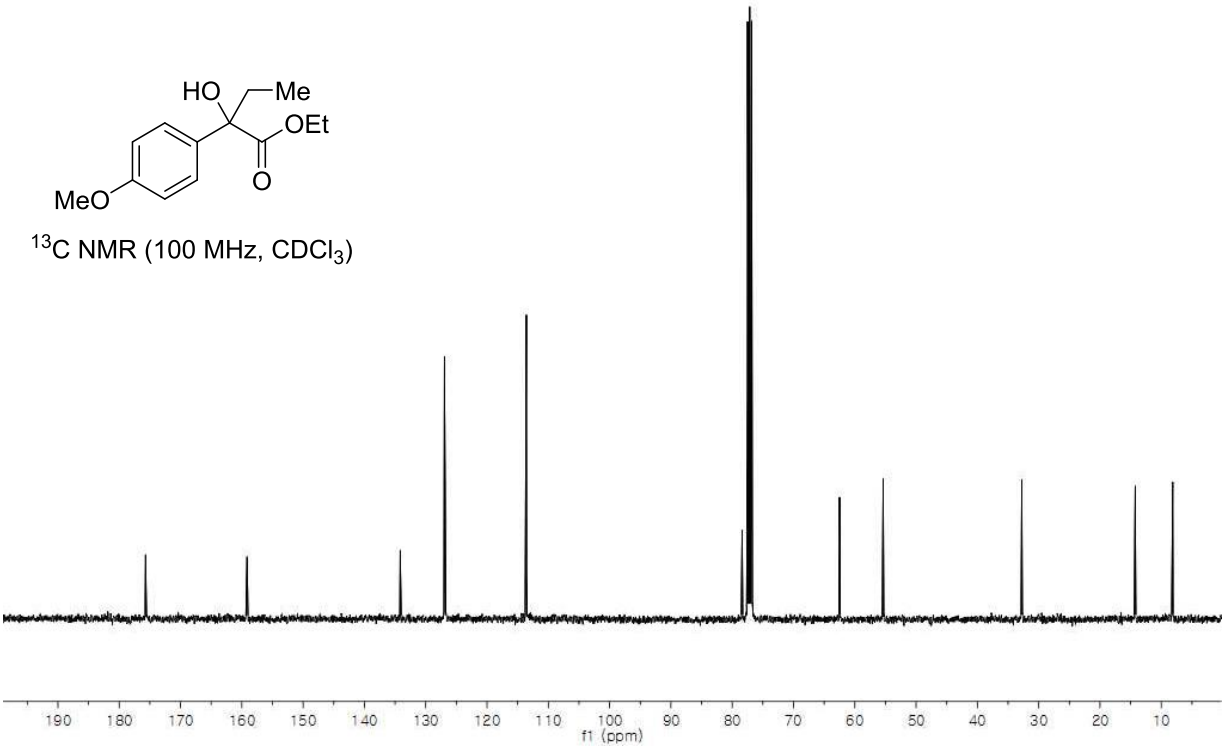


^{13}C NMR (100 MHz, $CDCl_3$)

Ethyl 2-hydroxy-2-(4-methoxyphenyl)butanoate (3c).

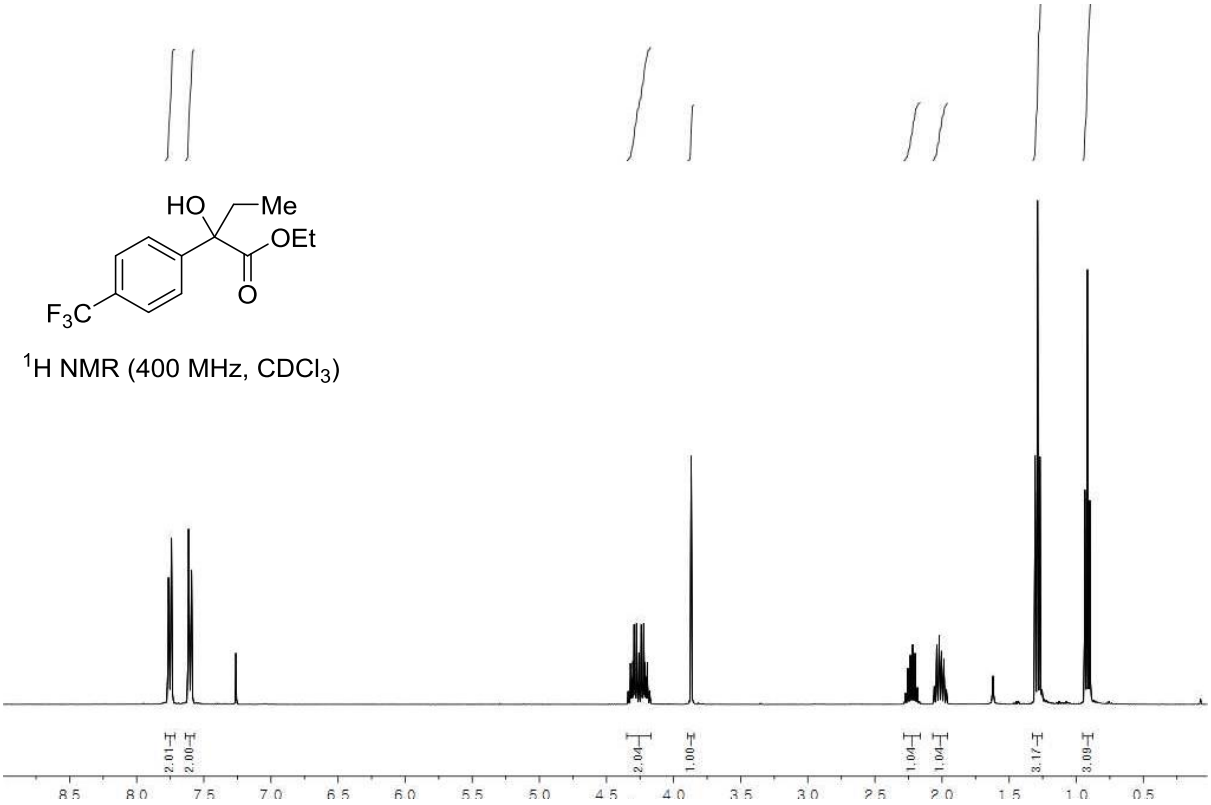


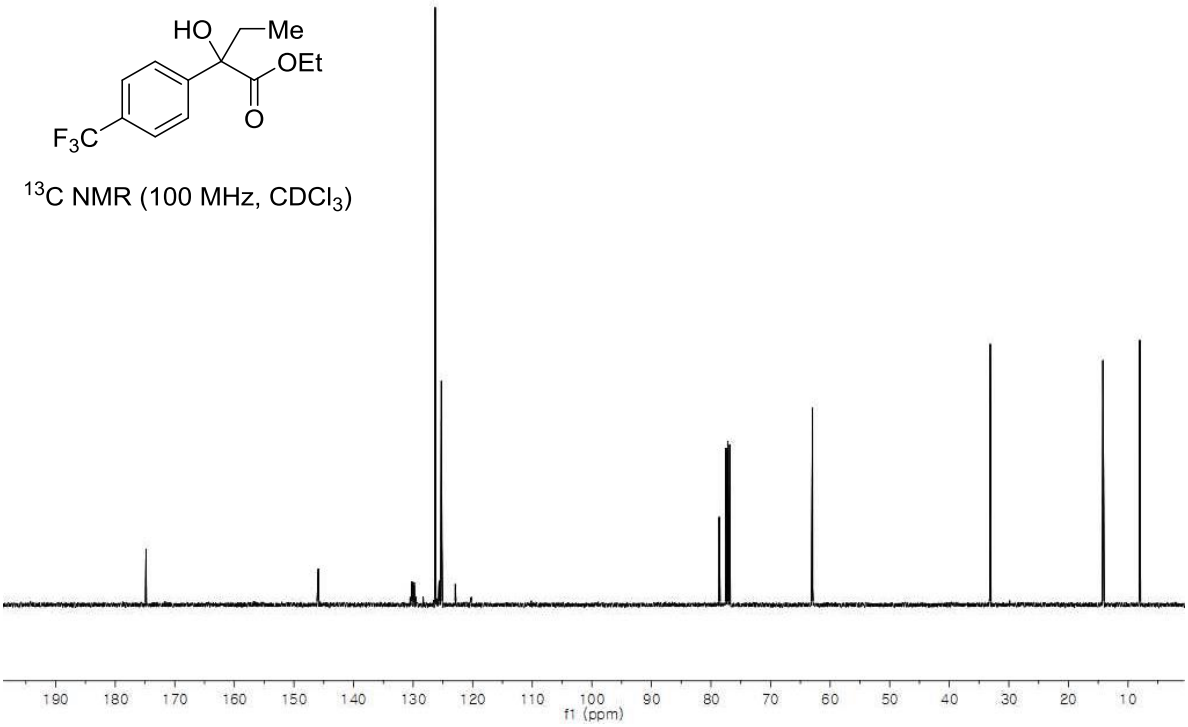
^{1}H NMR (400 MHz, $CDCl_3$)

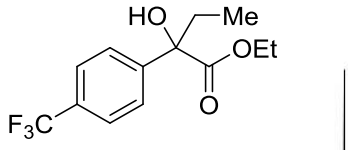


^{13}C NMR (100 MHz, $CDCl_3$)

Ethyl 2-hydroxy-2-(4-(trifluoromethyl)phenyl)butanoate (3d).

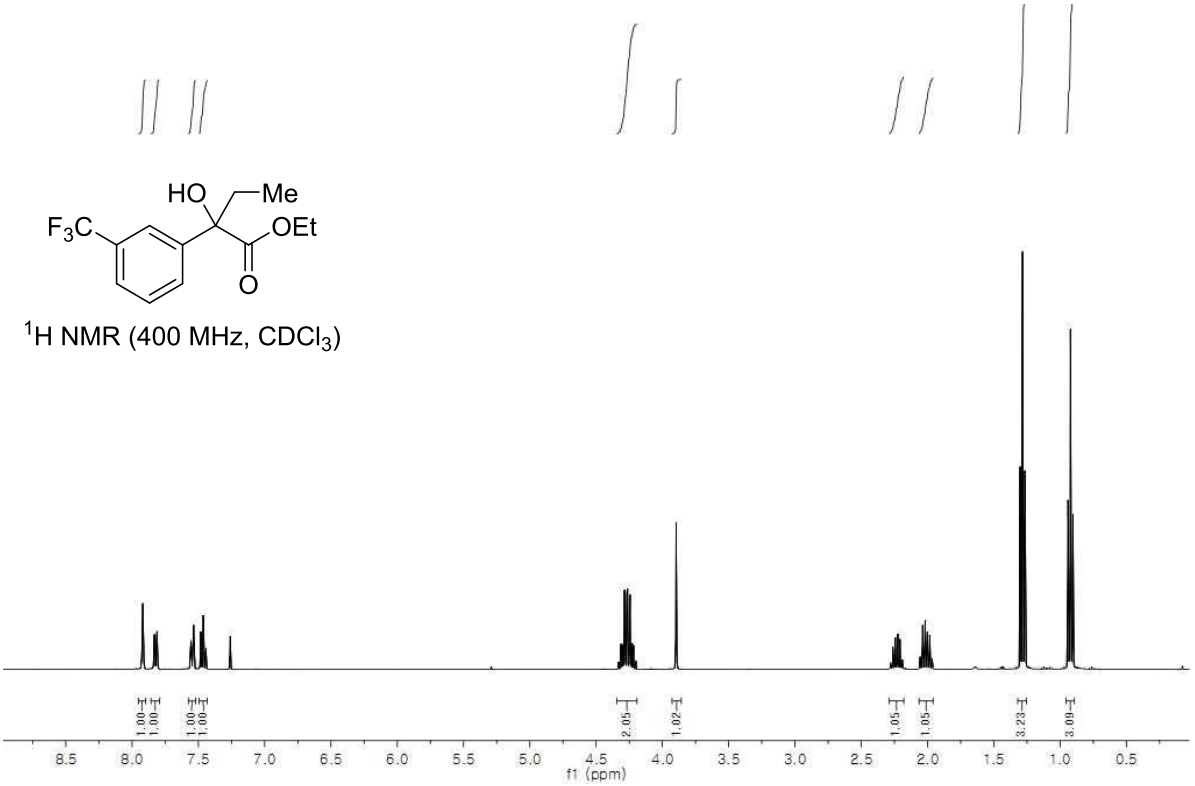


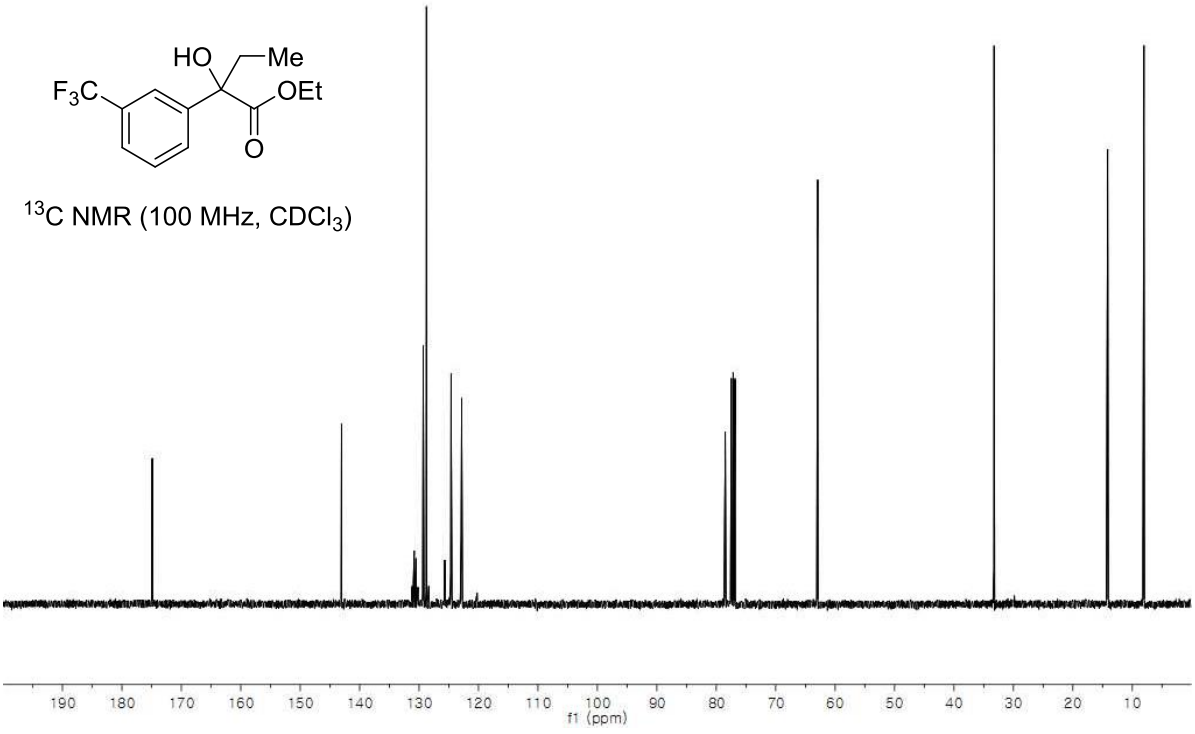


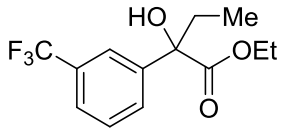


^{19}F NMR (376 MHz, $CDCl_3$)

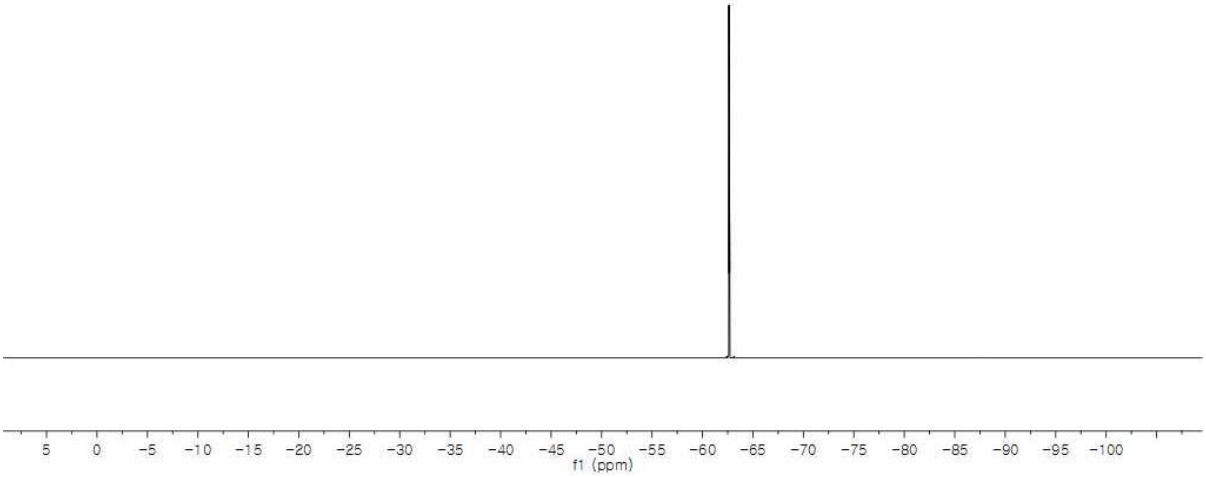
Ethyl 2-hydroxy-2-(3-(trifluoromethyl)phenyl)butanoate (3e).







^{19}F NMR (376 MHz, $CDCl_3$)



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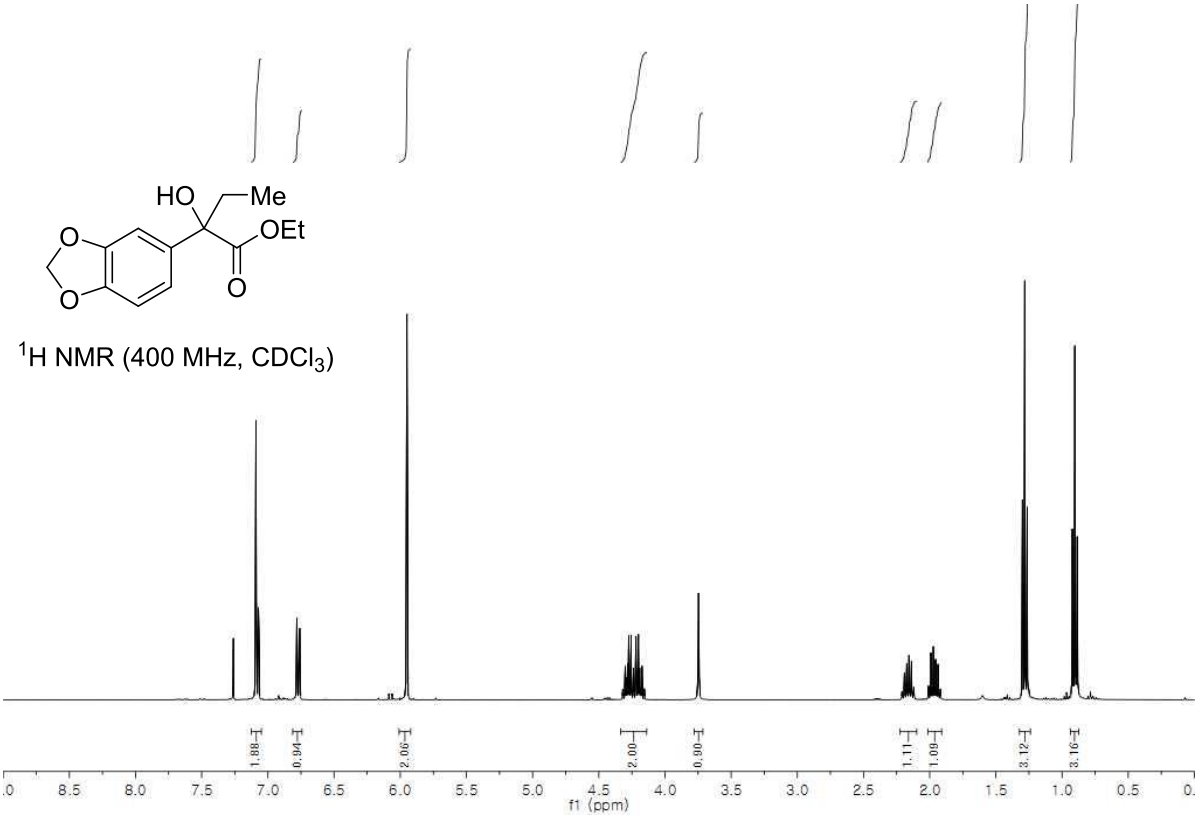
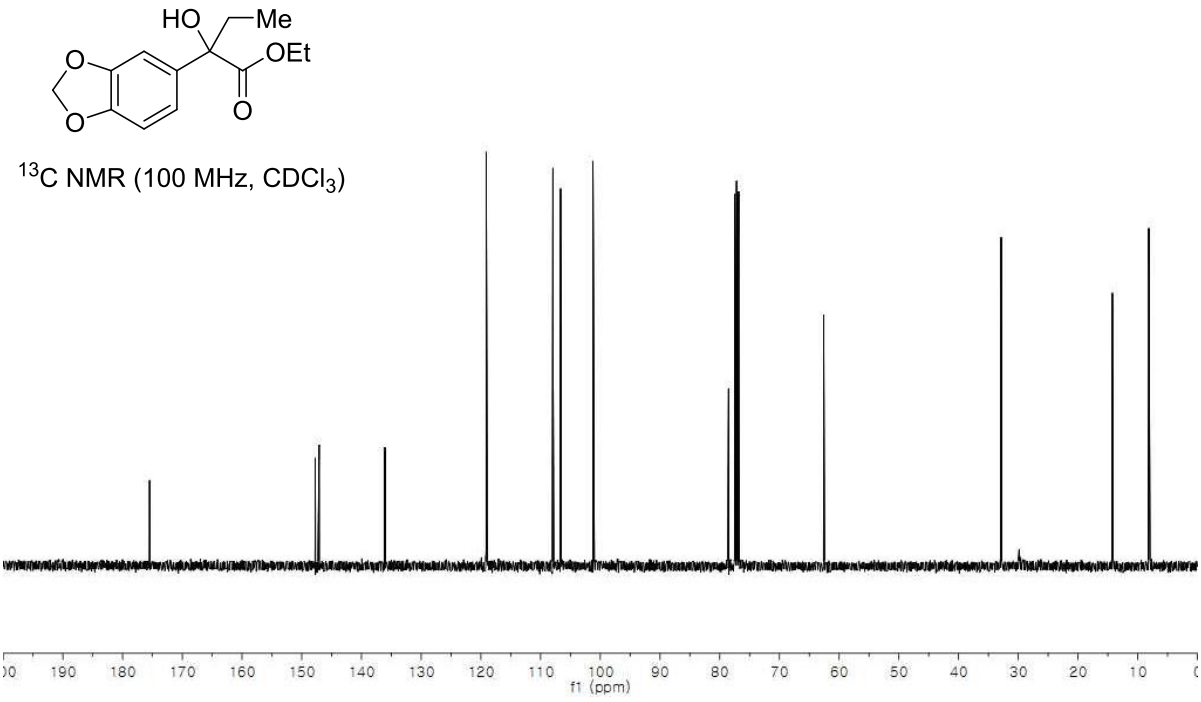


^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)

Ethyl 2-hydroxy-2-(4-(methylthio)phenyl)butanoate (3g).

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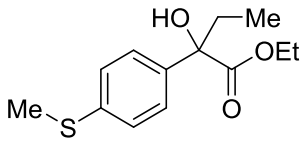
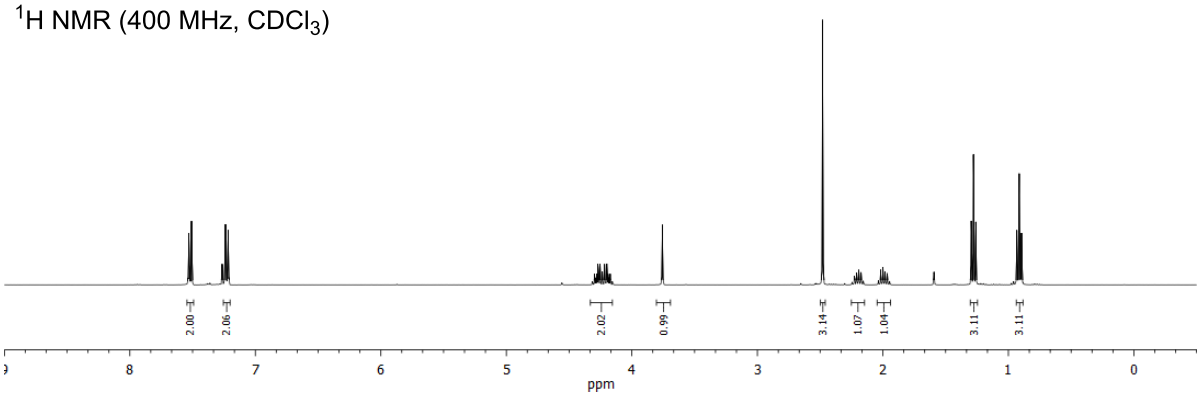
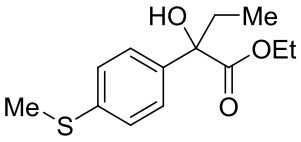
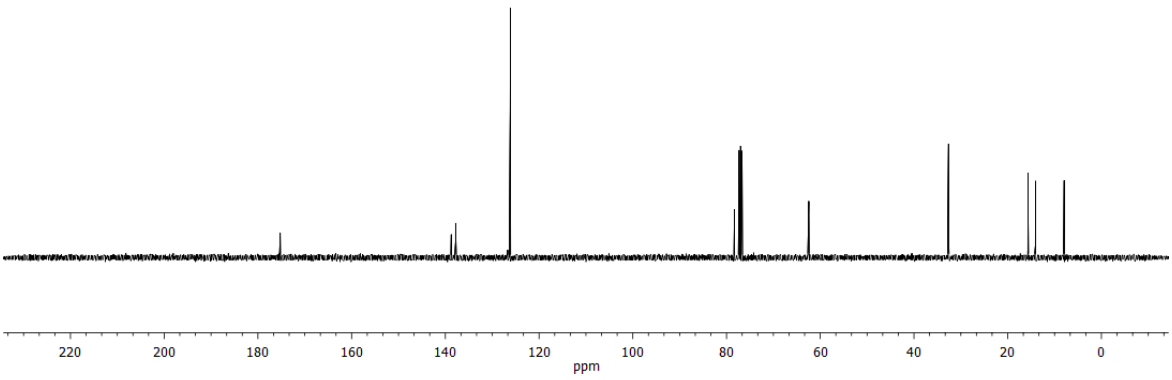


^{1}H NMR (400 MHz, $CDCl_3$)

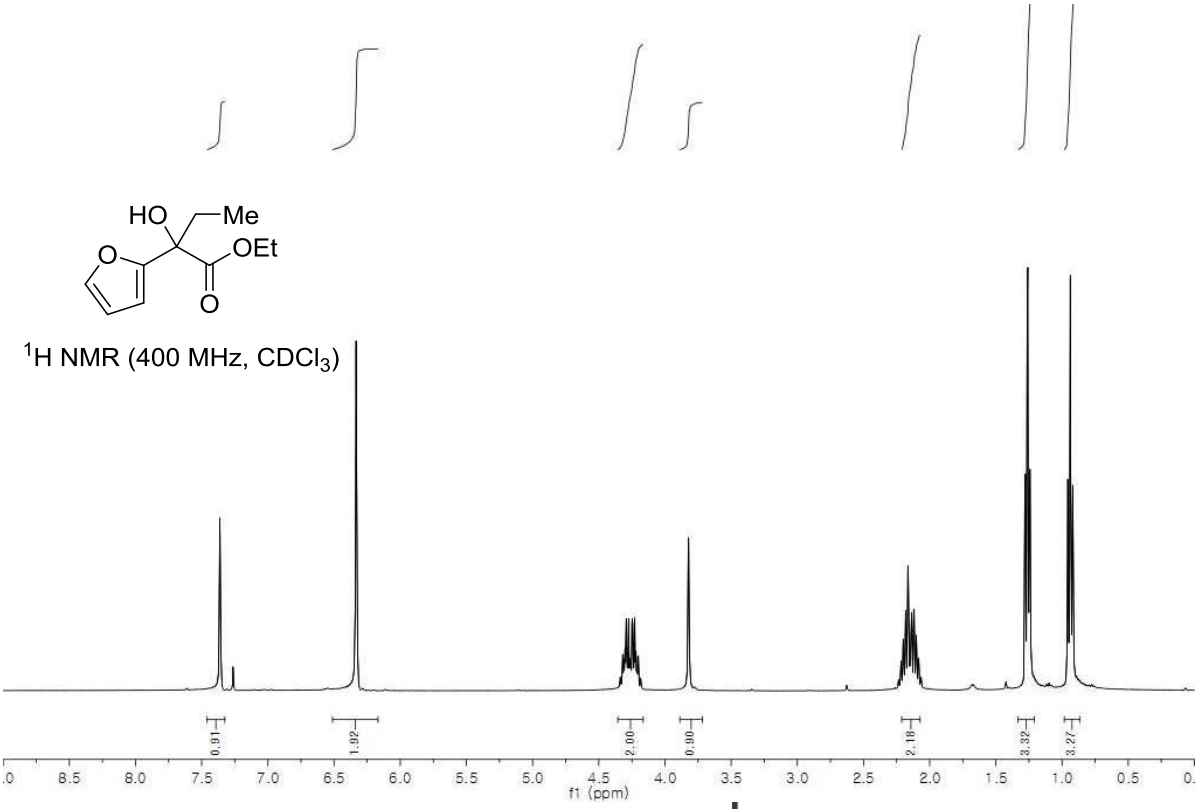


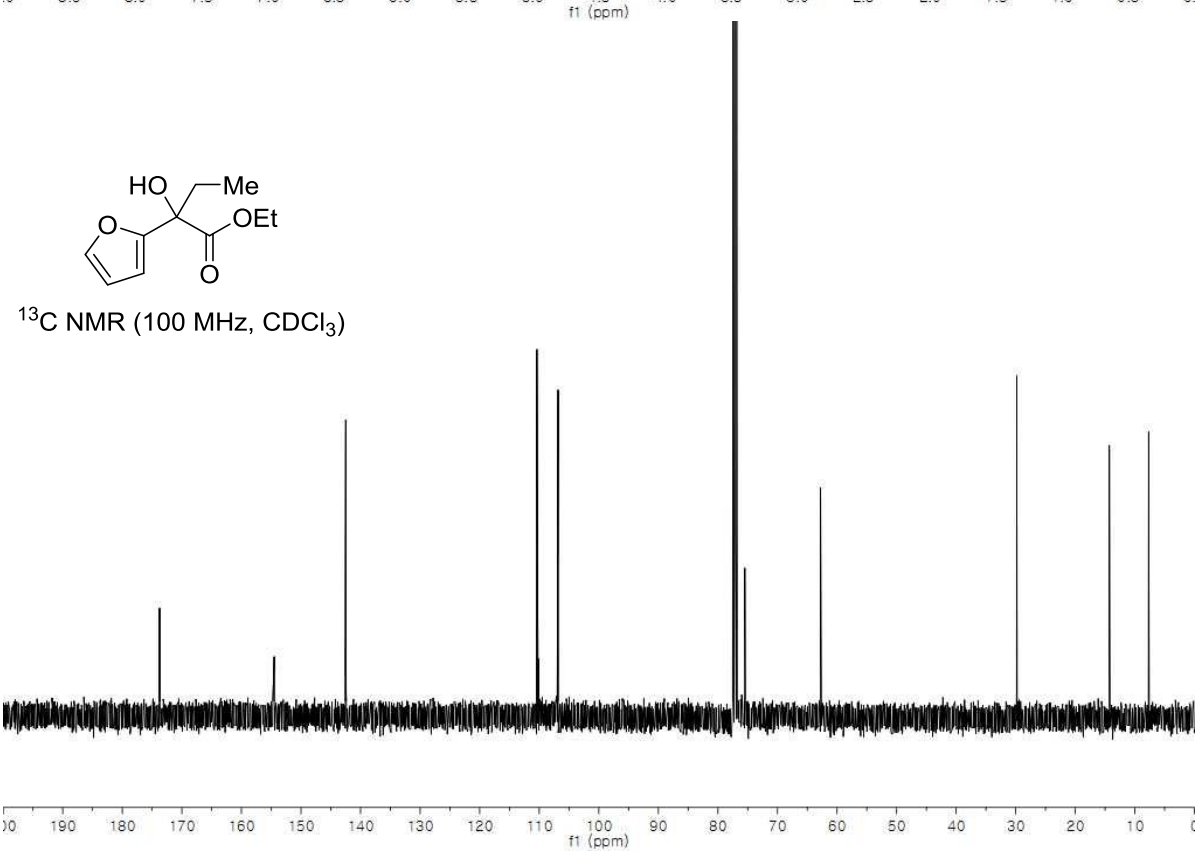


^{13}C NMR (100 MHz, $CDCl_3$)

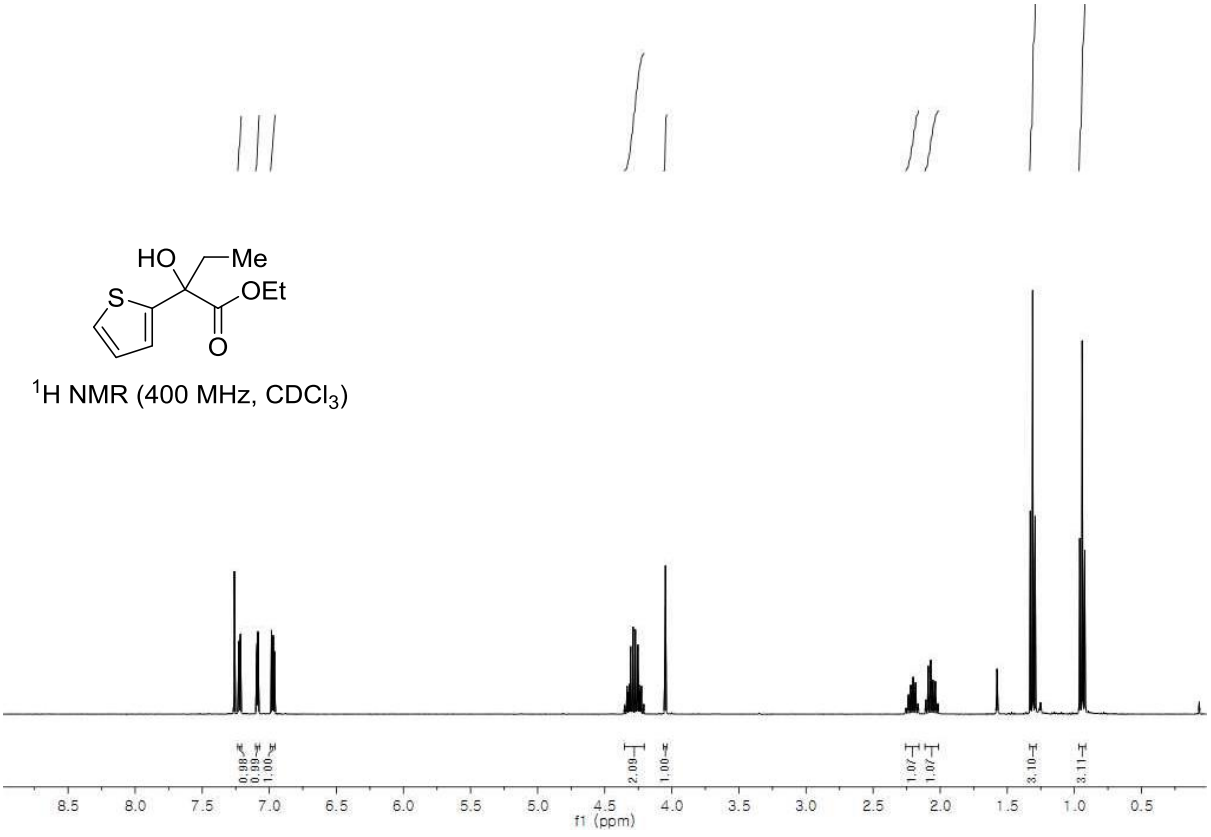


Ethyl 2-(furan-2-yl)-2-hydroxybutanoate (3h).

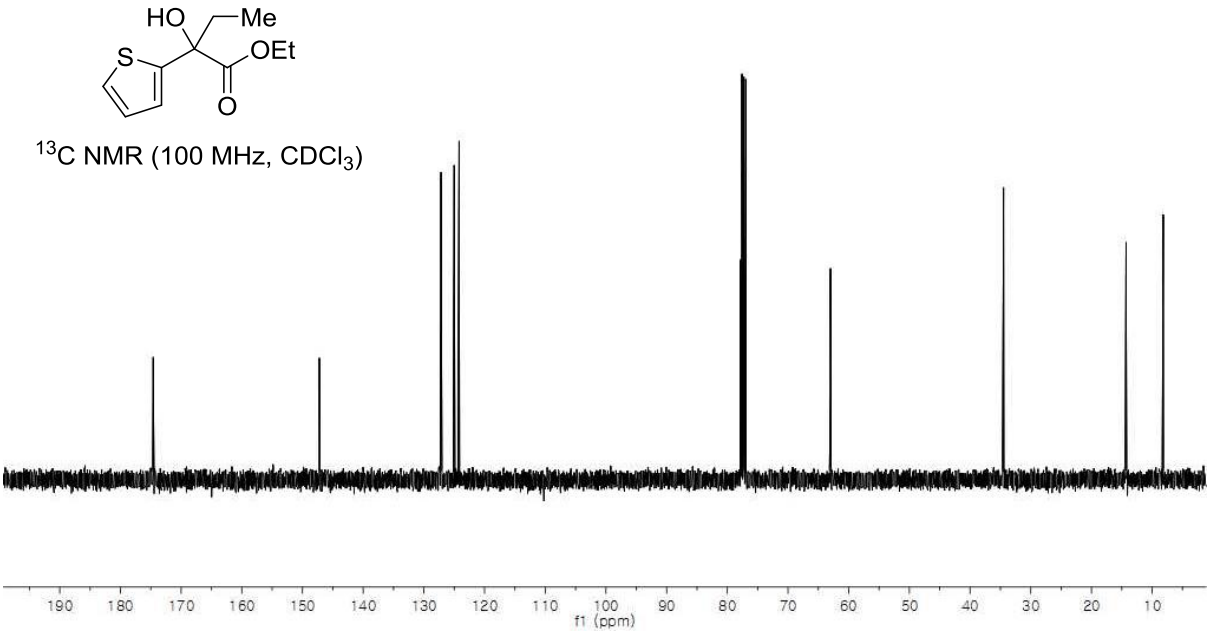




Ethyl 2-hydroxy-2-(thiophen-2-yl)butanoate (3i).

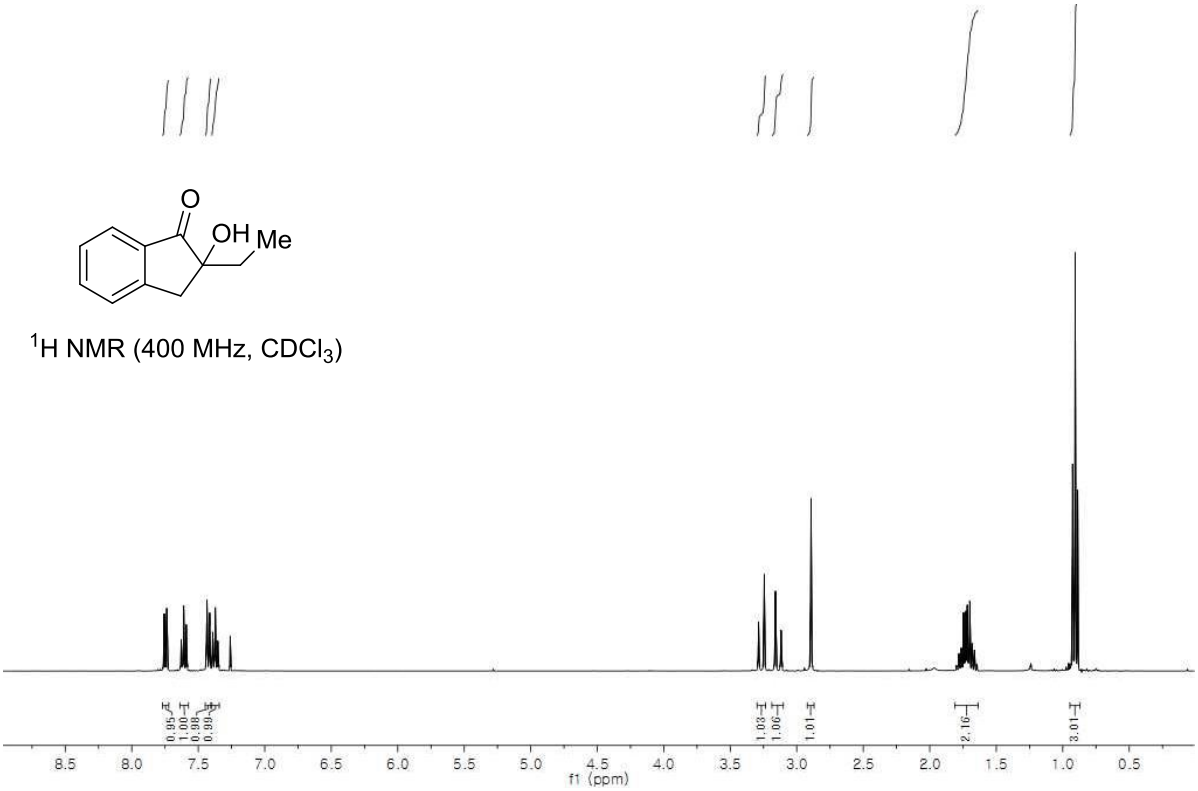


^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)

2-Ethyl-2-hydroxy-2,3-dihydro-1H-inden-1-one (3j).



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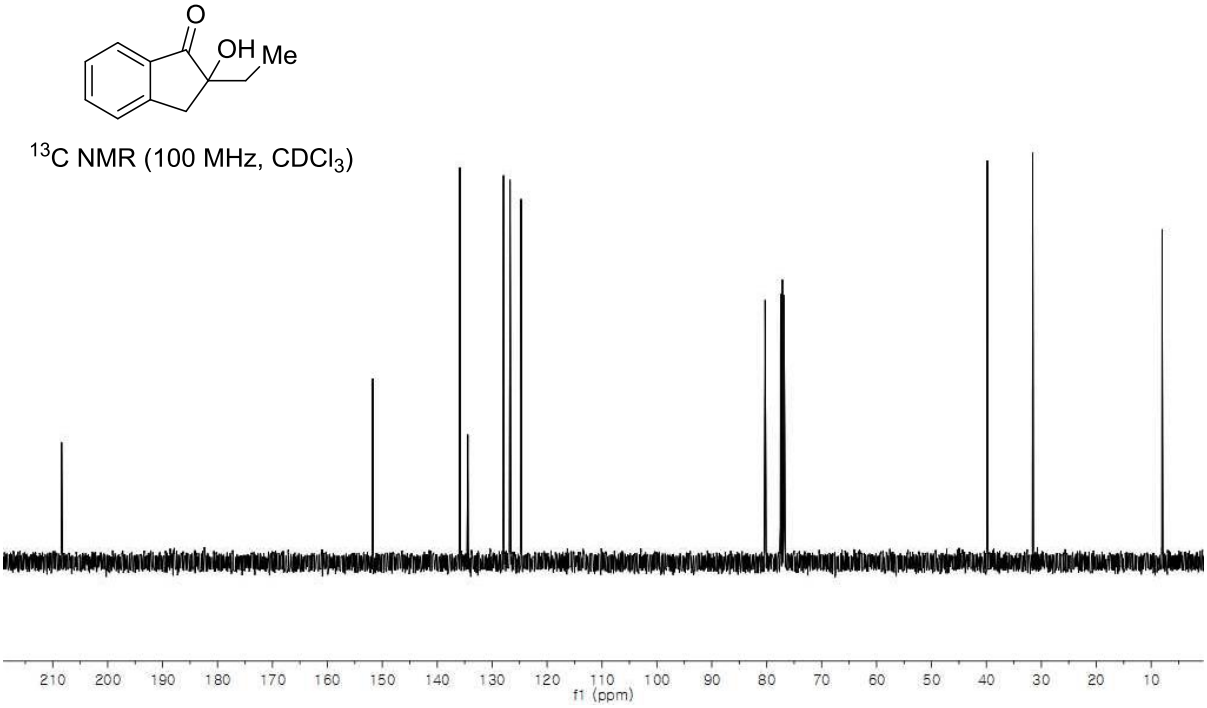
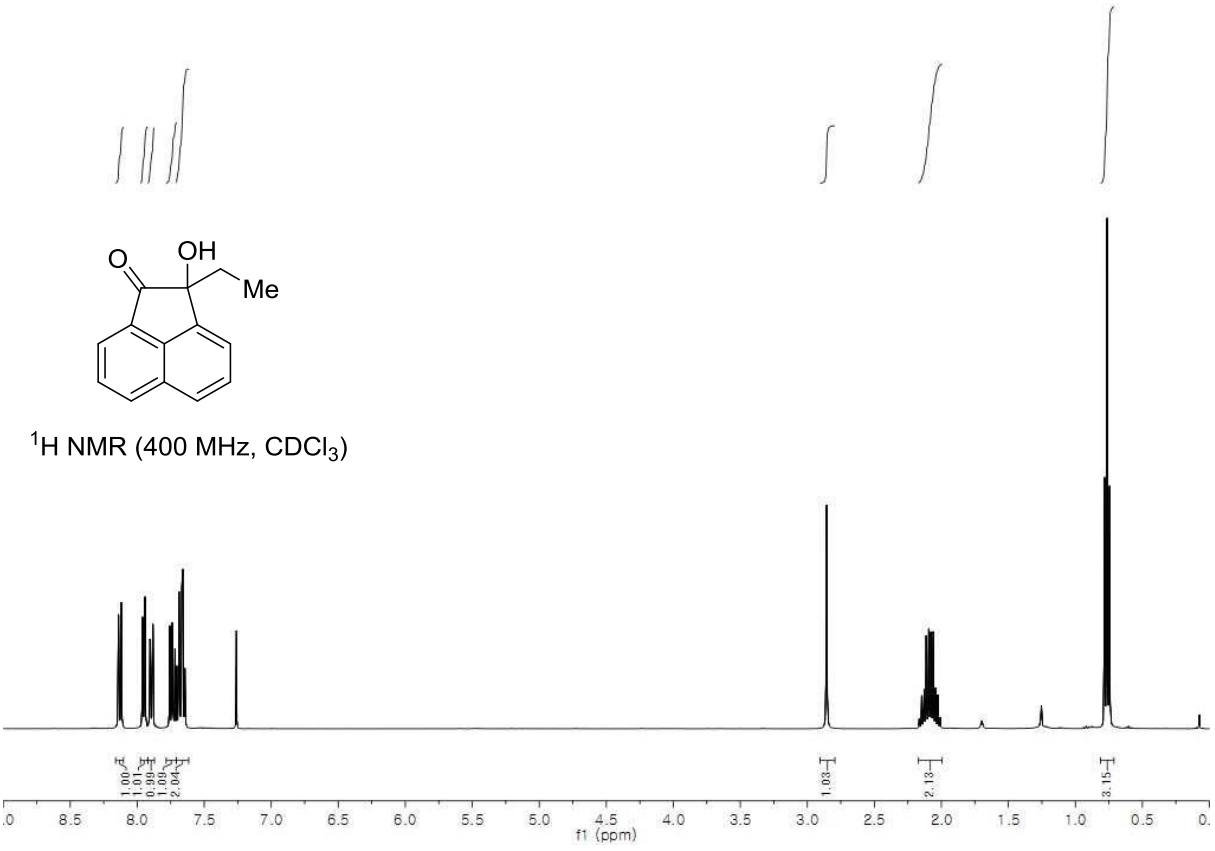
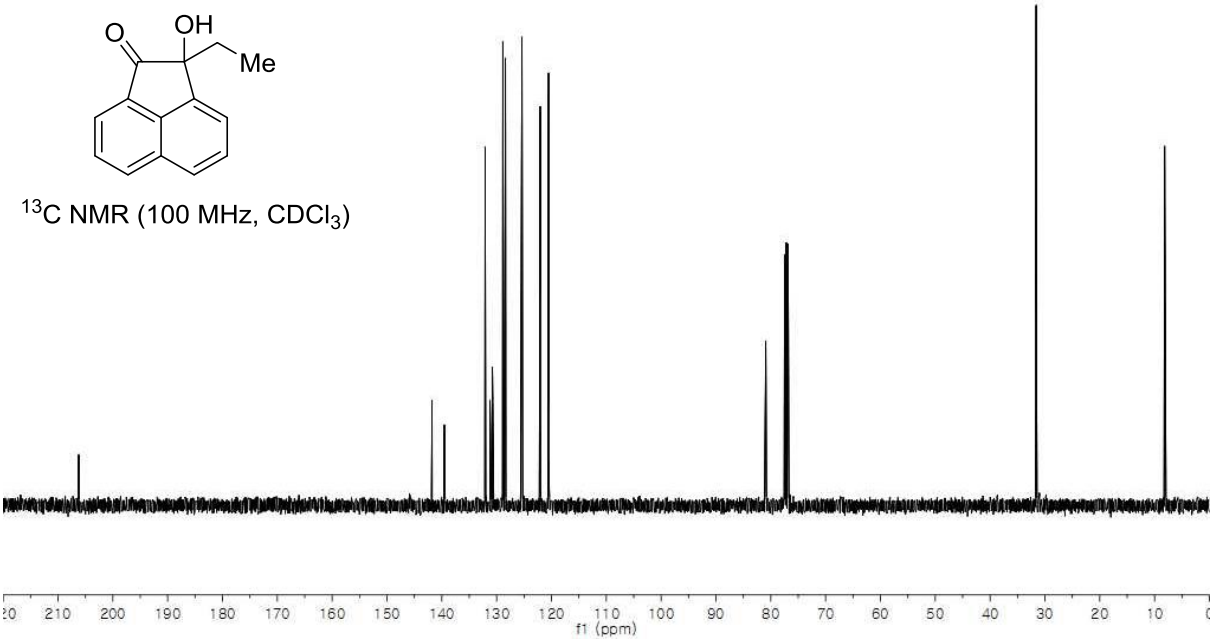


^{13}C NMR (100 MHz, $CDCl_3$)

2-Ethyl-2-hydroxyacenaphthylen-1(2H)-one (3k).

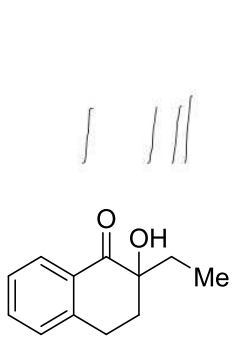


^{1}H NMR (400 MHz, $CDCl_3$)

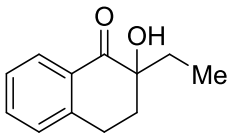


^{13}C NMR (100 MHz, $CDCl_3$)

2-Ethyl-2-hydroxy-3,4-dihydronaphthalen-1(2H)-one (3l).

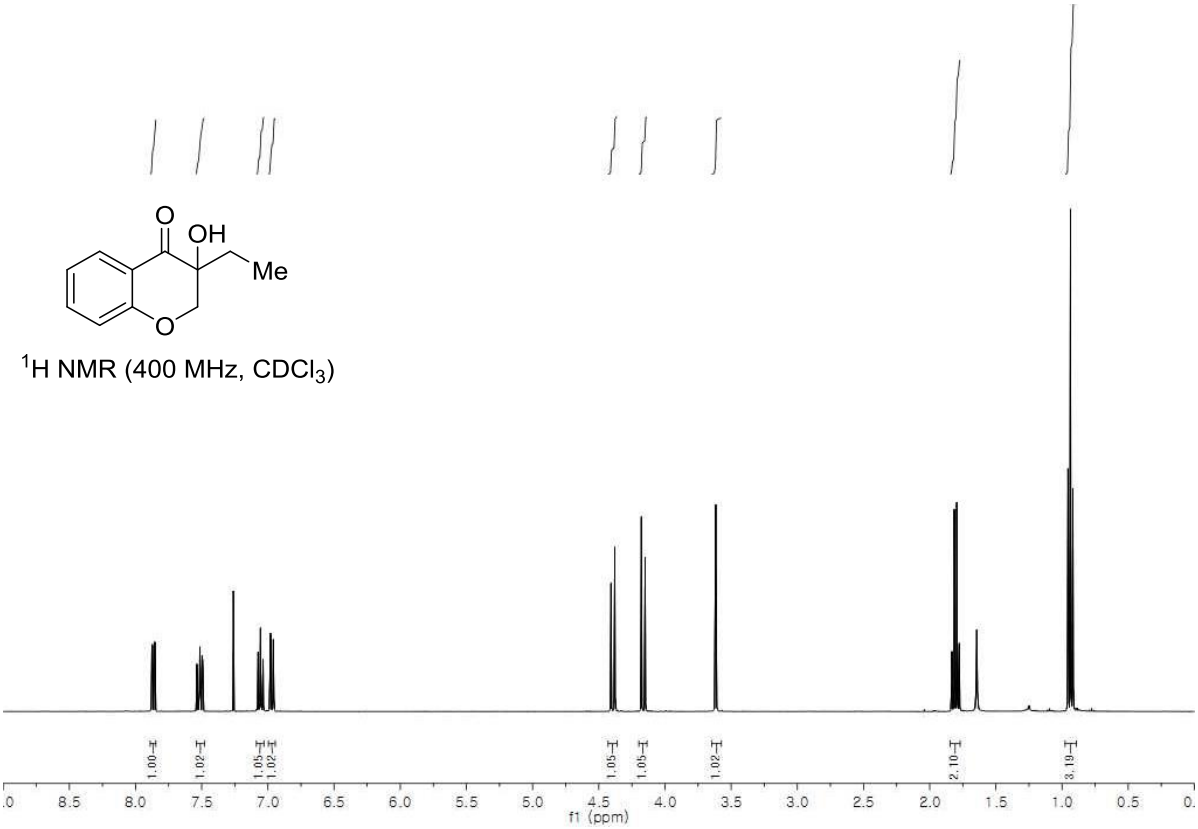


^{1}H NMR (400 MHz, $CDCl_3$)

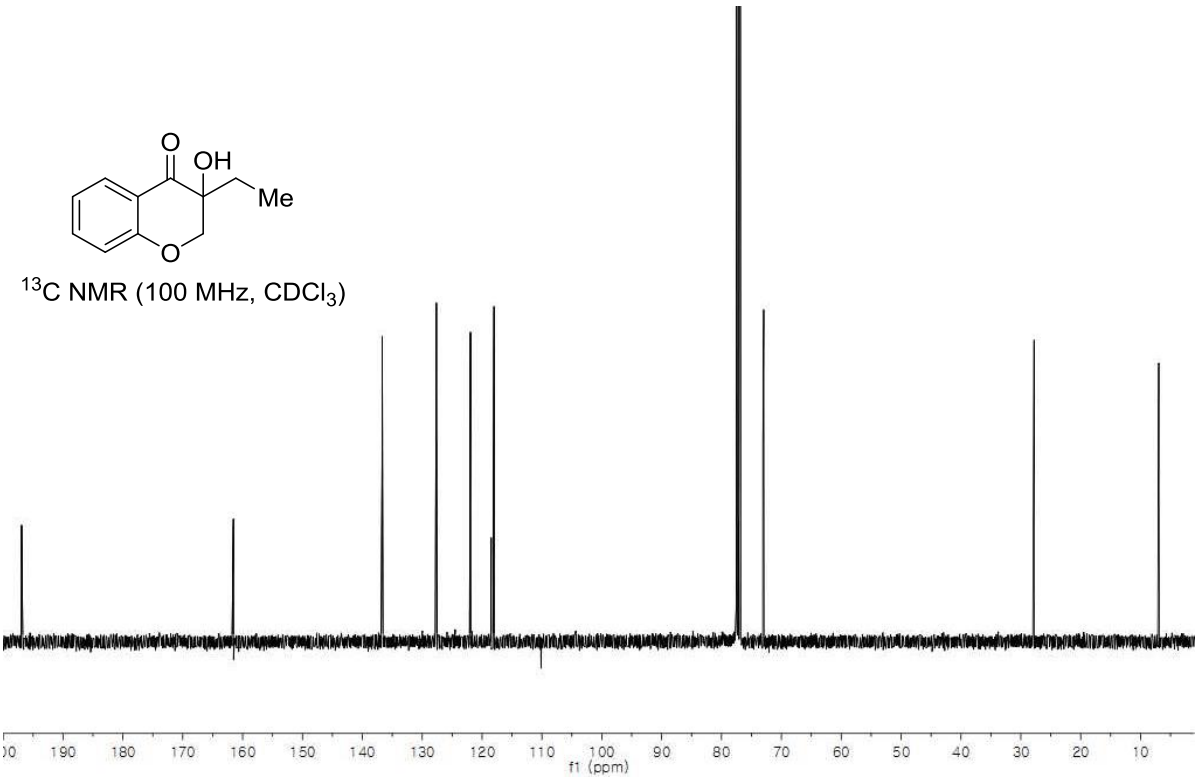


^{13}C NMR (100 MHz, $CDCl_3$)

3-Ethyl-3-hydroxychroman-4-one (3m).

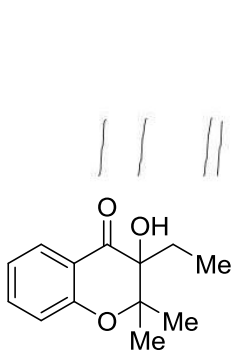


^{1}H NMR (400 MHz, $CDCl_3$)



^{13}C NMR (100 MHz, $CDCl_3$)

3-Ethyl-3-hydroxy-2,2-dimethylchroman-4-one (3n).



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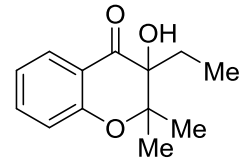

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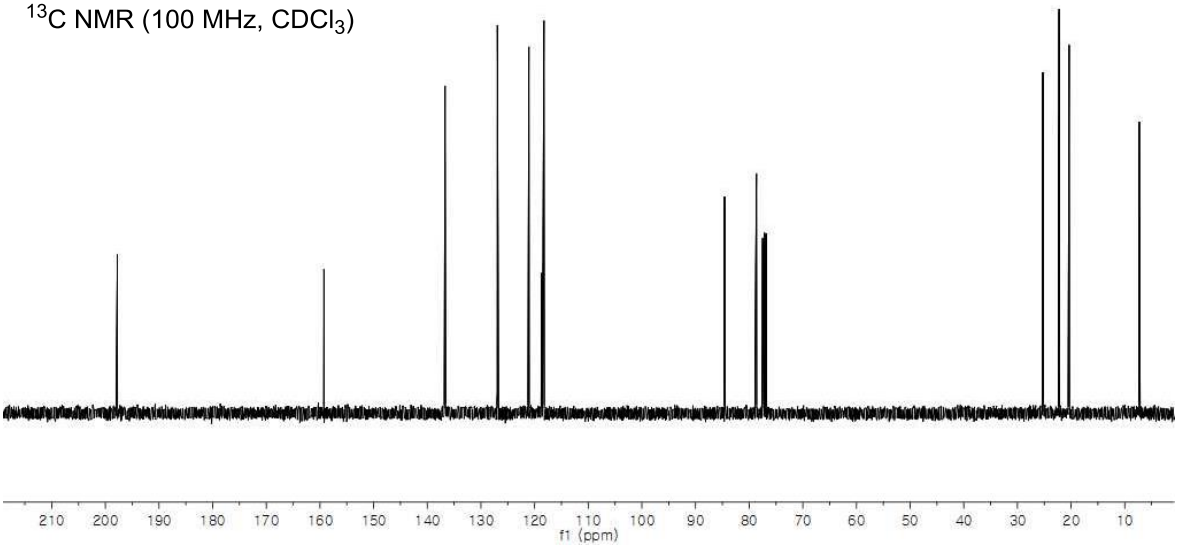
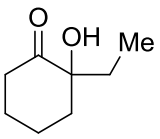
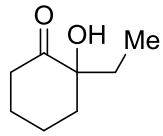


2-Ethyl-2-hydroxycyclohexan-1-one (3o).

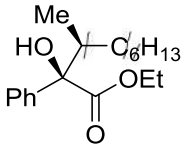


^{1}H NMR (400 MHz, $CDCl_3$)

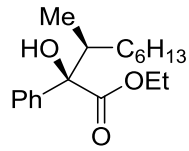


^{13}C NMR (100 MHz, $CDCl_3$)

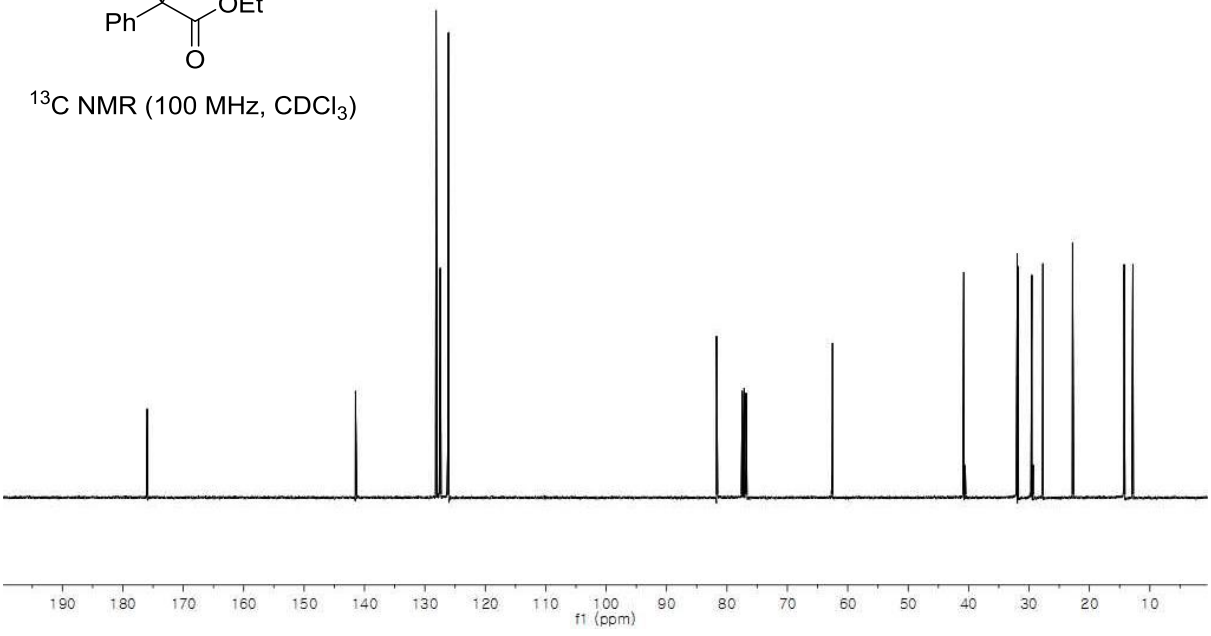
Ethyl 2-hydroxy-3-methyl-2-phenylnonanoate (4a).



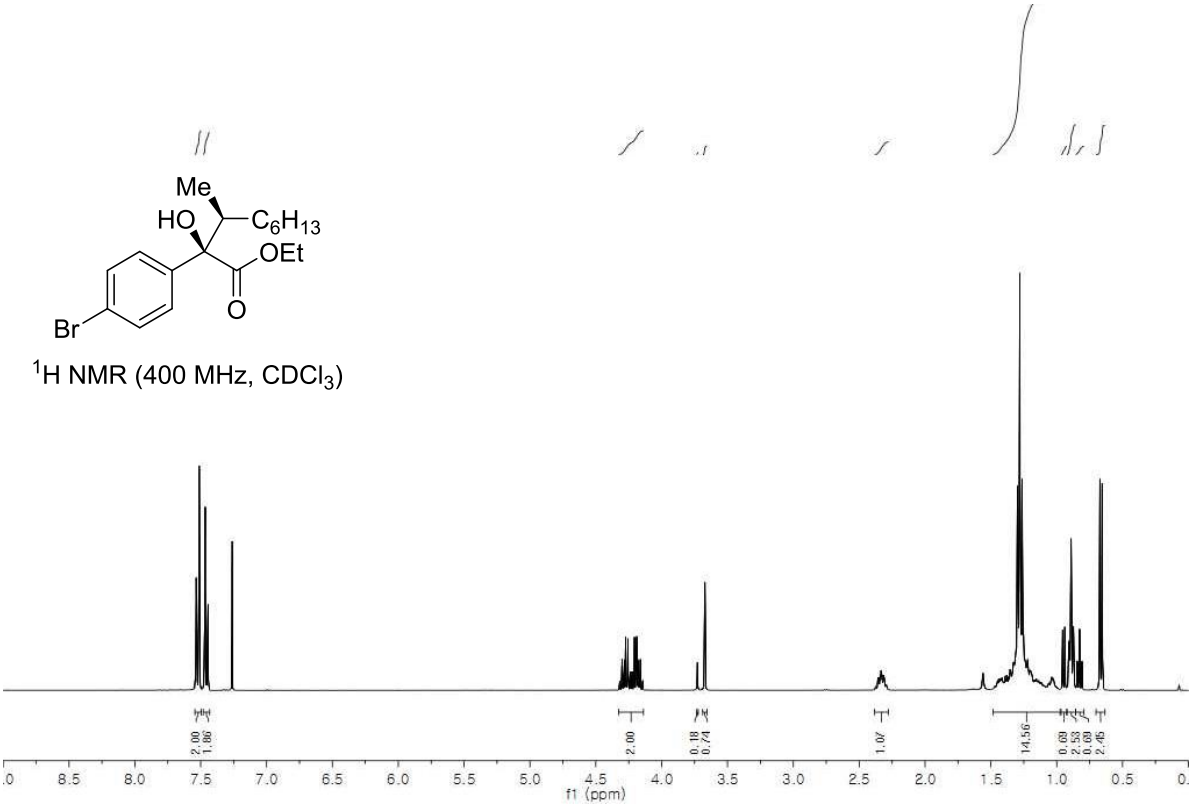
^{1}H NMR (400 MHz, $CDCl_3$)

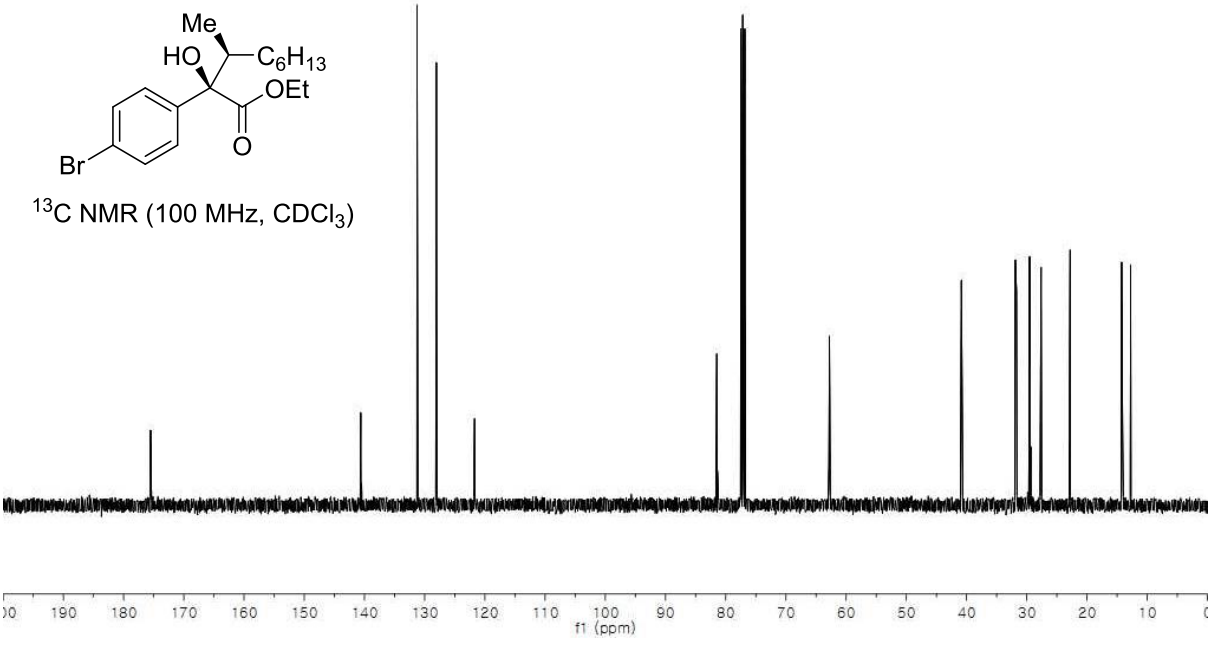


^{13}C NMR (100 MHz, $CDCl_3$)

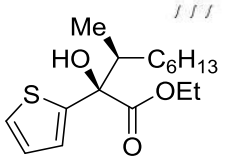


Ethyl 2-(4-bromophenyl)-2-hydroxy-3-methylnonanoate (4b).

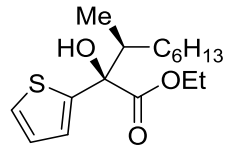




Ethyl 2-hydroxy-3-methyl-2-(thiophen-2-yl)nonanoate (4i).

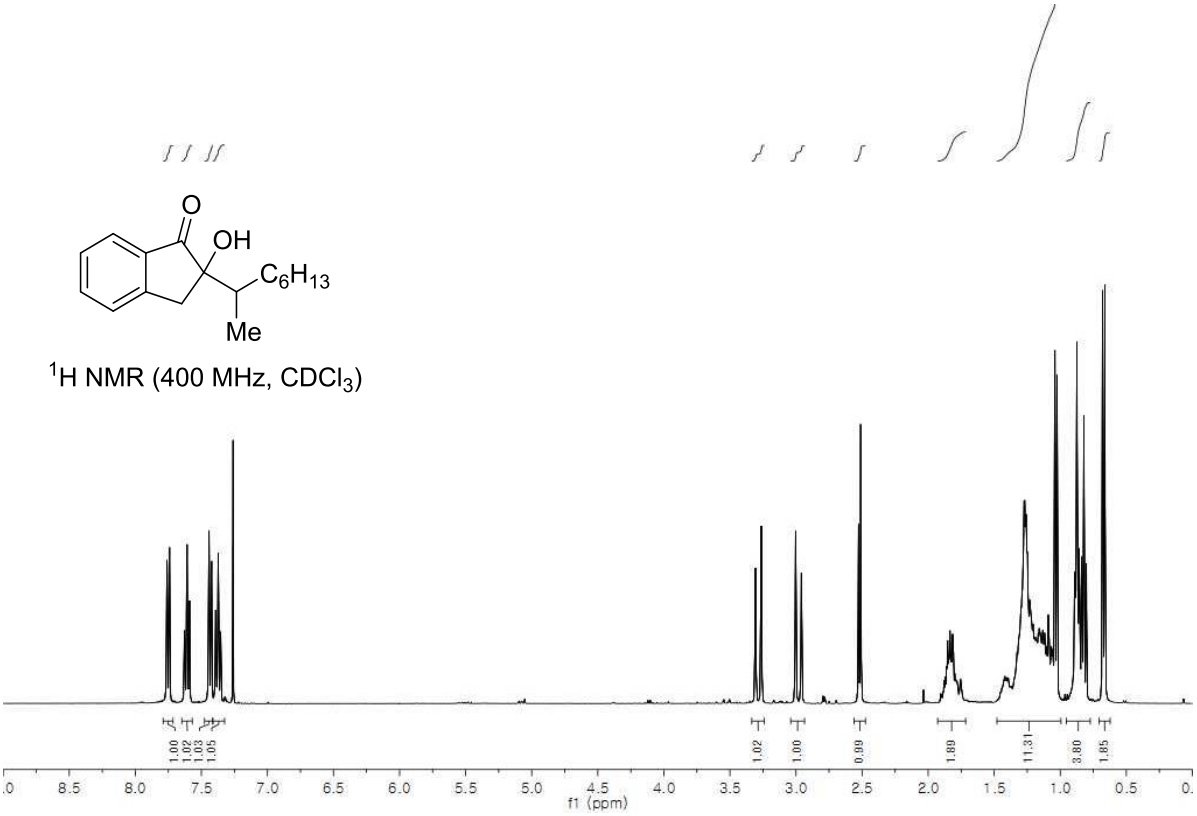


^{1}H NMR (400 MHz, $CDCl_3$)

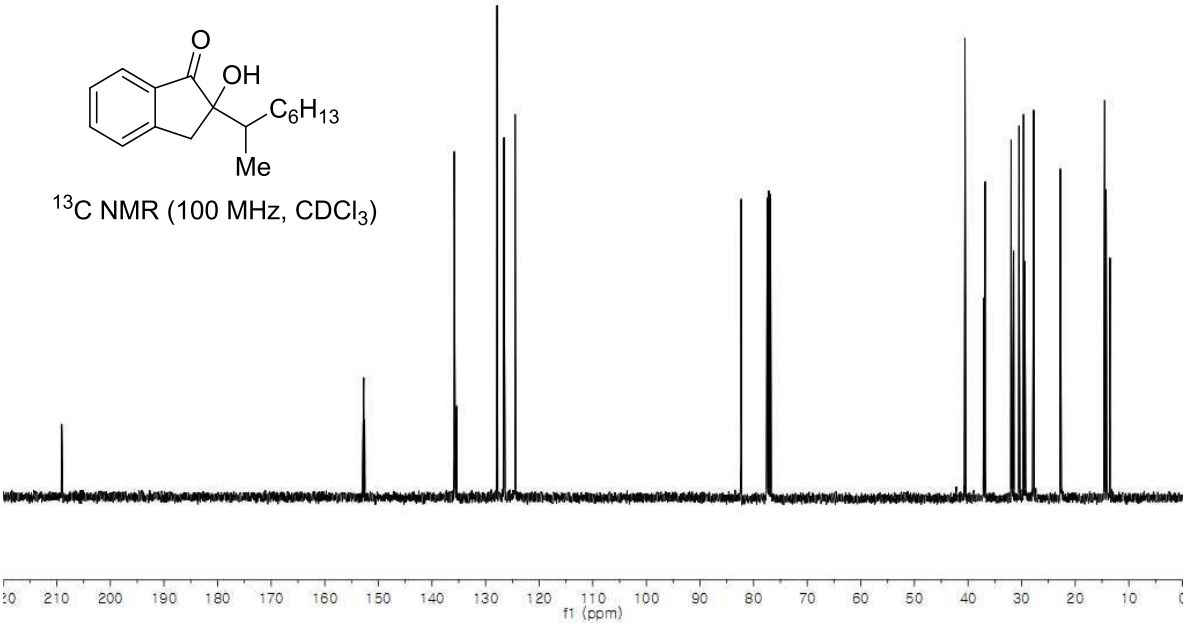


^{13}C NMR (100 MHz, $CDCl_3$)

2-Hydroxy-2-(octan-2-yl)-2,3-dihydro-1H-inden-1-one (4j).

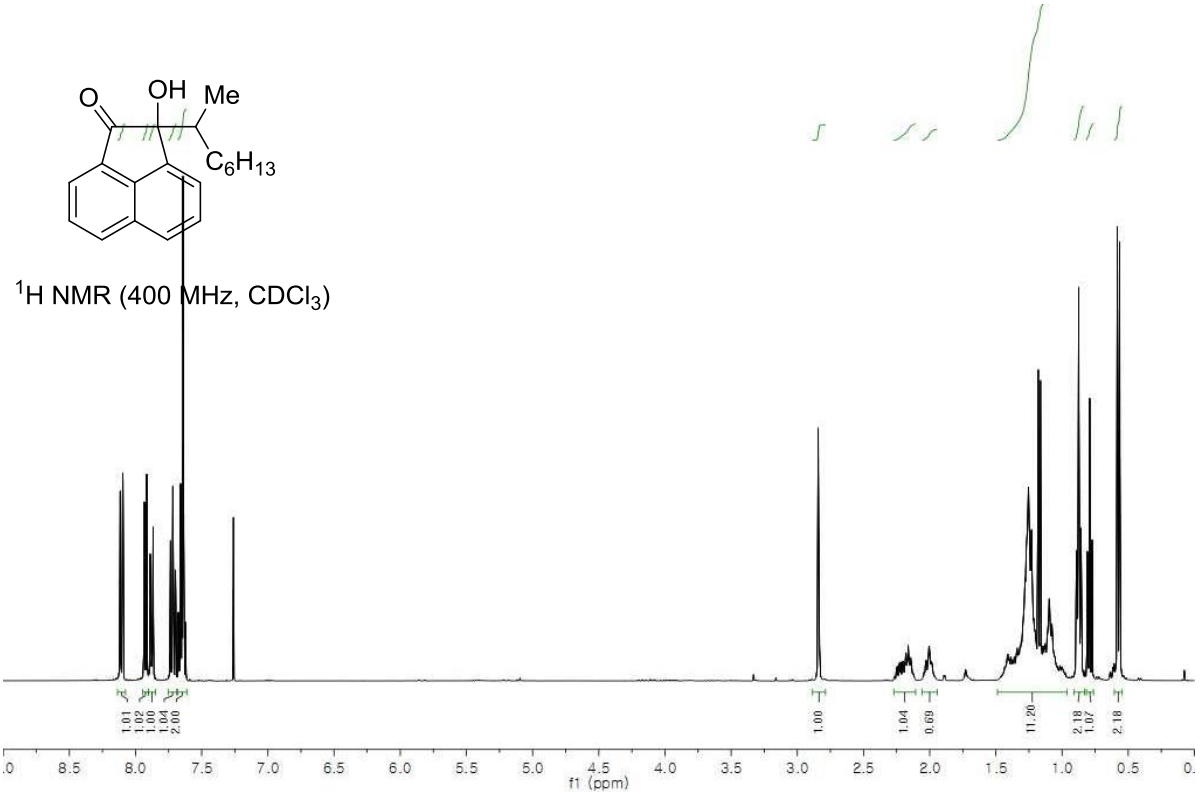


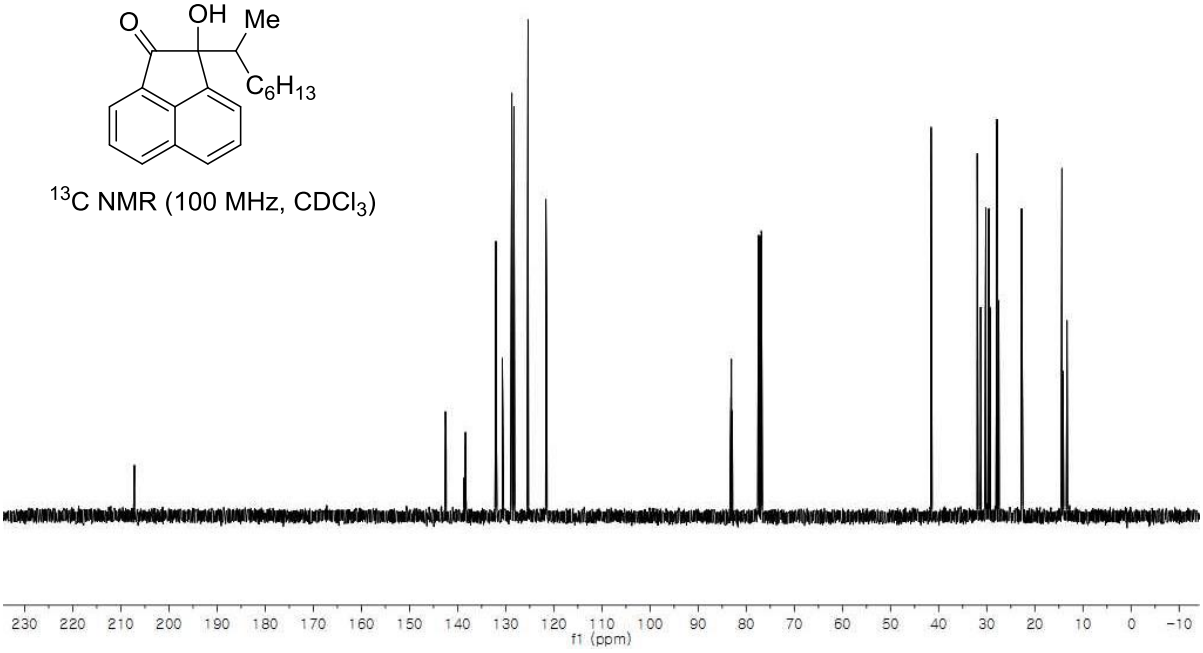
^{1}H NMR (400 MHz, $CDCl_3$)



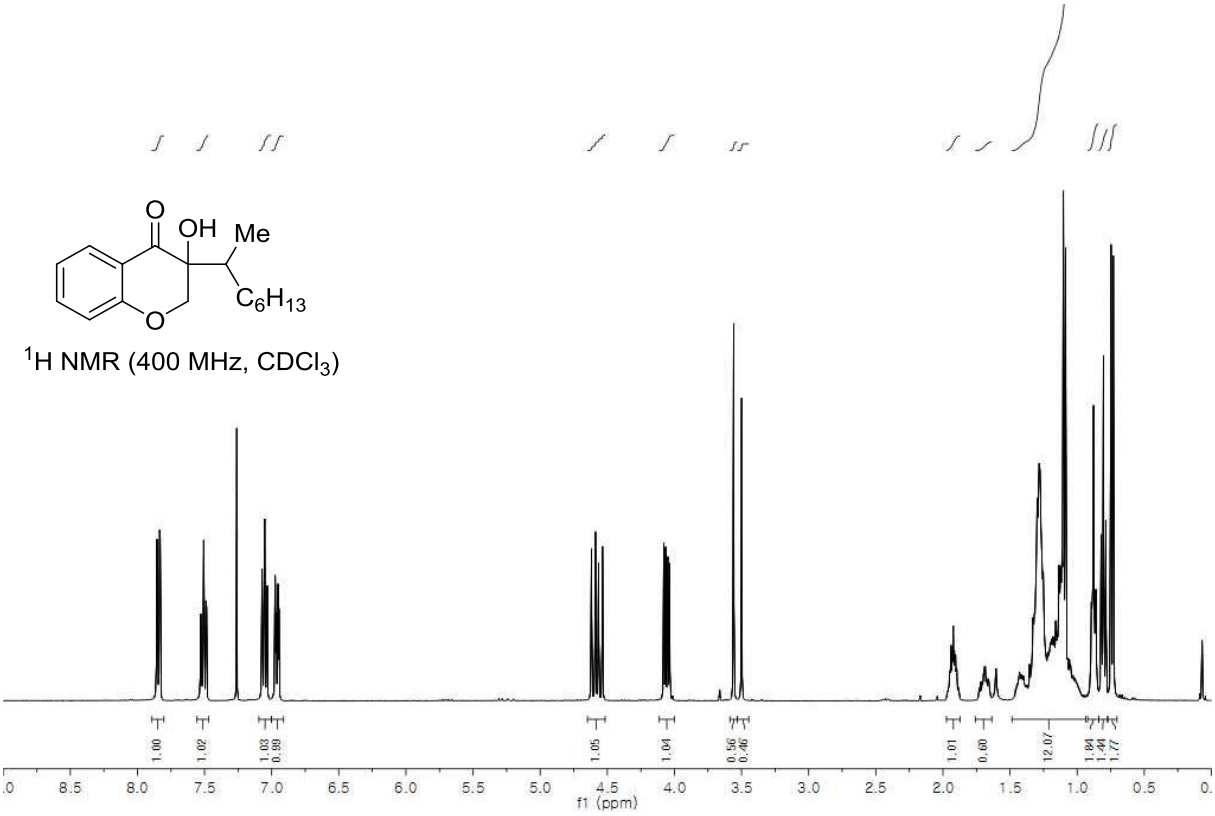
^{13}C NMR (100 MHz, $CDCl_3$)

2-Hydroxy-2-(octan-2-yl)acenaphthylen-1(2H)-one (4k).

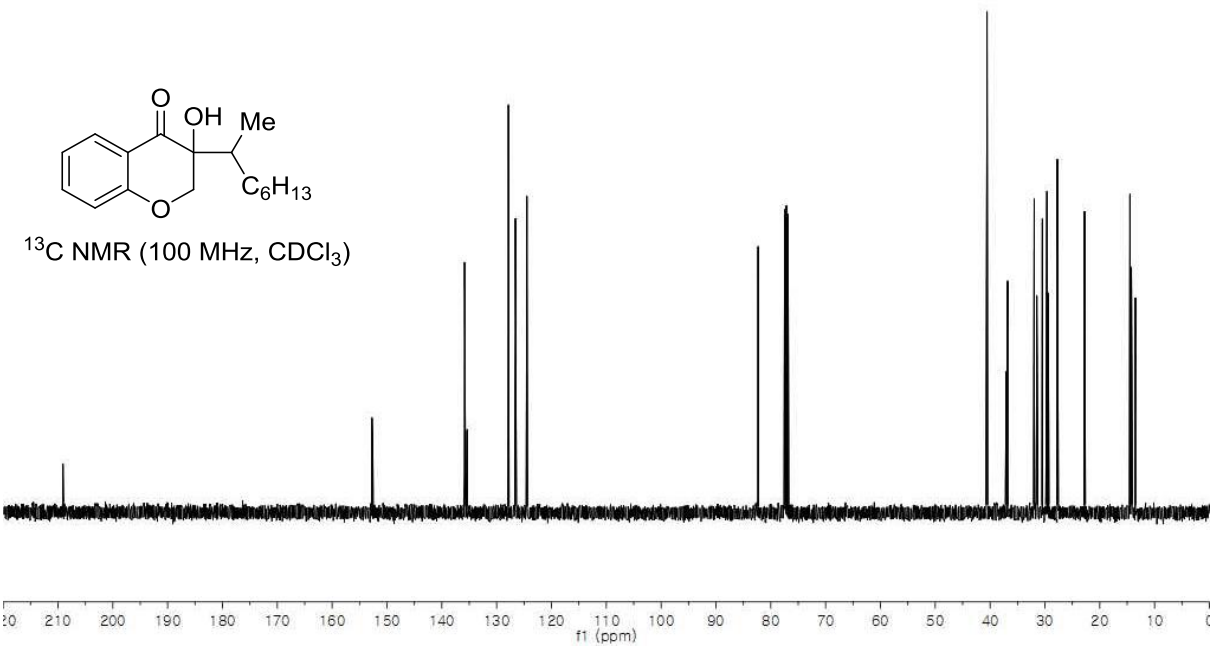




3-Hydroxy-3-(octan-2-yl)chroman-4-one (4m).

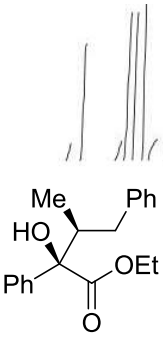


^{1}H NMR (400 MHz, $CDCl_3$)

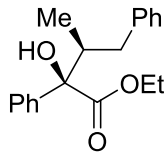


^{13}C NMR (100 MHz, $CDCl_3$)

Ethyl 2-hydroxy-3-methyl-2,4-diphenylbutanoate (5a).

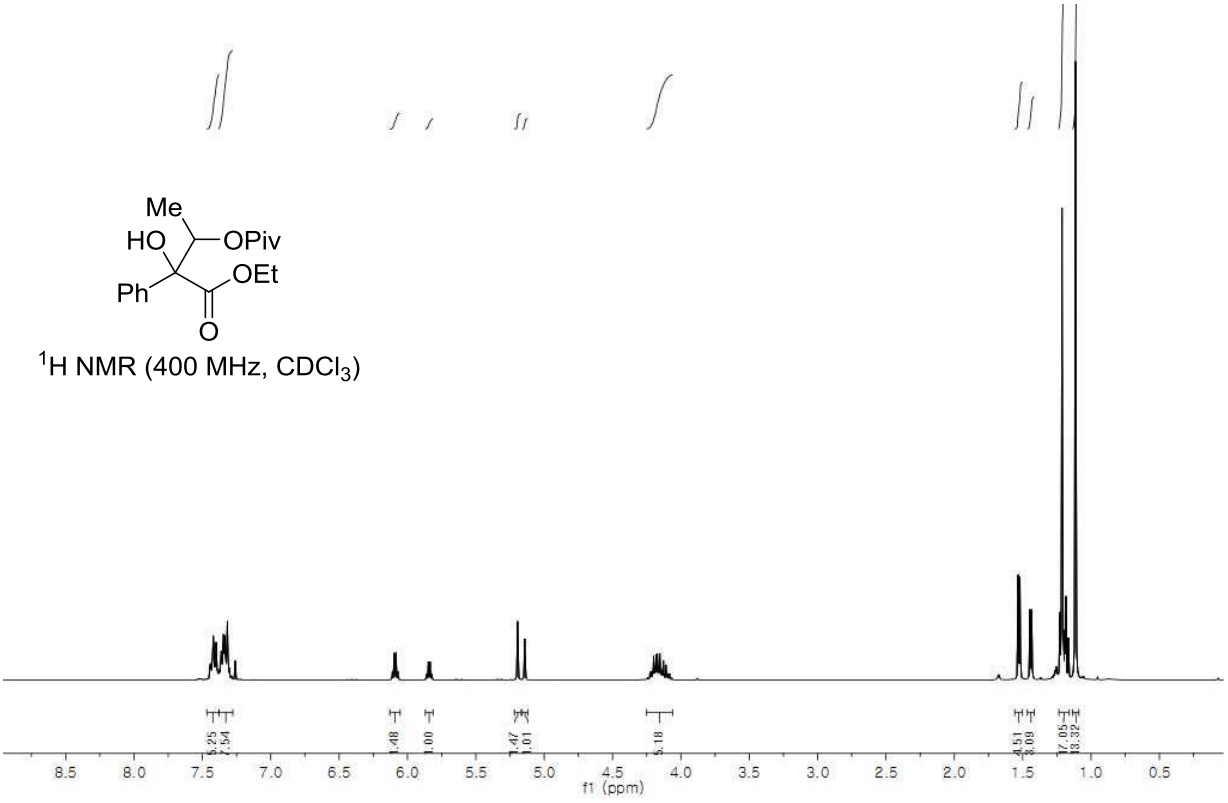


^{1}H NMR (400 MHz, $CDCl_3$)

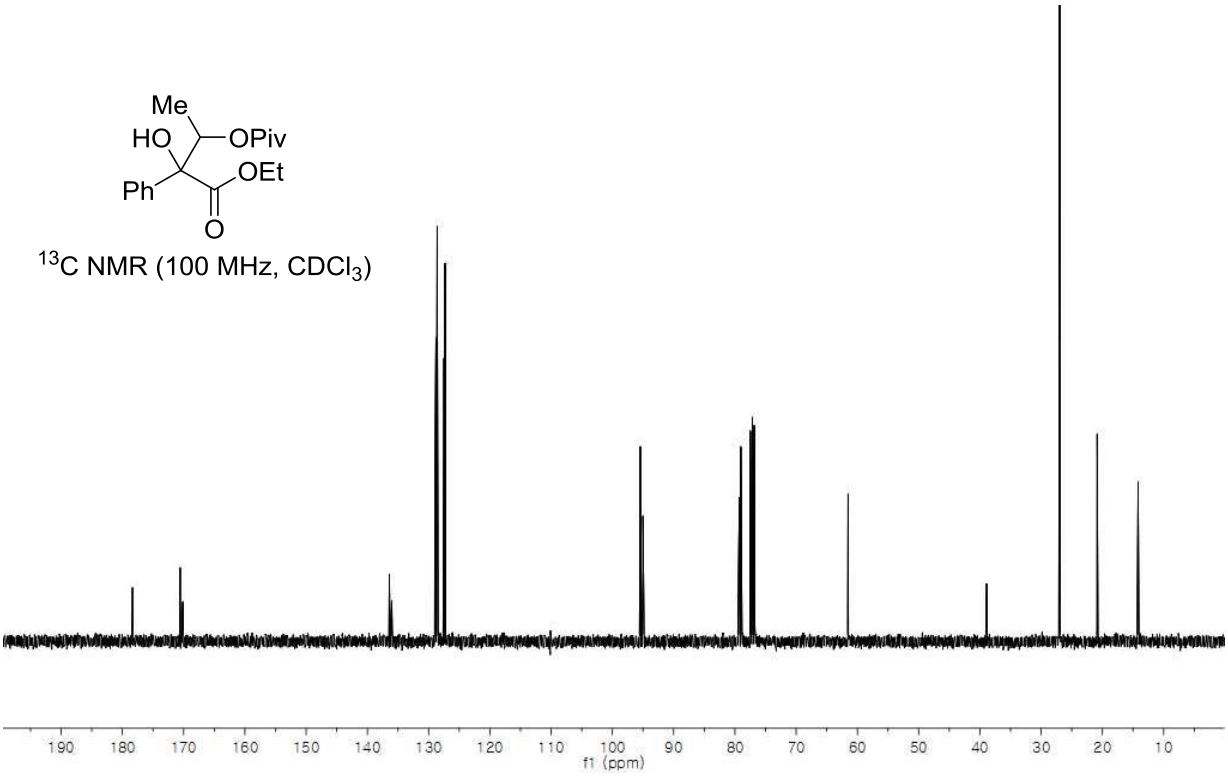


^{13}C NMR (100 MHz, $CDCl_3$)

Ethyl 2-hydroxy-2-phenyl-3-(pivaloyloxy)butanoate (6a).

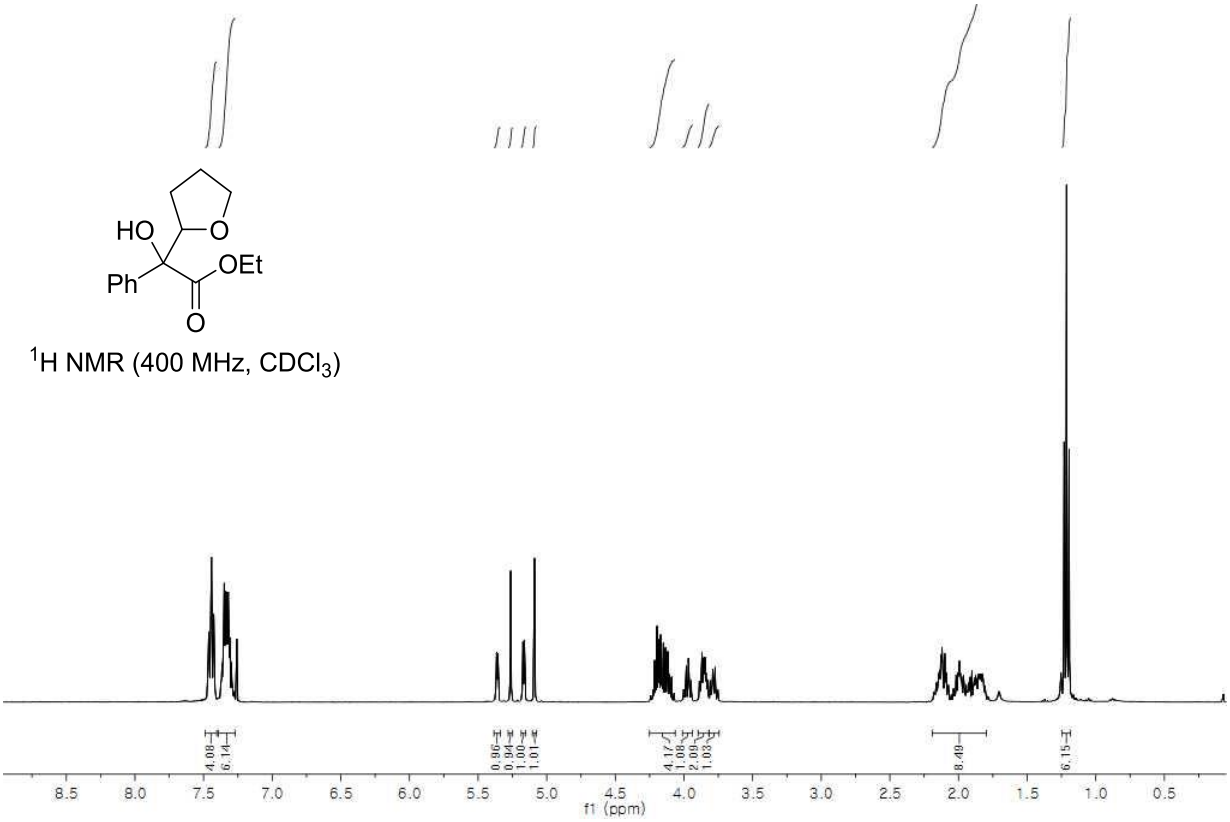


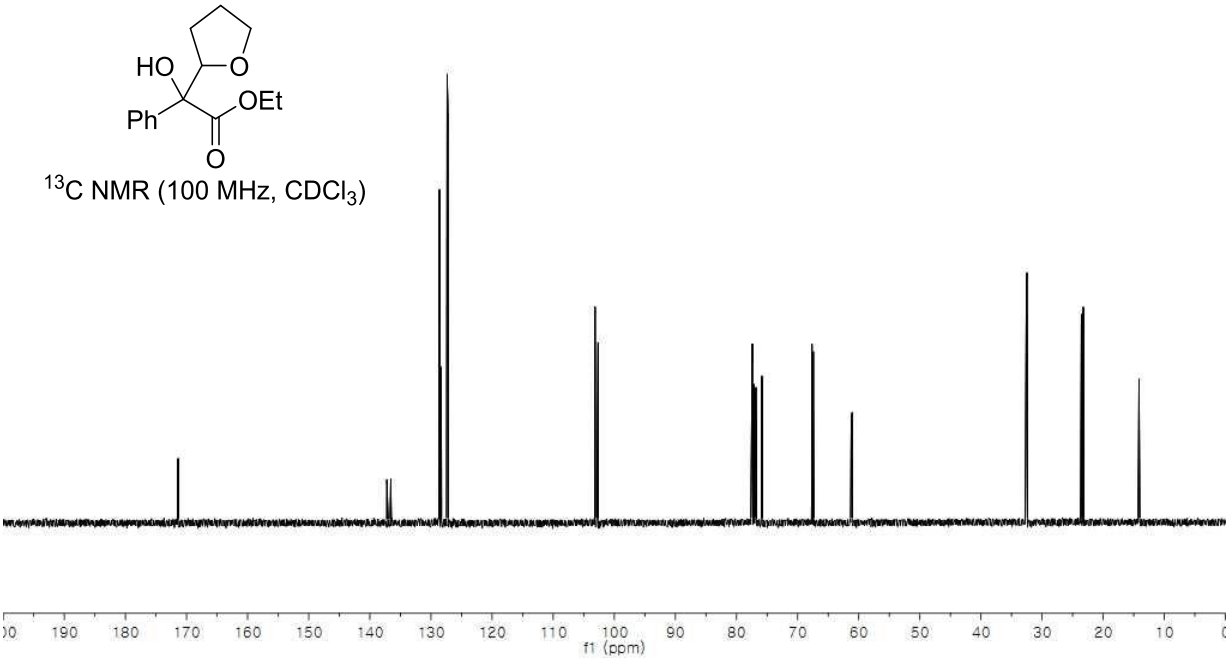
^{1}H NMR (400 MHz, $CDCl_3$)



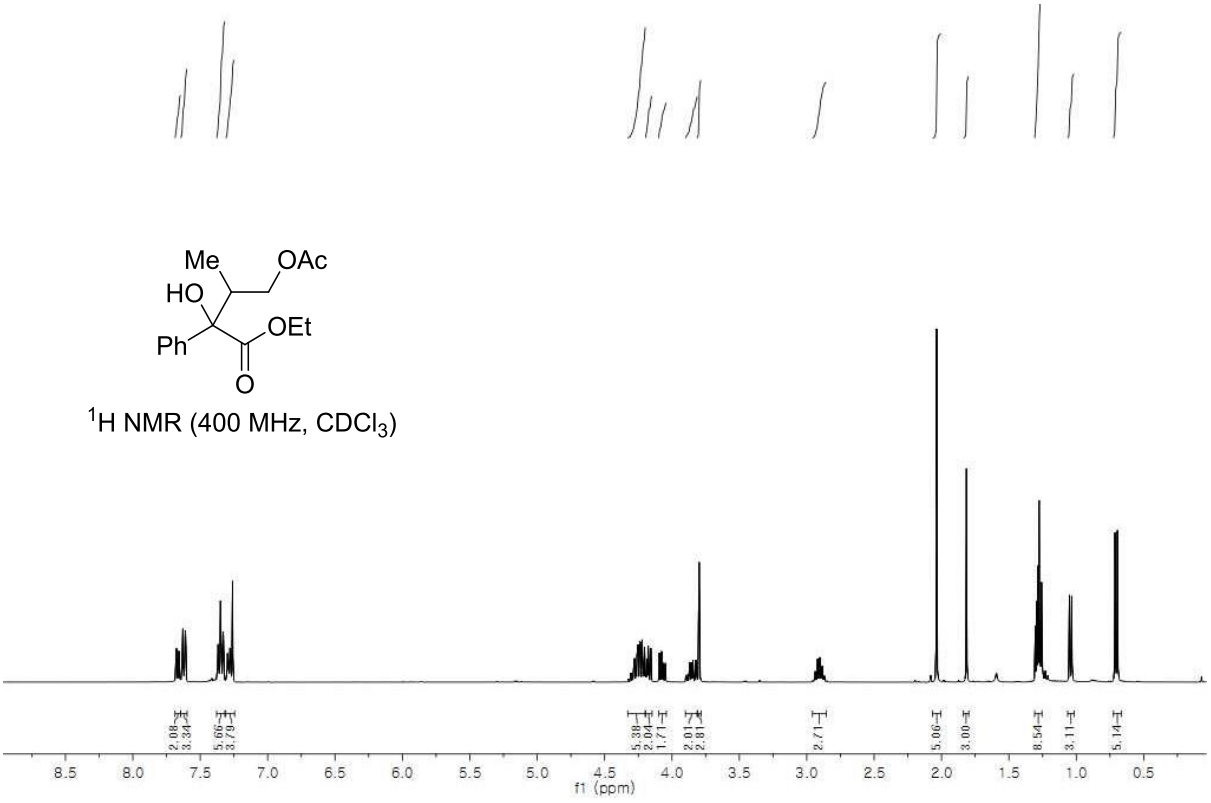
^{13}C NMR (100 MHz, $CDCl_3$)

Ethyl 2-hydroxy-2-phenyl-2-(tetrahydrofuran-2-yl)acetate (7a).

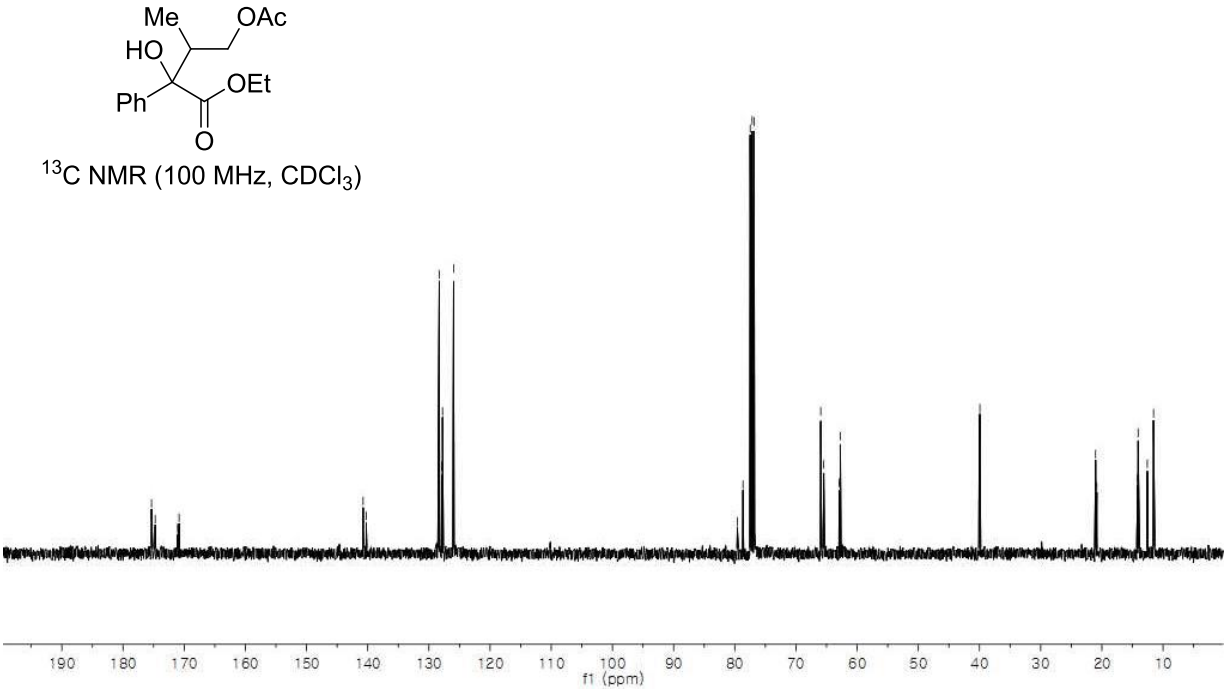




Ethyl 4-acetoxy-2-hydroxy-3-methyl-2-phenylbutanoate (8a).

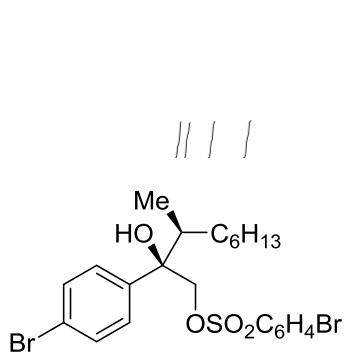


^{1}H NMR (400 MHz, $CDCl_3$)

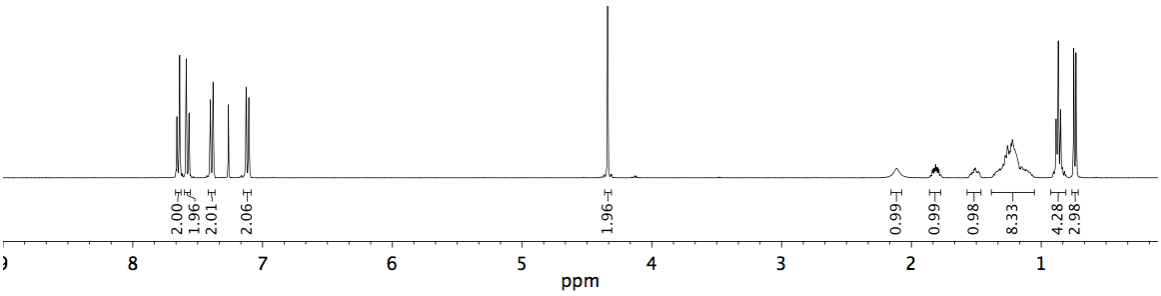


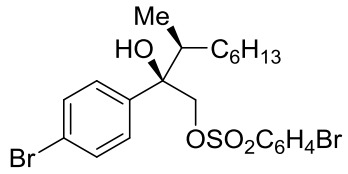
^{13}C NMR (100 MHz, $CDCl_3$)

2-(4-bromophenyl)-2-hydroxy-3-methylnonyl 4-bromobenzenesulfonate (4b Derivative).

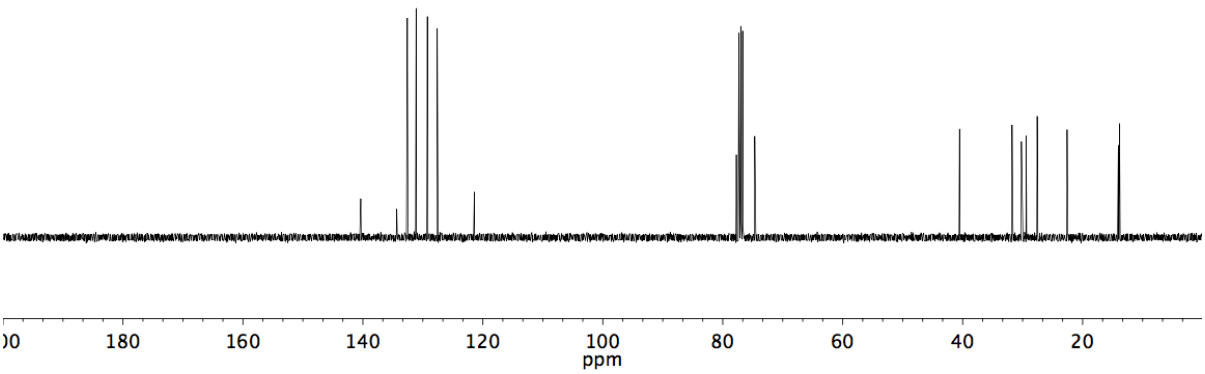


^{1}H NMR (400 MHz, $CDCl_3$)

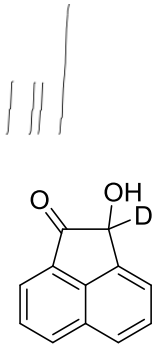




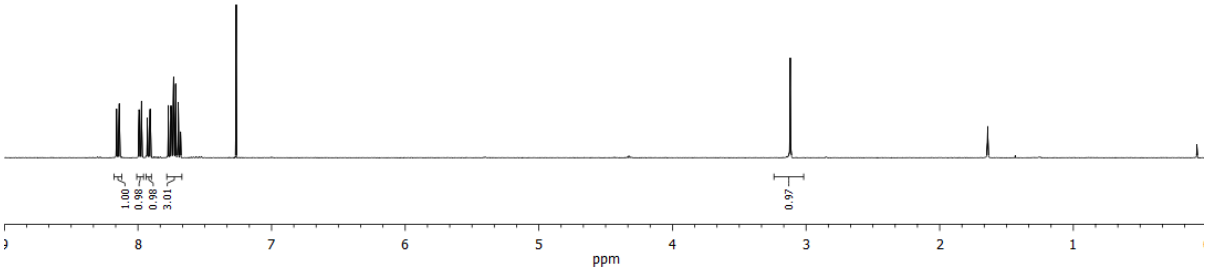
^{13}C NMR (100 MHz, $CDCl_3$)

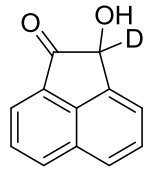


2-hydroxyacenaphtylen-1(2H)-one-2-d (*deuterio*-1k).

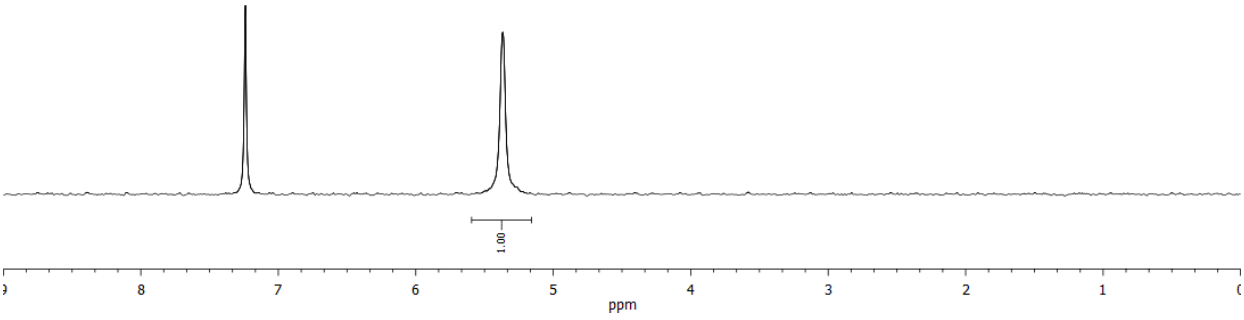


^{1}H NMR (400 MHz, $CDCl_3$)

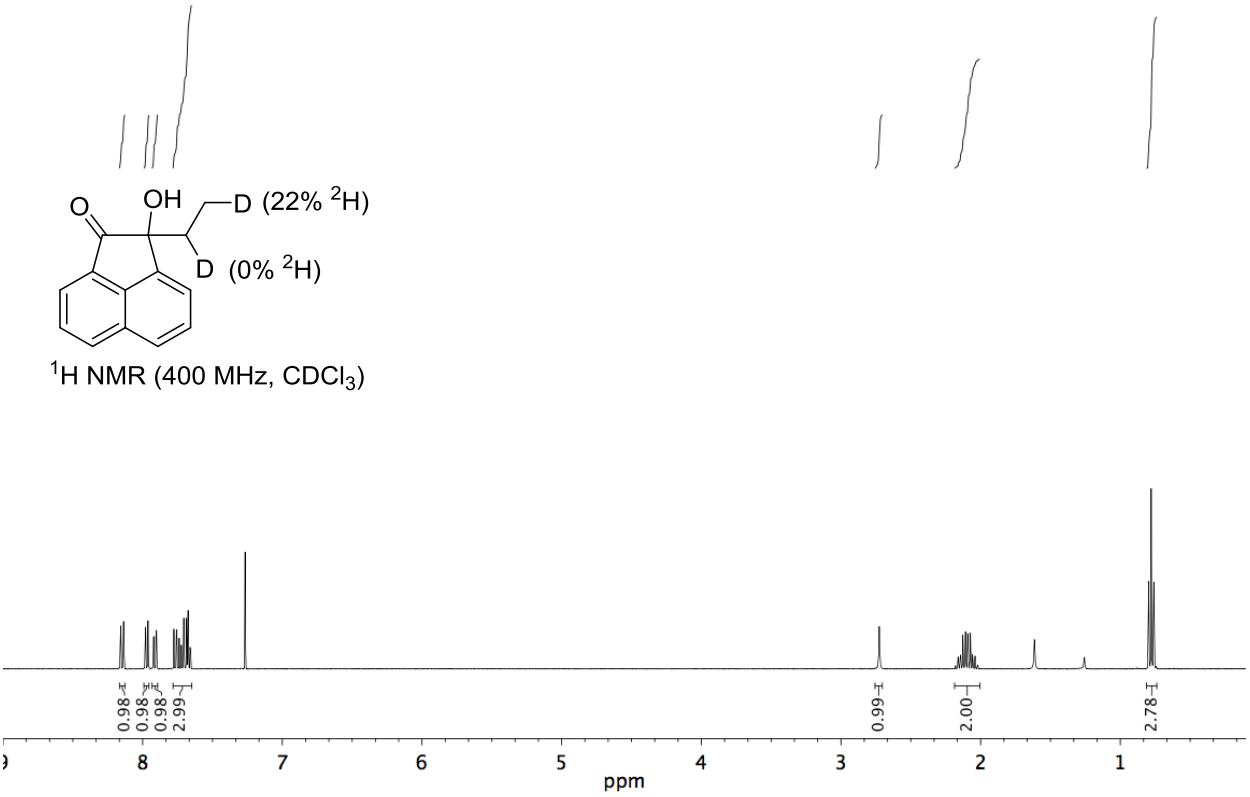




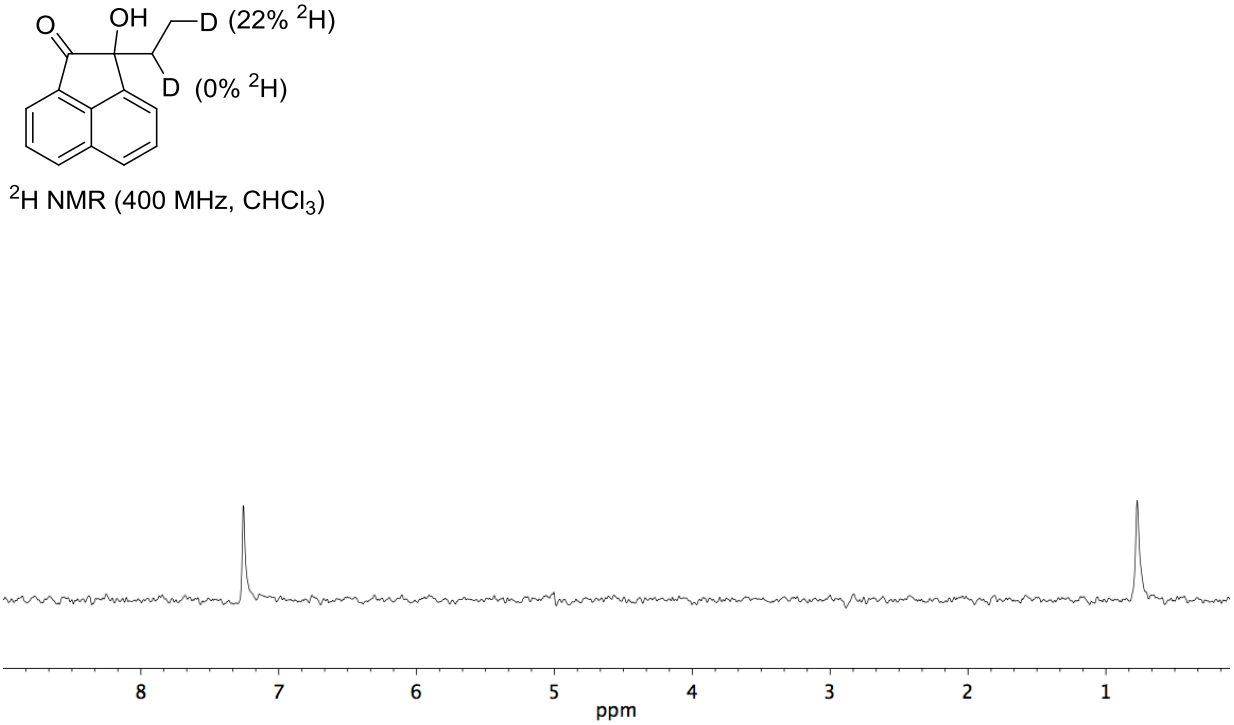
^{2}H NMR (400 MHz, $CHCl_3$)



2-(ethyl-1,2-d2)-2-hydroxyacenaphthylen-1(2H)-one (*deuterio*-3k).



^{1}H NMR (400 MHz, $CDCl_3$)



^{2}H NMR (400 MHz, $CHCl_3$)

Crystallographic Data

X-ray Experimental for complex **$Os_3(CO)_{11}$(XPhos)**:

Crystals grew as yellow prisms by vapor diffusion of pentane and methanol. The data crystal had approximate dimensions; 0.28 x 0.12 x 0.07 mm. The data were collected on a diffractometer using a μ-focus Cu Kα radiation source (λ = 1.5418Å) with collimating mirror monochromators. A total of 3355 frames of data were collected using ω-scans with a scan range of 1° and a counting time of 2 seconds per frame with a detector offset of +/- 40.8° and 5 seconds per frame with a detector offset of +/- 106.8°. The data were collected at 100 K. Details of crystal data, data collection and structure refinement are listed in Table 1. Data collection, unit cell refinement and data reduction were performed. The structure was solved by direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms . The hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms).

The function, $\Sigma w(|F_O|^2 - |F_C|^2)^2$, was minimized, where $w = 1/[(\sigma (F_O))^2 + (0.0178*P)^2 + (11.8122*P)]$ and $P = (|F_O|^2 + 2|F_C|^2)/3$. $R_W(F^2)$ refined to 0.0517, with R(F) equal to 0.0222 and a goodness of fit, S, = 1.00. Definitions used for calculating R(F), $R_W(F^2)$ and the goodness of fit, S, are given below.[1] The data were checked for secondary extinction effects but no correction was necessary. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[2] Tables of positional and thermal parameters, bond lengths and angles, torsion angles and figures are found elsewhere.

Crystallographic Material for **$Os_3(CO)_{11}$(XPhos)**

X-Ray Experimental

Table 1. Crystallographic Material for **$Os_3(CO)_{11}$(XPhos)**

Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of **$Os_3(CO)_{11}$(XPhos)**

Table 3. Bond Lengths (Å) and Angles (^{o}) for the non-hydrogen atoms of **$Os_3(CO)_{11}$(XPhos)**.

Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of **$Os_3(CO)_{11}$(XPhos)**.

Table 5. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of **$Os_3(CO)_{11}$(XPhos)**.

Table 6. Torsion Angles (^{o}) for the non-hydrogen atoms of **$Os_3(CO)_{11}$(XPhos)**.

Figure 1 View of **$Os_3(CO)_{11}$(XPhos)** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The methyl group hydrogen atoms were omitted for clarity.

Table 1. Crystal data and structure refinement for **$Os_3(CO)_{11}$(XPhos)**.

Empirical formula	C44 H49 O11 Os3 P	
Formula weight	1355.40	
Temperature	100(2) K	
Wavelength	1.54184 Å	
Crystal system	triclinic	
Space group	P -1	
Unit cell dimensions	a = 11.6048(2) Å	α= 74.3970(10)°.
	b = 13.5575(2) Å	β= 75.4040(10)°.
	c = 15.1224(2) Å	γ= 84.1860(10)°.
Volume	2216.17(6) $Å^3$	
Z	2	
Density (calculated)	2.031 Mg/m^3	
Absorption coefficient	16.715 mm^{-1}	
F(000)	1288	
Crystal size	0.280 x 0.120 x 0.070 mm^3	
Theta range for data collection	3.121 to 73.521°.	
Index ranges	-14<=h<=14, -16<=k<=16, -18<=l<=18	
Reflections collected	40515	
Independent reflections	8704 [R(int) = 0.0304]	
Completeness to theta = 67.684°	99.3 %	
Absorption correction	Gaussian	
Max. and min. transmission	1.00 and 0.183	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8704 / 0 / 538	
Goodness-of-fit on F^2	1.004	
Final R indices [I>2sigma(I)]	R1 = 0.0222, wR2 = 0.0515	
R indices (all data)	R1 = 0.0229, wR2 = 0.0517	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.482 and -1.160 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 1. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C1	5953(3)	7616(3)	3080(3)	15(1)
C2	5537(3)	6831(3)	2794(3)	15(1)
C3	4554(3)	6289(3)	3407(3)	20(1)
C4	3934(3)	6536(3)	4232(3)	20(1)
C5	4275(3)	7368(3)	4464(3)	18(1)
C6	5277(3)	7885(3)	3901(3)	15(1)
C7	5925(3)	6554(3)	1859(3)	16(1)
C8	6807(3)	5774(3)	1715(3)	16(1)
C9	7010(3)	5467(3)	880(3)	18(1)
C10	6386(3)	5906(3)	181(3)	17(1)
C11	5513(3)	6656(3)	351(3)	18(1)
C12	5262(3)	6976(3)	1186(3)	16(1)
C13	7492(3)	5247(3)	2456(3)	18(1)
C14	6873(4)	4286(3)	3113(3)	29(1)
C15	8788(4)	4978(3)	2030(3)	26(1)
C16	6690(3)	5522(3)	-714(3)	22(1)
C17	7977(4)	5736(5)	-1247(4)	48(1)
C18	5861(4)	5947(3)	-1375(3)	26(1)
C19	4217(3)	7744(3)	1353(3)	21(1)
C20	4236(4)	8675(3)	519(3)	28(1)
C21	3029(4)	7216(4)	1605(4)	32(1)
C22	8089(3)	8103(3)	1441(2)	14(1)
C23	7445(3)	8699(3)	677(3)	17(1)
C24	7942(3)	8384(3)	-246(3)	21(1)
C25	9275(4)	8563(3)	-598(3)	25(1)
C26	9934(3)	8006(3)	161(3)	22(1)
C27	9434(3)	8298(3)	1096(3)	18(1)
C28	6899(3)	9653(3)	2540(3)	15(1)
C29	7921(3)	10397(3)	2082(3)	16(1)
C30	7517(3)	11474(3)	2221(3)	20(1)
C31	6418(3)	11870(3)	1842(3)	23(1)

C32	5414(3)	11115(3)	2263(3)	22(1)
C33	5808(3)	10040(3)	2125(3)	18(1)
C34	7386(3)	6604(3)	4465(3)	17(1)
C35	9584(3)	6633(3)	3280(3)	17(1)
C36	9815(3)	8612(3)	3067(3)	17(1)
C37	6432(3)	7798(3)	5795(3)	20(1)
C38	7772(3)	8993(3)	6413(3)	20(1)
C39	6619(3)	9890(3)	4887(3)	20(1)
C40	8967(3)	9707(3)	4439(3)	18(1)
C41	10802(3)	8158(3)	4680(3)	20(1)
C42	10581(3)	6074(3)	5172(3)	19(1)
C43	8269(3)	6242(3)	6117(3)	18(1)
C44	9858(3)	7355(3)	6569(3)	21(1)
O1	6715(2)	5983(2)	4806(2)	18(1)
O2	10250(2)	6033(2)	3013(2)	26(1)
O3	10578(2)	9119(2)	2610(2)	21(1)
O4	5651(2)	7287(2)	6150(2)	23(1)
O5	7788(3)	9100(2)	7133(2)	29(1)
O6	6024(2)	10600(2)	4712(2)	26(1)
O7	9631(2)	10315(2)	4000(2)	22(1)
O8	11584(2)	8660(2)	4289(2)	24(1)
O9	11209(2)	5396(2)	5031(2)	27(1)
O10	7581(2)	5654(2)	6529(2)	22(1)
O11	10077(3)	7445(2)	7241(2)	28(1)
Os1	8583(1)	7636(1)	3775(1)	12(1)
Os2	7710(1)	8783(1)	5217(1)	14(1)
Os3	9551(1)	7198(1)	5429(1)	14(1)
P1	7390(1)	8278(1)	2639(1)	12(1)

Table 3. Bond lengths [Å] and angles [°] for 1.

C1-C6	1.408(5)	C16-H16	1.00
C1-C2	1.419(5)	C17-H17A	0.98
C1-P1	1.858(3)	C17-H17B	0.98
C2-C3	1.403(5)	C17-H17C	0.98
C2-C7	1.507(5)	C18-H18A	0.98
C3-C4	1.382(5)	C18-H18B	0.98
C3-H3	0.95	C18-H18C	0.98
C4-C5	1.387(5)	C19-C20	1.523(6)
C4-H4	0.95	C19-C21	1.532(5)
C5-C6	1.383(5)	C19-H19	1.00
C5-H5	0.95	C20-H20A	0.98
C6-H6	0.95	C20-H20B	0.98
C7-C12	1.396(5)	C20-H20C	0.98
C7-C8	1.417(5)	C21-H21A	0.98
C8-C9	1.391(5)	C21-H21B	0.98
C8-C13	1.516(5)	C21-H21C	0.98
C9-C10	1.400(6)	C22-C23	1.536(5)
C9-H9	0.95	C22-C27	1.541(5)
C10-C11	1.388(5)	C22-P1	1.856(4)
C10-C16	1.525(5)	C22-H22	1.00
C11-C12	1.397(5)	C23-C24	1.527(5)
C11-H11	0.95	C23-H23A	0.99
C12-C19	1.534(5)	C23-H23B	0.99
C13-C14	1.528(5)	C24-C25	1.525(5)
C13-C15	1.529(5)	C24-H24A	0.99
C13-H13	1.00	C24-H24B	0.99
C14-H14A	0.98	C25-C26	1.528(6)
C14-H14B	0.98	C25-H25A	0.99
C14-H14C	0.98	C25-H25B	0.99
C15-H15A	0.98	C26-C27	1.528(5)
C15-H15B	0.98	C26-H26A	0.99
C15-H15C	0.98	C26-H26B	0.99
C16-C17	1.522(6)	C27-H27A	0.99
C16-C18	1.524(6)	C27-H27B	0.99

C28-C29	1.535(5)	C36-O3	1.137(5)
C28-C33	1.536(5)	C36-Os1	1.947(4)
C28-P1	1.870(4)	C37-O4	1.127(5)
C28-H28	1.00	C37-Os2	1.968(4)
C29-C30	1.541(5)	C38-O5	1.141(5)
C29-H29A	0.99	C38-Os2	1.924(4)
C29-H29B	0.99	C39-O6	1.138(5)
C30-C31	1.519(5)	C39-Os2	1.906(4)
C30-H30A	0.99	C40-O7	1.136(5)
C30-H30B	0.99	C40-Os2	1.947(4)
C31-C32	1.526(5)	C41-O8	1.128(5)
C31-H31A	0.99	C41-Os3	1.961(4)
C31-H31B	0.99	C42-O9	1.146(5)
C32-C33	1.536(5)	C42-Os3	1.902(4)
C32-H32A	0.99	C43-O10	1.128(5)
C32-H32B	0.99	C43-Os3	1.965(4)
C33-H33A	0.99	C44-O11	1.147(5)
C33-H33B	0.99	C44-Os3	1.915(4)
C34-O1	1.130(5)	Os1-P1	2.4028(9)
C34-Os1	1.963(4)	Os1-Os3	2.8912(2)
C35-O2	1.147(5)	Os1-Os2	2.9312(2)
C35-Os1	1.884(4)	Os2-Os3	2.8857(2)

C6-C1-C2	118.1(3)	C6-C5-H5	120.4
C6-C1-P1	110.9(3)	C4-C5-H5	120.4
C2-C1-P1	130.5(3)	C5-C6-C1	122.5(3)
C3-C2-C1	117.6(3)	C5-C6-H6	118.8
C3-C2-C7	113.5(3)	C1-C6-H6	118.8
C1-C2-C7	128.6(3)	C12-C7-C8	120.2(3)
C4-C3-C2	123.0(3)	C12-C7-C2	117.7(3)
C4-C3-H3	118.5	C8-C7-C2	121.4(3)
C2-C3-H3	118.5	C9-C8-C7	118.2(4)
C3-C4-C5	119.1(3)	C9-C8-C13	119.8(3)
C3-C4-H4	120.4	C7-C8-C13	121.9(3)
C5-C4-H4	120.4	C8-C9-C10	122.5(3)
C6-C5-C4	119.2(3)	C8-C9-H9	118.7

C10-C9-H9	118.7	H17A-C17-H17B	109.5
C11-C10-C9	117.8(3)	C16-C17-H17C	109.5
C11-C10-C16	124.0(4)	H17A-C17-H17C	109.5
C9-C10-C16	118.2(3)	H17B-C17-H17C	109.5
C10-C11-C12	121.7(4)	C16-C18-H18A	109.5
C10-C11-H11	119.1	C16-C18-H18B	109.5
C12-C11-H11	119.1	H18A-C18-H18B	109.5
C7-C12-C11	119.5(3)	C16-C18-H18C	109.5
C7-C12-C19	122.0(3)	H18A-C18-H18C	109.5
C11-C12-C19	118.4(3)	H18B-C18-H18C	109.5
C8-C13-C14	111.1(3)	C20-C19-C21	109.6(3)
C8-C13-C15	113.0(3)	C20-C19-C12	113.6(3)
C14-C13-C15	109.9(3)	C21-C19-C12	110.4(3)
C8-C13-H13	107.5	C20-C19-H19	107.7
C14-C13-H13	107.5	C21-C19-H19	107.7
C15-C13-H13	107.5	C12-C19-H19	107.7
C13-C14-H14A	109.5	C19-C20-H20A	109.5
C13-C14-H14B	109.5	C19-C20-H20B	109.5
H14A-C14-H14B	109.5	H20A-C20-H20B	109.5
C13-C14-H14C	109.5	C19-C20-H20C	109.5
H14A-C14-H14C	109.5	H20A-C20-H20C	109.5
H14B-C14-H14C	109.5	H20B-C20-H20C	109.5
C13-C15-H15A	109.5	C19-C21-H21A	109.5
C13-C15-H15B	109.5	C19-C21-H21B	109.5
H15A-C15-H15B	109.5	H21A-C21-H21B	109.5
C13-C15-H15C	109.5	C19-C21-H21C	109.5
H15A-C15-H15C	109.5	H21A-C21-H21C	109.5
H15B-C15-H15C	109.5	H21B-C21-H21C	109.5
C17-C16-C10	110.8(3)	C23-C22-C27	109.6(3)
C17-C16-C18	109.9(4)	C23-C22-P1	114.1(2)
C10-C16-C18	114.2(3)	C27-C22-P1	115.2(2)
C17-C16-H16	107.2	C23-C22-H22	105.7
C10-C16-H16	107.2	C27-C22-H22	105.7
C18-C16-H16	107.2	P1-C22-H22	105.7
C16-C17-H17A	109.5	C24-C23-C22	111.0(3)
C16-C17-H17B	109.5	C24-C23-H23A	109.4

C22-C23-H23A	109.4	C30-C29-H29A	109.5
C24-C23-H23B	109.4	C28-C29-H29B	109.5
C22-C23-H23B	109.4	C30-C29-H29B	109.5
H23A-C23-H23B	108.0	H29A-C29-H29B	108.1
C25-C24-C23	110.8(3)	C31-C30-C29	111.3(3)
C25-C24-H24A	109.5	C31-C30-H30A	109.4
C23-C24-H24A	109.5	C29-C30-H30A	109.4
C25-C24-H24B	109.5	C31-C30-H30B	109.4
C23-C24-H24B	109.5	C29-C30-H30B	109.4
H24A-C24-H24B	108.1	H30A-C30-H30B	108.0
C24-C25-C26	110.5(3)	C30-C31-C32	111.7(3)
C24-C25-H25A	109.5	C30-C31-H31A	109.3
C26-C25-H25A	109.5	C32-C31-H31A	109.3
C24-C25-H25B	109.5	C30-C31-H31B	109.3
C26-C25-H25B	109.5	C32-C31-H31B	109.3
H25A-C25-H25B	108.1	H31A-C31-H31B	107.9
C25-C26-C27	112.5(3)	C31-C32-C33	112.4(3)
C25-C26-H26A	109.1	C31-C32-H32A	109.1
C27-C26-H26A	109.1	C33-C32-H32A	109.1
C25-C26-H26B	109.1	C31-C32-H32B	109.1
C27-C26-H26B	109.1	C33-C32-H32B	109.1
H26A-C26-H26B	107.8	H32A-C32-H32B	107.9
C26-C27-C22	110.3(3)	C32-C33-C28	109.6(3)
C26-C27-H27A	109.6	C32-C33-H33A	109.7
C22-C27-H27A	109.6	C28-C33-H33A	109.7
C26-C27-H27B	109.6	C32-C33-H33B	109.7
C22-C27-H27B	109.6	C28-C33-H33B	109.7
H27A-C27-H27B	108.1	H33A-C33-H33B	108.2
C29-C28-C33	110.8(3)	O1-C34-Os1	175.0(3)
C29-C28-P1	113.4(2)	O2-C35-Os1	175.1(3)
C33-C28-P1	117.5(2)	O3-C36-Os1	173.9(3)
C29-C28-H28	104.5	O4-C37-Os2	175.1(3)
C33-C28-H28	104.5	O5-C38-Os2	178.4(3)
P1-C28-H28	104.5	O6-C39-Os2	174.2(3)
C28-C29-C30	110.5(3)	O7-C40-Os2	173.9(3)
C28-C29-H29A	109.5	O8-C41-Os3	174.3(3)

O9-C42-Os3	178.8(4)	C37-Os2-Os3	92.57(11)
O10-C43-Os3	176.1(3)	C39-Os2-Os1	114.06(11)
O11-C44-Os3	178.0(4)	C38-Os2-Os1	148.27(12)
C35-Os1-C36	87.45(15)	C40-Os2-Os1	83.49(10)
C35-Os1-C34	89.96(15)	C37-Os2-Os1	87.80(11)
C36-Os1-C34	177.40(15)	Os3-Os2-Os1	59.602(5)
C35-Os1-P1	102.81(11)	C42-Os3-C44	103.39(17)
C36-Os1-P1	91.93(11)	C42-Os3-C41	90.45(15)
C34-Os1-P1	88.65(11)	C44-Os3-C41	90.20(16)
C35-Os1-Os3	92.71(11)	C42-Os3-C43	89.45(16)
C36-Os1-Os3	91.32(11)	C44-Os3-C43	92.86(16)
C34-Os1-Os3	88.79(11)	C41-Os3-C43	176.88(15)
P1-Os1-Os3	164.27(2)	C42-Os3-Os2	158.11(12)
C35-Os1-Os2	152.12(11)	C44-Os3-Os2	98.38(12)
C36-Os1-Os2	92.48(10)	C41-Os3-Os2	91.84(11)
C34-Os1-Os2	89.81(10)	C43-Os3-Os2	87.10(11)
P1-Os1-Os2	105.05(2)	C42-Os3-Os1	97.40(12)
Os3-Os1-Os2	59.417(5)	C44-Os3-Os1	159.09(12)
C39-Os2-C38	97.60(16)	C41-Os3-Os1	87.35(11)
C39-Os2-C40	86.50(16)	C43-Os3-Os1	89.57(11)
C38-Os2-C40	96.96(16)	Os2-Os3-Os1	60.982(5)
C39-Os2-C37	92.67(16)	C22-P1-C1	108.44(16)
C38-Os2-C37	93.00(16)	C22-P1-C28	109.09(16)
C40-Os2-C37	170.03(15)	C1-P1-C28	102.42(16)
C39-Os2-Os3	171.59(11)	C22-P1-Os1	114.51(12)
C38-Os2-Os3	88.69(12)	C1-P1-Os1	108.54(12)
C40-Os2-Os3	87.20(11)	C28-P1-Os1	113.05(12)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 1. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	15(2)	13(2)	15(2)	-2(1)	-1(1)	-4(1)
C2	15(2)	14(2)	15(2)	-2(1)	-3(1)	-1(1)
C3	20(2)	18(2)	20(2)	-3(2)	-1(2)	-8(1)
C4	15(2)	18(2)	23(2)	-1(2)	0(1)	-8(1)
C5	15(2)	20(2)	16(2)	-4(2)	0(1)	-2(1)
C6	12(2)	16(2)	16(2)	-2(1)	-3(1)	-2(1)
C7	15(2)	12(2)	19(2)	-4(1)	0(1)	-6(1)
C8	16(2)	16(2)	16(2)	-3(1)	-2(1)	-6(1)
C9	17(2)	16(2)	22(2)	-6(2)	-2(1)	-1(1)
C10	18(2)	22(2)	13(2)	-5(2)	-1(1)	-8(1)
C11	20(2)	19(2)	16(2)	-1(2)	-5(1)	-6(1)
C12	15(2)	15(2)	17(2)	-3(2)	-1(1)	-5(1)
C13	20(2)	19(2)	16(2)	-7(2)	-5(1)	-3(1)
C14	34(2)	26(2)	25(2)	1(2)	-8(2)	-8(2)
C15	23(2)	27(2)	28(2)	-7(2)	-7(2)	3(2)
C16	23(2)	27(2)	18(2)	-9(2)	-4(2)	0(2)
C17	26(2)	91(5)	29(3)	-28(3)	0(2)	1(3)
C18	26(2)	35(2)	20(2)	-11(2)	-3(2)	-4(2)
C19	21(2)	21(2)	23(2)	-8(2)	-8(2)	2(2)
C20	30(2)	24(2)	34(2)	-6(2)	-14(2)	2(2)
C21	19(2)	33(2)	41(3)	-7(2)	-3(2)	0(2)
C22	15(2)	14(2)	12(2)	-2(1)	0(1)	-5(1)
C23	20(2)	16(2)	15(2)	-1(1)	-3(1)	-5(1)
C24	24(2)	25(2)	14(2)	-5(2)	0(2)	-9(2)
C25	28(2)	30(2)	12(2)	-4(2)	3(2)	-10(2)
C26	18(2)	26(2)	20(2)	-7(2)	4(2)	-6(2)
C27	15(2)	22(2)	15(2)	-3(2)	1(1)	-6(1)
C28	15(2)	14(2)	14(2)	-3(1)	-2(1)	-2(1)
C29	16(2)	12(2)	19(2)	0(1)	-3(1)	-5(1)
C30	23(2)	16(2)	21(2)	-4(2)	-3(2)	-7(1)
C31	25(2)	15(2)	29(2)	-6(2)	-7(2)	-1(2)

C32	19(2)	18(2)	28(2)	-7(2)	-4(2)	0(1)
C33	16(2)	17(2)	20(2)	-3(2)	-4(1)	-2(1)
C34	19(2)	16(2)	15(2)	-3(2)	-5(1)	3(1)
C35	17(2)	20(2)	14(2)	-3(2)	-5(1)	-4(1)
C36	21(2)	18(2)	16(2)	-6(2)	-9(2)	4(2)
C37	24(2)	20(2)	17(2)	-7(2)	-6(2)	1(2)
C38	18(2)	22(2)	22(2)	-7(2)	-4(2)	-3(1)
C39	19(2)	26(2)	14(2)	-6(2)	1(1)	-5(2)
C40	20(2)	18(2)	20(2)	-9(2)	-7(2)	0(2)
C41	26(2)	14(2)	21(2)	-6(2)	-10(2)	4(2)
C42	16(2)	17(2)	27(2)	-7(2)	-6(2)	-4(1)
C43	23(2)	20(2)	12(2)	-3(2)	-8(2)	2(2)
C44	24(2)	16(2)	22(2)	-4(2)	-4(2)	-4(1)
O1	17(1)	17(1)	18(1)	1(1)	-2(1)	-6(1)
O2	22(1)	23(1)	31(2)	-12(1)	-1(1)	3(1)
O3	18(1)	22(1)	20(1)	-2(1)	0(1)	-11(1)
O4	22(1)	23(1)	24(2)	-7(1)	1(1)	-9(1)
O5	32(2)	38(2)	18(2)	-10(1)	-5(1)	-9(1)
O6	25(1)	23(2)	29(2)	-8(1)	-6(1)	4(1)
O7	23(1)	20(1)	24(2)	-6(1)	-4(1)	-7(1)
O8	20(1)	21(1)	29(2)	-3(1)	-4(1)	-6(1)
O9	19(1)	19(1)	45(2)	-10(1)	-5(1)	-1(1)
O10	24(1)	24(1)	16(1)	-1(1)	-2(1)	-9(1)
O11	41(2)	27(2)	22(2)	-4(1)	-15(1)	-6(1)
Os1	12(1)	13(1)	11(1)	-3(1)	-2(1)	-3(1)
Os2	14(1)	15(1)	14(1)	-5(1)	-2(1)	-3(1)
Os3	15(1)	14(1)	15(1)	-3(1)	-5(1)	-2(1)
P1	11(1)	13(1)	12(1)	-3(1)	-1(1)	-4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 1.

	x	y	z	U(eq)
H3	4303	5727	3248	24
H4	3283	6142	4635	24
H5	3825	7580	5004	21
H6	5518	8441	4074	18
H9	7595	4940	781	22
H11	5074	6959	-113	22
H13	7498	5733	2848	21
H14A	6070	4475	3435	43
H14B	7333	3959	3583	43
H14C	6817	3807	2743	43
H15A	8813	4454	1686	39
H15B	9210	4713	2536	39
H15C	9172	5593	1594	39
H16	6618	4761	-512	27
H17A	8080	6478	-1459	71
H17B	8169	5445	-1796	71
H17C	8509	5424	-829	71
H18A	5033	5825	-1023	40
H18B	6049	5604	-1891	40
H18C	5967	6685	-1637	40
H19	4266	7995	1907	25
H20A	5012	8992	338	43
H20B	3606	9172	698	43
H20C	4104	8460	-16	43
H21A	2959	6958	1075	49
H21B	2370	7709	1735	49
H21C	2997	6643	2167	49
H22	8013	7362	1487	17
H23A	6583	8566	898	21
H23B	7541	9443	567	21

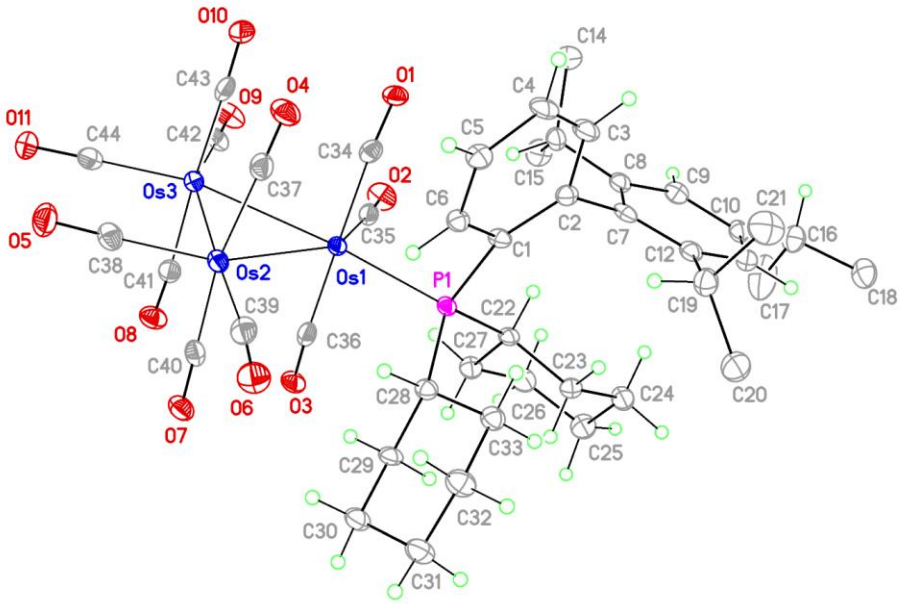
H24A	7801	7649	-146	25
H24B	7523	8786	-730	25
H25A	9411	9306	-766	30
H25B	9590	8311	-1173	30
H26A	10787	8171	-66	27
H26B	9879	7257	268	27
H27A	9567	9031	1011	22
H27B	9854	7888	1576	22
H28	6641	9709	3208	18
H29A	8182	10433	1399	20
H29B	8607	10142	2369	20
H30A	7339	11450	2902	24
H30B	8172	11952	1890	24
H31A	6629	11989	1146	27
H31B	6140	12533	1993	27
H32A	4740	11376	1962	27
H32B	5129	11071	2947	27
H33A	6011	10064	1443	21
H33B	5149	9565	2444	21

Table 6. Torsion angles [°] for 1.

C6-C1-C2-C3	-7.3(5)	C7-C8-C13-C15	-144.3(3)
P1-C1-C2-C3	163.6(3)	C11-C10-C16-C17	118.4(4)
C6-C1-C2-C7	165.6(4)	C9-C10-C16-C17	-62.6(5)
P1-C1-C2-C7	-23.5(6)	C11-C10-C16-C18	-6.4(5)
C1-C2-C3-C4	4.7(6)	C9-C10-C16-C18	172.7(3)
C7-C2-C3-C4	-169.2(4)	C7-C12-C19-C20	133.6(4)
C2-C3-C4-C5	1.3(6)	C11-C12-C19-C20	-49.5(5)
C3-C4-C5-C6	-4.6(6)	C7-C12-C19-C21	-102.9(4)
C4-C5-C6-C1	1.8(6)	C11-C12-C19-C21	74.0(4)
C2-C1-C6-C5	4.3(6)	C27-C22-C23-C24	58.4(4)
P1-C1-C6-C5	-168.3(3)	P1-C22-C23-C24	-170.7(2)
C3-C2-C7-C12	78.2(4)	C22-C23-C24-C25	-58.2(4)
C1-C2-C7-C12	-94.9(5)	C23-C24-C25-C26	55.4(4)
C3-C2-C7-C8	-91.9(4)	C24-C25-C26-C27	-55.0(4)
C1-C2-C7-C8	94.9(5)	C25-C26-C27-C22	55.8(4)
C12-C7-C8-C9	1.8(5)	C23-C22-C27-C26	-56.5(4)
C2-C7-C8-C9	171.7(3)	P1-C22-C27-C26	173.2(3)
C12-C7-C8-C13	-176.0(3)	C33-C28-C29-C30	57.9(4)
C2-C7-C8-C13	-6.0(5)	P1-C28-C29-C30	-167.4(3)
C7-C8-C9-C10	0.6(5)	C28-C29-C30-C31	-55.8(4)
C13-C8-C9-C10	178.4(3)	C29-C30-C31-C32	53.9(4)
C8-C9-C10-C11	-1.7(5)	C30-C31-C32-C33	-54.5(5)
C8-C9-C10-C16	179.2(3)	C31-C32-C33-C28	55.7(4)
C9-C10-C11-C12	0.5(5)	C29-C28-C33-C32	-57.3(4)
C16-C10-C11-C12	179.5(3)	P1-C28-C33-C32	170.0(3)
C8-C7-C12-C11	-3.0(5)	C23-C22-P1-C1	70.3(3)
C2-C7-C12-C11	-173.3(3)	C27-C22-P1-C1	-161.7(3)
C8-C7-C12-C19	173.9(3)	C23-C22-P1-C28	-40.6(3)
C2-C7-C12-C19	3.6(5)	C27-C22-P1-C28	87.5(3)
C10-C11-C12-C7	1.8(5)	C23-C22-P1-Os1	-168.4(2)
C10-C11-C12-C19	-175.2(3)	C27-C22-P1-Os1	-40.3(3)
C9-C8-C13-C14	-86.2(4)	C6-C1-P1-C22	-168.7(3)
C7-C8-C13-C14	91.6(4)	C2-C1-P1-C22	19.9(4)
C9-C8-C13-C15	37.9(5)	C6-C1-P1-C28	-53.5(3)

C2-C1-P1-C28	135.2(4)	C29-C28-P1-C1	-173.8(3)
C6-C1-P1-Os1	66.3(3)	C33-C28-P1-C1	-42.3(3)
C2-C1-P1-Os1	-105.0(4)	C29-C28-P1-Os1	69.6(3)
C29-C28-P1-C22	-59.0(3)	C33-C28-P1-Os1	-158.9(2)
C33-C28-P1-C22	72.4(3)		

Figure 1. View of **$Os_3(CO)_{11}$(XPhos)** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The methyl group hydrogen atoms were omitted for clarity.



X-ray Experimental for complex **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$**:

Crystals grew as colorless laths by vapor diffusion using pentane and methanol. The data crystal had approximate dimensions; 0.43 x 0.08 x 0.02 mm. The data were collected using a μ-focus Cu Kα radiation source (λ = 1.5418Å) with collimating mirror monochromators. A total of 2084 frames of data were collected using ω-scans with a scan range of 0.5° and a counting time of 10 seconds per frame with a detector offset of +/- 40.6° and 30 seconds per frame with a detector offset of +/- 106.8°. The data were collected at 100 K. Details of crystal data, data collection and structure refinement are listed in Table 1. Data collection, unit cell refinement and data reduction were performed.The structure was solved by direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms. The hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms).

One of the Os dimer complexes was disordered near a crystallographic mirror plane of symmetry at ½, y, z. The complex was displaced from the mirror by approximately 0.3Å. The disordered complex was refined using a large number of geometric and displacement parameter restraints. The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma (F_o))^2 + (0.1205*P)^2 + (64.9258*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.216, with R(F) equal to 0.0720 and a goodness of fit, S, = 1.07. Definitions used for calculating R(F), $R_w(F^2)$ and the goodness of fit, S, are given below.[1] The data were checked for secondary extinction effects but no correction was necessary. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[2] Tables of positional and thermal parameters, bond lengths and angles, torsion angles and figures are found elsewhere.

Crystallographic Material for **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$**

X-Ray Experimental

Table 7. Crystallographic Material for **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$**

Table 8. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$**

Table 9. Bond Lengths (Å) and Angles (^{o}) for the non-hydrogen atoms of **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$**

Table 10. Anisotropic thermal parameters for the non-hydrogen atoms of **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$**

Table 11. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$**

Figure 2 View of complex 1 of **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The hydrogen atoms have been omitted for clarity. The complex resides on a crystallographic mirror plane of symmetry at 0, y, z. Atoms with labels appended by a are related by –x, y, z.

Figure 3 View of complex 2 of **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The hydrogen atoms have been omitted for clarity. The complex is disordered near a crystallographic mirror plane of symmetry at ½, y, z.

Table 7. Crystal data and structure refinement for **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$**.

Empirical formula	C92 H128 O8 Os2 P2	
Formula weight	1804.28	
Temperature	100(2) K	
Wavelength	1.54184 Å	
Crystal system	orthorhombic	
Space group	P m c 21	
Unit cell dimensions	a = 36.3787(8) Å	α= 90°.
	b = 13.1217(6) Å	β= 90°.
	c = 17.3763(5) Å	γ = 90°.
Volume	8294.6(5) $Å^3$	
Z	4	
Density (calculated)	1.445 Mg/m^3	
Absorption coefficient	6.488 mm^{-1}	
F(000)	3704	
Crystal size	0.430 x 0.082 x 0.019 mm^3	
Theta range for data collection	2.429 to 73.404°.	
Index ranges	-45<=h<=40, -16<=k<=9, -21<=l<=21	
Reflections collected	48508	
Independent reflections	15805 [R(int) = 0.0500]	
Completeness to theta = 67.684°	100.0 %	
Absorption correction	Gaussian	
Max. and min. transmission	1.00 and 0.364	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	15805 / 3785 / 1440	
Goodness-of-fit on F^2	1.142	
Final R indices [I>2sigma(I)]	R1 = 0.0719, wR2 = 0.1993	
R indices (all data)	R1 = 0.0850, wR2 = 0.2157	
Absolute structure parameter	0.50(3)	
Extinction coefficient	0.00012(3)	
Largest diff. peak and hole	3.677 and -4.246 $e.Å^{-3}$	

Table 8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 1. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Os1	382(1)	6452(1)	4353(2)	13(1)
P1	1038(1)	6262(3)	4130(3)	21(1)
O1	310(3)	7198(8)	3269(6)	23(2)
O2	310(3)	5086(7)	3670(6)	20(2)
O3	393(3)	8512(6)	5106(8)	23(2)
O4	440(3)	5249(8)	5833(6)	29(2)
C1	1192(4)	6765(11)	3177(9)	20(2)
C2	1007(4)	6280(11)	2591(9)	21(3)
C3	1095(5)	6406(11)	1808(10)	27(3)
C4	1375(5)	7075(14)	1622(12)	36(3)
C5	1546(6)	7641(14)	2217(11)	31(3)
C6	1461(4)	7515(11)	2980(10)	25(3)
C7	1664(5)	8237(13)	3519(10)	33(3)
C8	2023(5)	8046(13)	3728(10)	35(3)
C9	2212(6)	8806(16)	4169(12)	47(4)
C10	2049(5)	9706(13)	4351(17)	49(3)
C11	1690(6)	9894(15)	4128(12)	45(4)
C12	1492(5)	9170(13)	3696(11)	35(3)
C13	2220(5)	7090(16)	3495(14)	46(4)
C14	2520(6)	7350(19)	2886(17)	61(6)
C15	2398(6)	6550(15)	4193(15)	53(5)
C16	2249(8)	10560(20)	4805(19)	80(6)
C17	2634(9)	10680(30)	4730(30)	107(10)
C18	2062(7)	10810(17)	5574(15)	58(5)
C19	1113(5)	9418(12)	3417(11)	34(3)
C20	1155(6)	10003(15)	2629(12)	44(4)
C21	881(5)	10059(13)	3985(13)	40(4)
C22	1175(4)	4906(10)	3958(9)	24(3)
C23	1594(5)	4808(12)	3816(12)	31(3)
C24	1700(6)	3695(13)	3578(13)	39(4)
C25	1554(5)	2930(12)	4175(12)	38(4)

C26	1149(5)	3031(9)	4283(17)	34(3)
C27	1032(5)	4108(10)	4525(9)	24(3)
C28	1349(4)	6719(12)	4888(9)	24(3)
C29	1220(5)	7742(11)	5220(10)	27(3)
C30	1492(6)	8220(13)	5788(12)	36(4)
C31	1541(7)	7469(16)	6463(13)	35(4)
C32	1666(5)	6440(11)	6193(12)	30(3)
C33	1400(5)	6005(13)	5583(11)	33(3)
C34	0	7387(12)	2968(10)	14(3)
C35	0	7804(15)	2141(11)	18(3)
C36	347(4)	8466(10)	2008(9)	23(3)
C37	345(4)	8841(13)	1182(10)	25(3)
C38	341(5)	7931(14)	639(10)	33(3)
C39	0	7259(15)	766(12)	24(4)
C40	0	6885(14)	1606(11)	21(4)
C41	0	9488(16)	1025(14)	31(4)
C42	0	4730(14)	3495(10)	15(3)
C43	0	3724(14)	3056(11)	14(3)
C44	-351(4)	3657(11)	2551(10)	24(3)
C45	-349(4)	2598(13)	2122(10)	29(3)
C46	-347(5)	1785(15)	2743(12)	35(4)
C47	0	1847(19)	3241(15)	36(4)
C48	0	2897(18)	3665(14)	36(4)
C49	0	2540(20)	1648(17)	39(5)
C50	395(4)	7703(9)	4846(9)	20(3)
C51	421(4)	5739(10)	5278(7)	19(2)
Os1A	4623(2)	8420(5)	6235(4)	62(1)
P1A	3966(3)	8458(7)	6463(7)	40(2)
O1A	4713(5)	7700(20)	7276(13)	53(5)
O2A	5331(5)	8010(30)	7338(13)	55(5)
O3A	4613(8)	6390(18)	5480(20)	53(7)
C45A	4625(6)	7190(20)	5800(20)	52(5)
O4A	4548(9)	9590(30)	4760(16)	55(8)
C46A	4572(6)	9090(30)	5297(16)	58(5)
C34A	5029(4)	7647(15)	7624(14)	57(4)
C35A	5041(6)	7120(20)	8435(13)	59(4)

C36A	5328(11)	6480(30)	8581(17)	61(6)
C37A	5315(10)	5990(30)	9450(20)	63(5)
C38A	5350(18)	6980(30)	9977(17)	61(6)
C39A	5046(15)	7690(20)	9816(17)	61(5)
C40A	5039(14)	8123(15)	9017(11)	58(5)
C41A	4655(10)	6640(30)	8575(17)	61(5)
C42A	4956(16)	5510(30)	9556(19)	65(6)
C43A	4661(17)	7140(50)	9973(17)	61(6)
C44A	4631(9)	6260(30)	9400(20)	64(5)
C1A	3801(8)	7940(20)	7360(20)	39(4)
C2A	3990(10)	8520(30)	8082(14)	43(5)
C3A	3893(10)	8360(30)	8760(30)	43(5)
C4A	3616(10)	7660(30)	8940(30)	43(5)
C5A	3450(10)	7160(30)	8383(18)	42(5)
C6A	3550(6)	7310(18)	7538(15)	37(4)
C7A	3367(9)	6520(30)	7040(13)	34(4)
C8A	2985(7)	6694(18)	6804(12)	35(4)
C9A	2799(10)	5930(30)	6419(16)	37(5)
C10A	2959(8)	4960(20)	6234(15)	39(4)
C11A	3325(10)	4880(30)	6487(18)	39(5)
C12A	3531(7)	5580(20)	6868(14)	39(4)
C13A	2765(7)	7620(20)	7016(14)	36(5)
C14A	2475(8)	7400(30)	7612(19)	41(7)
C15A	2604(9)	8160(20)	6326(19)	37(6)
C16A	2769(7)	4140(20)	5783(18)	42(5)
C17A	2359(7)	4250(30)	5750(30)	50(7)
C18A	2939(12)	3950(30)	5007(17)	49(8)
C19A	3897(7)	5260(20)	7193(15)	45(5)
C20A	3864(11)	4730(40)	7960(20)	53(8)
C21A	4132(10)	4700(30)	6620(20)	48(8)
C22A	3818(11)	9815(16)	6640(30)	44(4)
C23A	3403(8)	9950(30)	6800(30)	43(5)
C24A	3302(12)	11020(30)	7020(30)	44(5)
C25A	3428(12)	11800(40)	6480(30)	46(6)
C26A	3855(13)	11710(40)	6330(30)	46(6)
C27A	3950(13)	10612(17)	6100(30)	48(5)

C28A	3664(10)	7970(30)	5665(18)	37(4)
C29A	3791(11)	6920(30)	5330(20)	43(5)
C30A	3508(11)	6540(30)	4790(30)	37(5)
C31A	3462(11)	7240(30)	4130(20)	37(5)
C32A	3354(9)	8280(30)	4400(30)	35(5)
C33A	3605(10)	8760(30)	4996(16)	37(5)
Os1B	5386(2)	8663(5)	6249(4)	72(2)
P1B	6036(3)	8925(8)	6480(6)	36(2)
O1B	5268(4)	9890(20)	6955(19)	59(5)
O2B	4651(4)	9843(19)	6810(20)	56(5)
O3B	5452(12)	6630(20)	5480(30)	93(11)
C45B	5467(7)	7450(20)	5730(30)	76(6)
O4B	5390(13)	9850(40)	4778(17)	79(11)
C46B	5394(7)	9400(40)	5353(17)	68(6)
C34B	4944(3)	10269(19)	7061(12)	60(5)
C35B	4919(6)	11310(20)	7520(20)	71(5)
C36B	5043(13)	12151(17)	6953(15)	78(6)
C37B	5053(15)	13180(20)	7360(20)	77(6)
C38B	4702(13)	13340(20)	7830(30)	76(7)
C39B	4654(14)	12580(30)	8450(30)	73(6)
C40B	4635(12)	11450(20)	7990(20)	72(6)
C41B	5318(13)	11190(30)	8060(30)	74(6)
C42B	5382(13)	13090(30)	7990(30)	75(7)
C43B	5010(20)	12450(30)	8940(20)	77(6)
C44B	5332(14)	12200(30)	8460(30)	75(6)
C1B	6195(8)	8450(20)	7430(30)	44(4)
C2B	5987(10)	9050(30)	8096(14)	48(5)
C3B	6080(11)	8880(30)	8790(30)	49(6)
C4B	6355(11)	8190(30)	8990(30)	48(6)
C5B	6528(11)	7690(30)	8483(19)	48(5)
C6B	6439(6)	7840(20)	7607(16)	50(5)
C7B	6650(11)	7060(30)	7152(16)	53(5)
C8B	7032(8)	7320(20)	6926(15)	54(5)
C9B	7224(14)	6590(40)	6480(20)	58(6)
C10B	7095(13)	5690(30)	6234(19)	59(5)
C11B	6718(13)	5460(40)	6470(20)	58(6)

C12B	6504(8)	6130(20)	6914(17)	56(5)
C13B	7235(8)	8250(20)	7206(16)	55(6)
C14B	7506(12)	8020(30)	7830(20)	59(9)
C15B	7413(12)	8850(30)	6570(20)	57(9)
C16B	7317(10)	4930(30)	5783(17)	63(6)
C17B	7490(11)	4120(30)	6290(20)	59(8)
C18B	7101(14)	4420(40)	5150(20)	66(9)
C19B	6138(9)	5750(30)	7200(18)	58(6)
C20B	6160(13)	5210(40)	7960(20)	59(9)
C21B	5927(11)	5160(40)	6610(30)	63(9)
C22B	6179(11)	10286(16)	6640(30)	42(4)
C23B	6584(9)	10480(30)	6770(30)	46(5)
C24B	6670(13)	11580(40)	6960(30)	50(6)
C25B	6546(13)	12300(40)	6390(30)	49(6)
C26B	6121(12)	12220(40)	6290(30)	47(6)
C27B	6027(12)	11057(18)	6050(30)	45(5)
C28B	6353(10)	8410(30)	5720(20)	40(4)
C29B	6226(12)	7360(30)	5380(20)	44(5)
C30B	6488(13)	7050(40)	4810(30)	49(6)
C31B	6561(14)	7680(40)	4140(30)	49(6)
C32B	6672(12)	8740(30)	4460(30)	46(6)
C33B	6417(11)	9210(40)	5036(17)	42(5)

Table 9. Bond lengths [Å] and angles [°] for 1.

Os1-C50	1.852(12)	C12-C19	1.50(2)
Os1-C51	1.866(11)	C13-C15	1.55(3)
Os1-O1	2.139(10)	C13-C14	1.56(3)
Os1-O2	2.166(9)	C13-H13	1.00
Os1-P1	2.429(4)	C14-H14A	0.98
Os1-Os1#1	2.7820(10)	C14-H14B	0.98
P1-C28	1.837(15)	C14-H14C	0.98
P1-C1	1.868(16)	C15-H15A	0.98
P1-C22	1.872(14)	C15-H15B	0.98
O1-C34	1.267(13)	C15-H15C	0.98
O2-C42	1.259(13)	C16-C17	1.42(4)
O3-C50	1.154(12)	C16-C18	1.53(4)
O4-C51	1.160(12)	C16-H16	1.00
C1-C2	1.38(2)	C17-H17A	0.98
C1-C6	1.43(2)	C17-H17B	0.98
C2-C3	1.41(2)	C17-H17C	0.98
C2-H2	0.95	C18-H18A	0.98
C3-C4	1.38(2)	C18-H18B	0.98
C3-H3	0.95	C18-H18C	0.98
C4-C5	1.42(3)	C19-C21	1.55(2)
C4-H4	0.95	C19-C20	1.58(2)
C5-C6	1.37(3)	C19-H19	1.00
C5-H5	0.95	C20-H20A	0.98
C6-C7	1.52(2)	C20-H20B	0.98
C7-C8	1.38(3)	C20-H20C	0.98
C7-C12	1.41(2)	C21-H21A	0.98
C8-C9	1.43(3)	C21-H21B	0.98
C8-C13	1.50(3)	C21-H21C	0.98
C9-C10	1.36(3)	C22-C27	1.53(2)
C9-H9	0.95	C22-C23	1.55(2)
C10-C11	1.38(3)	C22-H22	1.00
C10-C16	1.56(3)	C23-C24	1.57(2)
C11-C12	1.41(3)	C23-H23A	0.99
C11-H11	0.95	C23-H23B	0.99

C24-C25	1.54(3)	C37-C38	1.52(2)
C24-H24A	0.99	C37-C41	1.541(19)
C24-H24B	0.99	C37-H37	1.00
C25-C26	1.49(3)	C38-C39	1.54(2)
C25-H25A	0.99	C38-H38A	0.99
C25-H25B	0.99	C38-H38B	0.99
C26-C27	1.535(19)	C39-C38#1	1.54(2)
C26-H26A	0.99	C39-C40	1.54(3)
C26-H26B	0.99	C39-H39	1.00
C27-H27A	0.99	C40-H40A	0.99
C27-H27B	0.99	C40-H40B	0.99
C28-C29	1.53(2)	C41-C37#1	1.541(19)
C28-C33	1.54(2)	C41-H41A	0.99
C28-H28	1.00	C41-H41B	0.99
C29-C30	1.53(2)	C42-O2#1	1.259(13)
C29-H29A	0.99	C42-C43	1.52(2)
C29-H29B	0.99	C43-C48	1.52(3)
C30-C31	1.54(3)	C43-C44#1	1.550(17)
C30-H30A	0.99	C43-C44	1.551(16)
C30-H30B	0.99	C44-C45	1.58(2)
C31-C32	1.50(3)	C44-H44A	0.99
C31-H31A	0.99	C44-H44B	0.99
C31-H31B	0.99	C45-C49	1.51(2)
C32-C33	1.54(2)	C45-C46	1.52(2)
C32-H32A	0.99	C45-H45	1.00
C32-H32B	0.99	C46-C47	1.53(2)
C33-H33A	0.99	C46-H46A	0.99
C33-H33B	0.99	C46-H46B	0.99
C34-O1#1	1.267(13)	C47-C46#1	1.53(2)
C34-C35	1.54(2)	C47-C48	1.56(3)
C35-C40	1.52(3)	C47-H47	1.00
C35-C36	1.550(17)	C48-H48A	0.99
C35-C36#1	1.550(17)	C48-H48B	0.99
C36-C37	1.52(2)	C49-C45#1	1.51(2)
C36-H36A	0.99	C49-H49A	0.99
C36-H36B	0.99	C49-H49B	0.99

Os1A-C45A	1.79(2)	C42A-H42A	0.99
Os1A-C46A	1.86(2)	C42A-H42B	0.99
Os1A-O1A	2.066(17)	C43A-C44A	1.52(6)
Os1A-O2B	2.122(17)	C43A-H43A	0.99
Os1A-P1A	2.424(9)	C43A-H43B	0.99
Os1A-Os1B	2.794(3)	C44A-H44C	1.00
P1A-C1A	1.80(4)	C1A-C6A	1.27(4)
P1A-C28A	1.882(14)	C1A-C2A	1.62(5)
P1A-C22A	1.887(14)	C2A-C3A	1.24(5)
O1A-C34A	1.30(2)	C2A-H2A	0.95
O2A-C34A	1.29(2)	C3A-C4A	1.40(6)
O2A-Os1B	2.089(17)	C3A-H3A	0.95
O3A-C45A	1.182(14)	C4A-C5A	1.32(5)
O4A-C46A	1.148(15)	C4A-H4A	0.95
C34A-C35A	1.57(3)	C5A-C6A	1.53(4)
C35A-C36A	1.36(3)	C5A-H5A	0.95
C35A-C41A	1.55(3)	C6A-C7A	1.508(14)
C35A-C40A	1.66(3)	C7A-C12A	1.40(4)
C36A-C37A	1.64(4)	C7A-C8A	1.47(4)
C36A-H36C	0.99	C8A-C9A	1.39(5)
C36A-H36D	0.99	C8A-C13A	1.505(14)
C37A-C42A	1.46(6)	C9A-C10A	1.43(5)
C37A-C38A	1.59(6)	C9A-H9A	0.95
C37A-H37A	1.00	C10A-C11A	1.41(5)
C38A-C39A	1.47(7)	C10A-C16A	1.500(14)
C38A-H38C	0.99	C11A-C12A	1.36(5)
C38A-H38D	0.99	C11A-H11A	0.95
C39A-C40A	1.50(4)	C12A-C19A	1.504(14)
C39A-C43A	1.60(7)	C13A-C15A	1.507(14)
C39A-H39A	1.00	C13A-C14A	1.508(14)
C40A-H40C	0.99	C13A-H13A	1.00
C40A-H40D	0.99	C14A-H14D	0.98
C41A-C44A	1.53(4)	C14A-H14E	0.98
C41A-H41C	0.99	C14A-H14F	0.98
C41A-H41D	0.99	C15A-H15D	0.98
C42A-C44A	1.56(6)	C15A-H15E	0.98

C15A-H15F	0.98	C28A-C29A	1.56(5)
C16A-C17A	1.499(14)	C28A-C33A	1.58(5)
C16A-C18A	1.504(14)	C28A-H28A	1.00
C16A-H16A	1.00	C29A-C30A	1.48(5)
C17A-H17D	0.98	C29A-H29C	0.99
C17A-H17E	0.98	C29A-H29D	0.99
C17A-H17F	0.98	C30A-C31A	1.46(6)
C18A-H18D	0.98	C30A-H30C	0.99
C18A-H18E	0.98	C30A-H30D	0.99
C18A-H18F	0.98	C31A-C32A	1.50(5)
C19A-C21A	1.505(14)	C31A-H31C	0.99
C19A-C20A	1.506(14)	C31A-H31D	0.99
C19A-H19A	1.00	C32A-C33A	1.51(5)
C20A-H20D	0.98	C32A-H32C	0.99
C20A-H20E	0.98	C32A-H32D	0.99
C20A-H20F	0.98	C33A-H33C	0.99
C21A-H21D	0.98	C33A-H33D	0.99
C21A-H21E	0.98	Os1B-C46B	1.83(2)
C21A-H21F	0.98	Os1B-C45B	1.86(2)
C22A-C27A	1.49(5)	Os1B-O1B	2.065(18)
C22A-C23A	1.55(5)	Os1B-P1B	2.424(9)
C22A-H22A	1.00	P1B-C1B	1.86(5)
C23A-C24A	1.50(5)	P1B-C22B	1.879(14)
C23A-H23C	0.99	P1B-C28B	1.884(14)
C23A-H23D	0.99	O1B-C34B	1.30(2)
C24A-C25A	1.46(6)	O2B-C34B	1.28(2)
C24A-H24C	0.99	O3B-C45B	1.156(15)
C24A-H24D	0.99	O4B-C46B	1.158(14)
C25A-C26A	1.58(6)	C34B-C35B	1.58(3)
C25A-H25C	0.99	C35B-C40B	1.34(3)
C25A-H25D	0.99	C35B-C36B	1.55(4)
C26A-C27A	1.54(6)	C35B-C41B	1.74(5)
C26A-H26C	0.99	C36B-C37B	1.52(4)
C26A-H26D	0.99	C36B-H36E	0.99
C27A-H27C	0.99	C36B-H36F	0.99
C27A-H27D	0.99	C37B-C38B	1.53(6)

C37B-C42B	1.63(6)	C9B-H9B	0.95
C37B-H37B	1.00	C10B-C11B	1.46(7)
C38B-C39B	1.48(6)	C10B-C16B	1.505(14)
C38B-H38E	0.99	C11B-C12B	1.41(6)
C38B-H38F	0.99	C11B-H11B	0.95
C39B-C43B	1.57(9)	C12B-C19B	1.502(14)
C39B-C40B	1.68(4)	C13B-C15B	1.502(14)
C39B-H39B	1.00	C13B-C14B	1.503(14)
C40B-H40E	0.99	C13B-H13B	1.00
C40B-H40F	0.99	C14B-H14G	0.98
C41B-C44B	1.50(6)	C14B-H14H	0.98
C41B-H41E	0.99	C14B-H14I	0.98
C41B-H41F	0.99	C15B-H15G	0.98
C42B-C44B	1.44(7)	C15B-H15H	0.98
C42B-H42C	0.99	C15B-H15I	0.98
C42B-H42D	0.99	C16B-C18B	1.503(14)
C43B-C44B	1.46(10)	C16B-C17B	1.508(14)
C43B-H43C	0.99	C16B-H16B	1.00
C43B-H43D	0.99	C17B-H17G	0.98
C44B-H44D	1.00	C17B-H17H	0.98
C1B-C6B	1.23(4)	C17B-H17I	0.98
C1B-C2B	1.60(5)	C18B-H18G	0.98
C2B-C3B	1.28(5)	C18B-H18H	0.98
C2B-H2B	0.95	C18B-H18I	0.98
C3B-C4B	1.39(6)	C19B-C20B	1.499(14)
C3B-H3B	0.95	C19B-C21B	1.502(14)
C4B-C5B	1.27(6)	C19B-H19B	1.00
C4B-H4B	0.95	C20B-H20G	0.98
C5B-C6B	1.57(4)	C20B-H20H	0.98
C5B-H5B	0.95	C20B-H20I	0.98
C6B-C7B	1.498(14)	C21B-H21G	0.98
C7B-C12B	1.40(5)	C21B-H21H	0.98
C7B-C8B	1.48(5)	C21B-H21I	0.98
C8B-C9B	1.42(6)	C22B-C23B	1.51(5)
C8B-C13B	1.501(14)	C22B-C27B	1.54(5)
C9B-C10B	1.34(6)	C22B-H22B	1.00

C23B-C24B	1.51(5)	C28B-H28B	1.00
C23B-H23E	0.99	C29B-C30B	1.43(5)
C23B-H23F	0.99	C29B-H29E	0.99
C24B-C25B	1.45(7)	C29B-H29F	0.99
C24B-H24E	0.99	C30B-C31B	1.46(6)
C24B-H24F	0.99	C30B-H30E	0.99
C25B-C26B	1.56(6)	C30B-H30F	0.99
C25B-H25E	0.99	C31B-C32B	1.55(6)
C25B-H25F	0.99	C31B-H31E	0.99
C26B-C27B	1.62(5)	C31B-H31F	0.99
C26B-H26E	0.99	C32B-C33B	1.49(5)
C26B-H26F	0.99	C32B-H32E	0.99
C27B-H27E	0.99	C32B-H32F	0.99
C27B-H27F	0.99	C33B-H33E	0.99
C28B-C29B	1.57(5)	C33B-H33F	0.99
C28B-C33B	1.60(5)		

C50-Os1-C51	92.5(6)	C1-P1-Os1	113.6(5)
C50-Os1-O1	90.3(5)	C22-P1-Os1	112.6(5)
C51-Os1-O1	176.1(5)	C34-O1-Os1	124.3(10)
C50-Os1-O2	171.9(5)	C42-O2-Os1	123.2(9)
C51-Os1-O2	93.8(5)	C2-C1-C6	118.4(14)
O1-Os1-O2	83.2(4)	C2-C1-P1	110.2(11)
C50-Os1-P1	98.1(4)	C6-C1-P1	131.3(12)
C51-Os1-P1	90.7(5)	C1-C2-C3	123.3(14)
O1-Os1-P1	91.6(3)	C1-C2-H2	118.3
O2-Os1-P1	86.9(3)	C3-C2-H2	118.3
C50-Os1-Os1#1	91.4(4)	C4-C3-C2	117.9(16)
C51-Os1-Os1#1	94.3(4)	C4-C3-H3	121.0
O1-Os1-Os1#1	82.9(3)	C2-C3-H3	121.0
O2-Os1-Os1#1	83.1(3)	C3-C4-C5	119.0(18)
P1-Os1-Os1#1	169.06(10)	C3-C4-H4	120.5
C28-P1-C1	109.6(7)	C5-C4-H4	120.5
C28-P1-C22	105.1(7)	C6-C5-C4	122.9(18)
C1-P1-C22	96.5(7)	C6-C5-H5	118.6
C28-P1-Os1	117.1(6)	C4-C5-H5	118.6

C5-C6-C1	118.0(16)	C13-C15-H15C	109.5
C5-C6-C7	114.1(14)	H15A-C15-H15C	109.5
C1-C6-C7	127.8(15)	H15B-C15-H15C	109.5
C8-C7-C12	121.4(16)	C17-C16-C18	120(3)
C8-C7-C6	120.6(16)	C17-C16-C10	120(3)
C12-C7-C6	117.3(15)	C18-C16-C10	113(2)
C7-C8-C9	118.0(17)	C17-C16-H16	99.3
C7-C8-C13	122.2(16)	C18-C16-H16	99.3
C9-C8-C13	119.8(16)	C10-C16-H16	99.3
C10-C9-C8	121.3(19)	C16-C17-H17A	109.5
C10-C9-H9	119.3	C16-C17-H17B	109.5
C8-C9-H9	119.3	H17A-C17-H17B	109.5
C9-C10-C11	120.1(19)	C16-C17-H17C	109.5
C9-C10-C16	123(2)	H17A-C17-H17C	109.5
C11-C10-C16	117(2)	H17B-C17-H17C	109.5
C10-C11-C12	120.7(19)	C16-C18-H18A	109.5
C10-C11-H11	119.6	C16-C18-H18B	109.5
C12-C11-H11	119.6	H18A-C18-H18B	109.5
C11-C12-C7	118.4(17)	C16-C18-H18C	109.5
C11-C12-C19	119.8(16)	H18A-C18-H18C	109.5
C7-C12-C19	121.8(15)	H18B-C18-H18C	109.5
C8-C13-C15	111.7(18)	C12-C19-C21	114.4(15)
C8-C13-C14	109.6(16)	C12-C19-C20	107.3(15)
C15-C13-C14	109.9(17)	C21-C19-C20	110.1(15)
C8-C13-H13	108.5	C12-C19-H19	108.3
C15-C13-H13	108.5	C21-C19-H19	108.3
C14-C13-H13	108.5	C20-C19-H19	108.3
C13-C14-H14A	109.5	C19-C20-H20A	109.5
C13-C14-H14B	109.5	C19-C20-H20B	109.5
H14A-C14-H14B	109.5	H20A-C20-H20B	109.5
C13-C14-H14C	109.5	C19-C20-H20C	109.5
H14A-C14-H14C	109.5	H20A-C20-H20C	109.5
H14B-C14-H14C	109.5	H20B-C20-H20C	109.5
C13-C15-H15A	109.5	C19-C21-H21A	109.5
C13-C15-H15B	109.5	C19-C21-H21B	109.5
H15A-C15-H15B	109.5	H21A-C21-H21B	109.5

C19-C21-H21C	109.5	C22-C27-H27B	109.4
H21A-C21-H21C	109.5	C26-C27-H27B	109.4
H21B-C21-H21C	109.5	H27A-C27-H27B	108.0
C27-C22-C23	112.4(13)	C29-C28-C33	105.9(13)
C27-C22-P1	117.1(10)	C29-C28-P1	111.6(11)
C23-C22-P1	111.5(10)	C33-C28-P1	116.0(11)
C27-C22-H22	104.8	C29-C28-H28	107.6
C23-C22-H22	104.8	C33-C28-H28	107.6
P1-C22-H22	104.8	P1-C28-H28	107.6
C22-C23-C24	111.3(14)	C30-C29-C28	113.9(14)
C22-C23-H23A	109.4	C30-C29-H29A	108.8
C24-C23-H23A	109.4	C28-C29-H29A	108.8
C22-C23-H23B	109.4	C30-C29-H29B	108.8
C24-C23-H23B	109.4	C28-C29-H29B	108.8
H23A-C23-H23B	108.0	H29A-C29-H29B	107.7
C25-C24-C23	110.2(16)	C29-C30-C31	107.6(15)
C25-C24-H24A	109.6	C29-C30-H30A	110.2
C23-C24-H24A	109.6	C31-C30-H30A	110.2
C25-C24-H24B	109.6	C29-C30-H30B	110.2
C23-C24-H24B	109.6	C31-C30-H30B	110.2
H24A-C24-H24B	108.1	H30A-C30-H30B	108.5
C26-C25-C24	111.7(16)	C32-C31-C30	111.9(18)
C26-C25-H25A	109.3	C32-C31-H31A	109.2
C24-C25-H25A	109.3	C30-C31-H31A	109.2
C26-C25-H25B	109.3	C32-C31-H31B	109.2
C24-C25-H25B	109.3	C30-C31-H31B	109.2
H25A-C25-H25B	107.9	H31A-C31-H31B	107.9
C25-C26-C27	113.0(13)	C31-C32-C33	110.9(15)
C25-C26-H26A	109.0	C31-C32-H32A	109.5
C27-C26-H26A	109.0	C33-C32-H32A	109.5
C25-C26-H26B	109.0	C31-C32-H32B	109.5
C27-C26-H26B	109.0	C33-C32-H32B	109.5
H26A-C26-H26B	107.8	H32A-C32-H32B	108.0
C22-C27-C26	111.0(15)	C28-C33-C32	112.9(14)
C22-C27-H27A	109.4	C28-C33-H33A	109.0
C26-C27-H27A	109.4	C32-C33-H33A	109.0

C28-C33-H33B	109.0	C35-C40-C39	109.0(15)
C32-C33-H33B	109.0	C35-C40-H40A	109.9
H33A-C33-H33B	107.8	C39-C40-H40A	109.9
O1#1-C34-O1	125.6(17)	C35-C40-H40B	109.9
O1#1-C34-C35	117.1(8)	C39-C40-H40B	109.9
O1-C34-C35	117.1(8)	H40A-C40-H40B	108.3
C40-C35-C34	106.8(15)	C37-C41-C37#1	109.3(17)
C40-C35-C36	110.6(11)	C37-C41-H41A	109.8
C34-C35-C36	109.8(11)	C37#1-C41-H41A	109.8
C40-C35-C36#1	110.6(11)	C37-C41-H41B	109.8
C34-C35-C36#1	109.8(11)	C37#1-C41-H41B	109.8
C36-C35-C36#1	109.1(16)	H41A-C41-H41B	108.3
C37-C36-C35	108.7(13)	O2#1-C42-O2	127.5(16)
C37-C36-H36A	109.9	O2#1-C42-C43	116.2(8)
C35-C36-H36A	109.9	O2-C42-C43	116.2(8)
C37-C36-H36B	109.9	C48-C43-C42	105.7(16)
C35-C36-H36B	109.9	C48-C43-C44#1	110.7(11)
H36A-C36-H36B	108.3	C42-C43-C44#1	109.4(10)
C36-C37-C38	109.3(13)	C48-C43-C44	110.7(11)
C36-C37-C41	110.4(14)	C42-C43-C44	109.4(10)
C38-C37-C41	108.3(15)	C44#1-C43-C44	110.7(16)
C36-C37-H37	109.6	C43-C44-C45	108.4(12)
C38-C37-H37	109.6	C43-C44-H44A	110.0
C41-C37-H37	109.6	C45-C44-H44A	110.0
C37-C38-C39	111.7(14)	C43-C44-H44B	110.0
C37-C38-H38A	109.3	C45-C44-H44B	110.0
C39-C38-H38A	109.3	H44A-C44-H44B	108.4
C37-C38-H38B	109.3	C49-C45-C46	110.5(16)
C39-C38-H38B	109.3	C49-C45-C44	107.5(15)
H38A-C38-H38B	107.9	C46-C45-C44	106.4(14)
C38-C39-C38#1	107.6(19)	C49-C45-H45	110.8
C38-C39-C40	108.5(12)	C46-C45-H45	110.8
C38#1-C39-C40	108.5(12)	C44-C45-H45	110.8
C38-C39-H39	110.7	C45-C46-C47	111.6(16)
C38#1-C39-H39	110.7	C45-C46-H46A	109.3
C40-C39-H39	110.7	C47-C46-H46A	109.3

C45-C46-H46B	109.3	O2B-Os1A-Os1B	81.2(4)
C47-C46-H46B	109.3	P1A-Os1A-Os1B	167.4(4)
H46A-C46-H46B	108.0	C1A-P1A-C28A	108.2(17)
C46#1-C47-C46	111(2)	C1A-P1A-C22A	96.9(18)
C46#1-C47-C48	108.3(14)	C28A-P1A-C22A	106.2(19)
C46-C47-C48	108.3(14)	C1A-P1A-Os1A	117.5(10)
C46#1-C47-H47	109.8	C28A-P1A-Os1A	116.5(14)
C46-C47-H47	109.8	C22A-P1A-Os1A	109.1(13)
C48-C47-H47	109.8	C34A-O1A-Os1A	124.9(15)
C43-C48-C47	107.6(19)	C34A-O2A-Os1B	125.4(15)
C43-C48-H48A	110.2	O3A-C45A-Os1A	177(4)
C47-C48-H48A	110.2	O4A-C46A-Os1A	173(4)
C43-C48-H48B	110.2	O2A-C34A-O1A	123(3)
C47-C48-H48B	110.2	O2A-C34A-C35A	118.8(15)
H48A-C48-H48B	108.5	O1A-C34A-C35A	117.8(14)
C45-C49-C45#1	114(2)	C36A-C35A-C41A	115(3)
C45-C49-H49A	108.8	C36A-C35A-C34A	117(2)
C45#1-C49-H49A	108.8	C41A-C35A-C34A	106.9(16)
C45-C49-H49B	108.8	C36A-C35A-C40A	112.2(19)
C45#1-C49-H49B	108.8	C41A-C35A-C40A	102.6(19)
H49A-C49-H49B	107.7	C34A-C35A-C40A	101.2(17)
O3-C50-Os1	175.1(14)	C35A-C36A-C37A	113(2)
O4-C51-Os1	176.4(13)	C35A-C36A-H36C	109.0
C45A-Os1A-C46A	93.3(17)	C37A-C36A-H36C	109.0
C45A-Os1A-O1A	87.6(17)	C35A-C36A-H36D	109.0
C46A-Os1A-O1A	176.5(9)	C37A-C36A-H36D	109.0
C45A-Os1A-O2B	175.6(15)	H36C-C36A-H36D	107.8
C46A-Os1A-O2B	90.2(18)	C42A-C37A-C38A	111(3)
O1A-Os1A-O2B	88.9(15)	C42A-C37A-C36A	108(3)
C45A-Os1A-P1A	95.3(7)	C38A-C37A-C36A	102(2)
C46A-Os1A-P1A	92.1(7)	C42A-C37A-H37A	112.0
O1A-Os1A-P1A	91.3(5)	C38A-C37A-H37A	112.0
O2B-Os1A-P1A	87.3(5)	C36A-C37A-H37A	112.0
C45A-Os1A-Os1B	95.9(8)	C39A-C38A-C37A	110(3)
C46A-Os1A-Os1B	93.0(7)	C39A-C38A-H38C	109.6
O1A-Os1A-Os1B	83.5(4)	C37A-C38A-H38C	109.6

C39A-C38A-H38D	109.6	C43A-C44A-H44C	109.5
C37A-C38A-H38D	109.6	C41A-C44A-H44C	109.5
H38C-C38A-H38D	108.1	C42A-C44A-H44C	109.5
C38A-C39A-C40A	115(4)	C6A-C1A-C2A	115(4)
C38A-C39A-C43A	110(2)	C6A-C1A-P1A	134(3)
C40A-C39A-C43A	108(4)	C2A-C1A-P1A	110(2)
C38A-C39A-H39A	107.7	C3A-C2A-C1A	122(4)
C40A-C39A-H39A	107.7	C3A-C2A-H2A	119.1
C43A-C39A-H39A	107.7	C1A-C2A-H2A	119.1
C39A-C40A-C35A	105.1(16)	C2A-C3A-C4A	122(4)
C39A-C40A-H40C	110.7	C2A-C3A-H3A	118.9
C35A-C40A-H40C	110.7	C4A-C3A-H3A	118.9
C39A-C40A-H40D	110.7	C5A-C4A-C3A	120(4)
C35A-C40A-H40D	110.7	C5A-C4A-H4A	120.2
H40C-C40A-H40D	108.8	C3A-C4A-H4A	120.2
C44A-C41A-C35A	109(3)	C4A-C5A-C6A	122(4)
C44A-C41A-H41C	109.8	C4A-C5A-H5A	119.0
C35A-C41A-H41C	109.8	C6A-C5A-H5A	119.0
C44A-C41A-H41D	109.8	C1A-C6A-C7A	129(3)
C35A-C41A-H41D	109.8	C1A-C6A-C5A	120(3)
H41C-C41A-H41D	108.3	C7A-C6A-C5A	111(2)
C37A-C42A-C44A	112(2)	C12A-C7A-C8A	119(2)
C37A-C42A-H42A	109.1	C12A-C7A-C6A	123(3)
C44A-C42A-H42A	109.1	C8A-C7A-C6A	118(3)
C37A-C42A-H42B	109.1	C9A-C8A-C7A	119(3)
C44A-C42A-H42B	109.1	C9A-C8A-C13A	117(3)
H42A-C42A-H42B	107.9	C7A-C8A-C13A	124(2)
C44A-C43A-C39A	107(3)	C8A-C9A-C10A	124(3)
C44A-C43A-H43A	110.3	C8A-C9A-H9A	118.2
C39A-C43A-H43A	110.3	C10A-C9A-H9A	118.2
C44A-C43A-H43B	110.3	C11A-C10A-C9A	112(3)
C39A-C43A-H43B	110.3	C11A-C10A-C16A	123(3)
H43A-C43A-H43B	108.5	C9A-C10A-C16A	125(3)
C43A-C44A-C41A	111(3)	C12A-C11A-C10A	128(3)
C43A-C44A-C42A	108(4)	C12A-C11A-H11A	115.8
C41A-C44A-C42A	109(3)	C10A-C11A-H11A	115.8

C11A-C12A-C7A	118(3)	C16A-C18A-H18F	109.5
C11A-C12A-C19A	119(3)	H18D-C18A-H18F	109.5
C7A-C12A-C19A	123(2)	H18E-C18A-H18F	109.5
C8A-C13A-C15A	112.8(15)	C21A-C19A-C12A	113.1(15)
C8A-C13A-C14A	112.6(14)	C21A-C19A-C20A	113(3)
C15A-C13A-C14A	111(2)	C12A-C19A-C20A	112.8(15)
C8A-C13A-H13A	106.5	C21A-C19A-H19A	105.7
C15A-C13A-H13A	106.5	C12A-C19A-H19A	105.7
C14A-C13A-H13A	106.5	C20A-C19A-H19A	105.7
C13A-C14A-H14D	109.5	C19A-C20A-H20D	109.5
C13A-C14A-H14E	109.5	C19A-C20A-H20E	109.5
H14D-C14A-H14E	109.5	H20D-C20A-H20E	109.5
C13A-C14A-H14F	109.5	C19A-C20A-H20F	109.5
H14D-C14A-H14F	109.5	H20D-C20A-H20F	109.5
H14E-C14A-H14F	109.5	H20E-C20A-H20F	109.5
C13A-C15A-H15D	109.5	C19A-C21A-H21D	109.5
C13A-C15A-H15E	109.5	C19A-C21A-H21E	109.5
H15D-C15A-H15E	109.5	H21D-C21A-H21E	109.5
C13A-C15A-H15F	109.5	C19A-C21A-H21F	109.5
H15D-C15A-H15F	109.5	H21D-C21A-H21F	109.5
H15E-C15A-H15F	109.5	H21E-C21A-H21F	109.5
C17A-C16A-C10A	113.9(15)	C27A-C22A-C23A	110(3)
C17A-C16A-C18A	113(3)	C27A-C22A-P1A	118(3)
C10A-C16A-C18A	113.4(15)	C23A-C22A-P1A	115(2)
C17A-C16A-H16A	105.0	C27A-C22A-H22A	104.1
C10A-C16A-H16A	105.0	C23A-C22A-H22A	104.1
C18A-C16A-H16A	105.0	P1A-C22A-H22A	104.1
C16A-C17A-H17D	109.5	C24A-C23A-C22A	113(3)
C16A-C17A-H17E	109.5	C24A-C23A-H23C	109.0
H17D-C17A-H17E	109.5	C22A-C23A-H23C	109.0
C16A-C17A-H17F	109.5	C24A-C23A-H23D	109.0
H17D-C17A-H17F	109.5	C22A-C23A-H23D	109.0
H17E-C17A-H17F	109.5	H23C-C23A-H23D	107.8
C16A-C18A-H18D	109.5	C25A-C24A-C23A	115(4)
C16A-C18A-H18E	109.5	C25A-C24A-H24C	108.5
H18D-C18A-H18E	109.5	C23A-C24A-H24C	108.5

C25A-C24A-H24D	108.5	C31A-C30A-H30D	109.3
C23A-C24A-H24D	108.5	C29A-C30A-H30D	109.3
H24C-C24A-H24D	107.5	H30C-C30A-H30D	107.9
C24A-C25A-C26A	111(4)	C30A-C31A-C32A	111(4)
C24A-C25A-H25C	109.3	C30A-C31A-H31C	109.5
C26A-C25A-H25C	109.3	C32A-C31A-H31C	109.5
C24A-C25A-H25D	109.3	C30A-C31A-H31D	109.5
C26A-C25A-H25D	109.3	C32A-C31A-H31D	109.5
H25C-C25A-H25D	108.0	H31C-C31A-H31D	108.1
C27A-C26A-C25A	110(4)	C31A-C32A-C33A	116(3)
C27A-C26A-H26C	109.8	C31A-C32A-H32C	108.4
C25A-C26A-H26C	109.8	C33A-C32A-H32C	108.4
C27A-C26A-H26D	109.8	C31A-C32A-H32D	108.4
C25A-C26A-H26D	109.8	C33A-C32A-H32D	108.4
H26C-C26A-H26D	108.2	H32C-C32A-H32D	107.5
C22A-C27A-C26A	115(4)	C32A-C33A-C28A	108(3)
C22A-C27A-H27C	108.5	C32A-C33A-H33C	110.1
C26A-C27A-H27C	108.5	C28A-C33A-H33C	110.1
C22A-C27A-H27D	108.5	C32A-C33A-H33D	110.1
C26A-C27A-H27D	108.5	C28A-C33A-H33D	110.1
H27C-C27A-H27D	107.5	H33C-C33A-H33D	108.4
C29A-C28A-C33A	110(3)	C46B-Os1B-C45B	92.0(17)
C29A-C28A-P1A	114(2)	C46B-Os1B-O1B	96(2)
C33A-C28A-P1A	113(2)	C45B-Os1B-O1B	171.9(18)
C29A-C28A-H28A	106.2	C46B-Os1B-O2A	171.2(18)
C33A-C28A-H28A	106.2	C45B-Os1B-O2A	96.0(18)
P1A-C28A-H28A	106.2	O1B-Os1B-O2A	76.2(15)
C30A-C29A-C28A	109(3)	C46B-Os1B-P1B	92.9(7)
C30A-C29A-H29C	109.9	C45B-Os1B-P1B	92.8(7)
C28A-C29A-H29C	109.9	O1B-Os1B-P1B	89.6(5)
C30A-C29A-H29D	109.9	O2A-Os1B-P1B	90.1(5)
C28A-C29A-H29D	109.9	C46B-Os1B-Os1A	93.9(7)
H29C-C29A-H29D	108.3	C45B-Os1B-Os1A	93.2(7)
C31A-C30A-C29A	112(3)	O1B-Os1B-Os1A	83.6(4)
C31A-C30A-H30C	109.3	O2A-Os1B-Os1A	82.3(4)
C29A-C30A-H30C	109.3	P1B-Os1B-Os1A	170.8(4)

C1B-P1B-C22B	96.3(17)	H38E-C38B-H38F	107.8
C1B-P1B-C28B	108.2(18)	C38B-C39B-C43B	111(4)
C22B-P1B-C28B	106(2)	C38B-C39B-C40B	105(3)
C1B-P1B-Os1B	113.8(10)	C43B-C39B-C40B	101(3)
C22B-P1B-Os1B	115.3(13)	C38B-C39B-H39B	112.7
C28B-P1B-Os1B	115.4(14)	C43B-C39B-H39B	112.7
C34B-O1B-Os1B	125.1(15)	C40B-C39B-H39B	112.7
C34B-O2B-Os1A	125.9(15)	C35B-C40B-C39B	113(3)
O3B-C45B-Os1B	166(4)	C35B-C40B-H40E	109.1
O4B-C46B-Os1B	178(4)	C39B-C40B-H40E	109.1
O2B-C34B-O1B	123(3)	C35B-C40B-H40F	109.1
O2B-C34B-C35B	119.9(15)	C39B-C40B-H40F	109.1
O1B-C34B-C35B	117.4(14)	H40E-C40B-H40F	107.8
C40B-C35B-C36B	121(2)	C44B-C41B-C35B	101(3)
C40B-C35B-C34B	119(2)	C44B-C41B-H41E	111.5
C36B-C35B-C34B	106(2)	C35B-C41B-H41E	111.5
C40B-C35B-C41B	109(3)	C44B-C41B-H41F	111.5
C36B-C35B-C41B	99(2)	C35B-C41B-H41F	111.5
C34B-C35B-C41B	98.3(19)	H41E-C41B-H41F	109.3
C37B-C36B-C35B	110(2)	C44B-C42B-C37B	110(3)
C37B-C36B-H36E	109.6	C44B-C42B-H42C	109.6
C35B-C36B-H36E	109.6	C37B-C42B-H42C	109.6
C37B-C36B-H36F	109.6	C44B-C42B-H42D	109.6
C35B-C36B-H36F	109.6	C37B-C42B-H42D	109.6
H36E-C36B-H36F	108.2	H42C-C42B-H42D	108.1
C36B-C37B-C38B	111(3)	C44B-C43B-C39B	112(3)
C36B-C37B-C42B	106(3)	C44B-C43B-H43C	109.2
C38B-C37B-C42B	105(3)	C39B-C43B-H43C	109.2
C36B-C37B-H37B	111.6	C44B-C43B-H43D	109.2
C38B-C37B-H37B	111.6	C39B-C43B-H43D	109.2
C42B-C37B-H37B	111.6	H43C-C43B-H43D	107.9
C39B-C38B-C37B	113(3)	C42B-C44B-C43B	104(5)
C39B-C38B-H38E	109.0	C42B-C44B-C41B	117(5)
C37B-C38B-H38E	109.0	C43B-C44B-C41B	116(5)
C39B-C38B-H38F	109.0	C42B-C44B-H44D	106.3
C37B-C38B-H38F	109.0	C43B-C44B-H44D	106.3

C41B-C44B-H44D	106.3	C11B-C12B-C19B	118(3)
C6B-C1B-C2B	119(4)	C8B-C13B-C15B	113.4(16)
C6B-C1B-P1B	132(3)	C8B-C13B-C14B	113.3(16)
C2B-C1B-P1B	109(2)	C15B-C13B-C14B	111(3)
C3B-C2B-C1B	118(4)	C8B-C13B-H13B	106.2
C3B-C2B-H2B	120.8	C15B-C13B-H13B	106.2
C1B-C2B-H2B	120.8	C14B-C13B-H13B	106.2
C2B-C3B-C4B	122(5)	C13B-C14B-H14G	109.5
C2B-C3B-H3B	118.8	C13B-C14B-H14H	109.5
C4B-C3B-H3B	118.8	H14G-C14B-H14H	109.5
C5B-C4B-C3B	121(5)	C13B-C14B-H14I	109.5
C5B-C4B-H4B	119.3	H14G-C14B-H14I	109.5
C3B-C4B-H4B	119.3	H14H-C14B-H14I	109.5
C4B-C5B-C6B	121(4)	C13B-C15B-H15G	109.5
C4B-C5B-H5B	119.6	C13B-C15B-H15H	109.5
C6B-C5B-H5B	119.6	H15G-C15B-H15H	109.5
C1B-C6B-C7B	132(3)	C13B-C15B-H15I	109.5
C1B-C6B-C5B	118(3)	H15G-C15B-H15I	109.5
C7B-C6B-C5B	109(2)	H15H-C15B-H15I	109.5
C12B-C7B-C8B	119(3)	C18B-C16B-C10B	113.2(16)
C12B-C7B-C6B	124(3)	C18B-C16B-C17B	109(4)
C8B-C7B-C6B	118(3)	C10B-C16B-C17B	112.7(15)
C9B-C8B-C7B	117(3)	C18B-C16B-H16B	107.1
C9B-C8B-C13B	119(3)	C10B-C16B-H16B	107.1
C7B-C8B-C13B	124(2)	C17B-C16B-H16B	107.1
C10B-C9B-C8B	127(4)	C16B-C17B-H17G	109.5
C10B-C9B-H9B	116.7	C16B-C17B-H17H	109.5
C8B-C9B-H9B	116.7	H17G-C17B-H17H	109.5
C9B-C10B-C11B	115(4)	C16B-C17B-H17I	109.5
C9B-C10B-C16B	124(4)	H17G-C17B-H17I	109.5
C11B-C10B-C16B	121(4)	H17H-C17B-H17I	109.5
C12B-C11B-C10B	123(4)	C16B-C18B-H18G	109.5
C12B-C11B-H11B	118.5	C16B-C18B-H18H	109.5
C10B-C11B-H11B	118.5	H18G-C18B-H18H	109.5
C7B-C12B-C11B	120(3)	C16B-C18B-H18I	109.5
C7B-C12B-C19B	122(2)	H18G-C18B-H18I	109.5

H18H-C18B-H18I	109.5	H24E-C24B-H24F	107.6
C20B-C19B-C21B	112(3)	C24B-C25B-C26B	110(4)
C20B-C19B-C12B	113.5(16)	C24B-C25B-H25E	109.7
C21B-C19B-C12B	113.4(16)	C26B-C25B-H25E	109.7
C20B-C19B-H19B	105.6	C24B-C25B-H25F	109.7
C21B-C19B-H19B	105.6	C26B-C25B-H25F	109.7
C12B-C19B-H19B	105.6	H25E-C25B-H25F	108.2
C19B-C20B-H20G	109.5	C25B-C26B-C27B	108(4)
C19B-C20B-H20H	109.5	C25B-C26B-H26E	110.2
H20G-C20B-H20H	109.5	C27B-C26B-H26E	110.2
C19B-C20B-H20I	109.5	C25B-C26B-H26F	110.2
H20G-C20B-H20I	109.5	C27B-C26B-H26F	110.2
H20H-C20B-H20I	109.5	H26E-C26B-H26F	108.5
C19B-C21B-H21G	109.5	C22B-C27B-C26B	112(3)
C19B-C21B-H21H	109.5	C22B-C27B-H27E	109.3
H21G-C21B-H21H	109.5	C26B-C27B-H27E	109.3
C19B-C21B-H21I	109.5	C22B-C27B-H27F	109.3
H21G-C21B-H21I	109.5	C26B-C27B-H27F	109.3
H21H-C21B-H21I	109.5	H27E-C27B-H27F	107.9
C23B-C22B-C27B	110(3)	C29B-C28B-C33B	110(3)
C23B-C22B-P1B	117(2)	C29B-C28B-P1B	113(2)
C27B-C22B-P1B	115(3)	C33B-C28B-P1B	112(2)
C23B-C22B-H22B	104.3	C29B-C28B-H28B	106.9
C27B-C22B-H22B	104.3	C33B-C28B-H28B	106.9
P1B-C22B-H22B	104.3	P1B-C28B-H28B	106.9
C24B-C23B-C22B	113(3)	C30B-C29B-C28B	108(3)
C24B-C23B-H23E	108.9	C30B-C29B-H29E	110.1
C22B-C23B-H23E	108.9	C28B-C29B-H29E	110.1
C24B-C23B-H23F	108.9	C30B-C29B-H29F	110.1
C22B-C23B-H23F	108.9	C28B-C29B-H29F	110.1
H23E-C23B-H23F	107.7	H29E-C29B-H29F	108.4
C25B-C24B-C23B	114(4)	C29B-C30B-C31B	121(4)
C25B-C24B-H24E	108.7	C29B-C30B-H30E	107.1
C23B-C24B-H24E	108.7	C31B-C30B-H30E	107.1
C25B-C24B-H24F	108.7	C29B-C30B-H30F	107.1
C23B-C24B-H24F	108.7	C31B-C30B-H30F	107.1

H30E-C30B-H30F	106.8	C33B-C32B-H32F	108.2
C30B-C31B-C32B	106(4)	C31B-C32B-H32F	108.2
C30B-C31B-H31E	110.6	H32E-C32B-H32F	107.4
C32B-C31B-H31E	110.6	C32B-C33B-C28B	109(3)
C30B-C31B-H31F	110.6	C32B-C33B-H33E	109.9
C32B-C31B-H31F	110.6	C28B-C33B-H33E	109.9
H31E-C31B-H31F	108.8	C32B-C33B-H33F	109.9
C33B-C32B-C31B	116(4)	C28B-C33B-H33F	109.9
C33B-C32B-H32E	108.2	H33E-C33B-H33F	108.3
C31B-C32B-H32E	108.2		

Symmetry transformations used to generate equivalent atoms:

#1 -x,y,z

Table 10. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 1. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Os1	21(1)	7(1)	12(1)	1(1)	1(1)	0(1)
P1	23(2)	18(1)	22(2)	4(1)	7(1)	6(1)
O1	26(4)	25(5)	18(4)	8(3)	-2(3)	-1(3)
O2	23(4)	20(4)	18(5)	-12(3)	-6(3)	1(3)
O3	32(6)	9(4)	29(6)	-7(3)	3(4)	-4(3)
O4	41(7)	26(5)	19(4)	12(4)	6(4)	6(4)
C1	20(5)	26(5)	13(4)	1(4)	2(4)	0(4)
C2	19(5)	26(6)	19(5)	-1(4)	1(4)	4(4)
C3	30(6)	32(6)	20(5)	-1(4)	-1(4)	4(5)
C4	36(7)	45(7)	28(6)	4(5)	1(5)	-3(6)
C5	28(6)	35(7)	29(5)	11(5)	2(4)	-1(5)
C6	18(5)	28(5)	29(5)	10(4)	1(4)	-3(4)
C7	29(5)	35(5)	35(6)	5(5)	-4(5)	-4(4)
C8	26(5)	42(6)	38(7)	13(5)	-2(5)	-7(4)
C9	35(6)	57(6)	49(9)	7(6)	-7(6)	-14(5)
C10	41(6)	56(6)	50(7)	3(7)	0(6)	-13(5)
C11	41(6)	45(7)	50(8)	-4(6)	0(5)	-12(5)
C12	34(6)	34(5)	38(7)	3(5)	-1(5)	-4(4)
C13	27(6)	51(7)	61(8)	9(6)	-2(6)	-4(5)
C14	39(9)	64(12)	79(12)	20(9)	16(9)	3(8)
C15	24(8)	64(10)	71(12)	25(8)	1(7)	4(6)
C16	72(9)	87(10)	80(10)	-19(8)	-1(7)	-30(8)
C17	82(11)	106(19)	130(20)	-43(16)	7(10)	-33(10)
C18	65(12)	46(10)	61(10)	1(8)	-14(8)	-9(8)
C19	35(6)	26(6)	40(7)	0(5)	-6(5)	-2(5)
C20	53(10)	37(8)	43(8)	4(6)	-4(7)	-3(7)
C21	50(9)	23(7)	47(8)	5(6)	-1(7)	5(6)
C22	30(6)	22(5)	19(6)	0(4)	1(4)	-1(4)
C23	28(6)	30(6)	34(7)	-1(5)	0(5)	3(4)
C24	40(7)	37(6)	41(7)	-5(5)	6(6)	8(5)
C25	42(6)	27(6)	47(9)	-5(5)	0(6)	9(5)

C26	40(6)	18(5)	44(8)	-1(6)	-4(6)	3(4)
C27	27(6)	19(5)	27(7)	4(4)	0(5)	2(4)
C28	21(6)	29(5)	21(5)	1(4)	-2(4)	1(4)
C29	27(6)	25(5)	28(6)	-2(5)	2(5)	1(4)
C30	44(8)	25(6)	38(7)	-6(5)	-8(6)	-4(5)
C31	39(8)	33(6)	34(7)	-3(5)	-6(6)	0(6)
C32	31(7)	32(6)	28(6)	4(5)	-8(5)	1(5)
C33	34(7)	31(6)	33(6)	1(5)	-4(5)	1(5)
C34	22(6)	11(6)	8(5)	2(5)	0	0
C35	23(7)	15(6)	15(6)	5(5)	0	0
C36	32(6)	16(5)	20(6)	2(4)	3(4)	-7(4)
C37	30(6)	22(5)	22(6)	6(4)	3(4)	-5(4)
C38	43(7)	35(6)	20(6)	1(5)	9(5)	-3(5)
C39	37(8)	18(7)	17(6)	-4(5)	0	0
C40	27(8)	18(6)	18(6)	-2(5)	0	0
C41	40(8)	26(7)	26(8)	5(6)	0	0
C42	13(5)	22(6)	10(6)	-8(5)	0	0
C43	1(5)	27(6)	15(6)	-8(5)	0	0
C44	20(6)	29(6)	23(6)	-9(5)	-5(4)	-1(4)
C45	34(6)	34(6)	19(5)	-3(5)	-2(4)	-4(4)
C46	40(7)	34(7)	32(6)	3(6)	-2(5)	-1(5)
C47	42(9)	37(7)	30(8)	2(6)	0	0
C48	44(9)	35(7)	29(7)	1(6)	0	0
C49	39(8)	46(10)	33(8)	3(7)	0	0
C50	18(6)	22(4)	19(5)	7(4)	4(4)	1(4)
C51	26(6)	17(5)	14(4)	-7(4)	-3(4)	-7(4)
Os1A	16(2)	131(3)	40(2)	2(2)	-2(1)	1(2)
P1A	32(4)	46(4)	43(4)	-4(4)	-1(3)	-4(4)
O1A	30(7)	86(10)	43(7)	-3(7)	2(6)	8(6)
O2A	34(7)	90(10)	42(8)	-4(7)	-2(6)	-5(7)
O3A	6(10)	102(11)	51(14)	-3(10)	6(10)	6(8)
C45A	17(8)	100(8)	41(9)	2(6)	-1(7)	6(6)
O4A	3(9)	108(16)	54(11)	8(11)	-3(8)	-12(10)
C46A	28(9)	98(9)	49(7)	0(7)	0(6)	-4(7)
C34A	34(6)	86(8)	50(6)	-1(6)	1(6)	2(6)
C35A	44(7)	80(7)	54(6)	1(5)	1(6)	-1(6)

C36A	47(8)	79(9)	56(8)	3(7)	1(7)	0(7)
C37A	54(8)	80(8)	57(8)	4(6)	0(7)	-2(7)
C38A	49(8)	77(9)	56(9)	6(7)	0(7)	-1(7)
C39A	49(8)	80(8)	55(7)	4(6)	1(7)	1(7)
C40A	44(9)	75(8)	54(7)	2(6)	2(7)	1(7)
C41A	47(8)	79(9)	56(8)	1(7)	0(6)	0(7)
C42A	54(9)	82(9)	58(9)	4(7)	2(7)	0(7)
C43A	49(8)	79(9)	56(9)	4(7)	1(7)	1(7)
C44A	53(8)	81(8)	56(8)	3(7)	1(6)	2(7)
C1A	32(7)	44(7)	41(6)	-2(5)	0(5)	-1(6)
C2A	38(8)	47(9)	44(7)	-5(6)	-1(6)	0(7)
C3A	38(9)	48(9)	43(8)	-4(7)	0(6)	2(7)
C4A	37(8)	49(9)	42(8)	1(7)	-2(6)	3(7)
C5A	36(8)	50(8)	40(7)	0(6)	0(6)	2(7)
C6A	31(7)	43(7)	38(7)	1(5)	-2(5)	0(6)
C7A	29(6)	39(6)	35(7)	2(5)	3(5)	-3(5)
C8A	29(6)	40(6)	35(7)	3(6)	2(6)	-2(5)
C9A	31(7)	40(7)	40(8)	2(6)	-1(7)	-1(6)
C10A	34(7)	41(7)	43(8)	4(6)	0(6)	1(5)
C11A	34(7)	39(7)	44(8)	1(7)	0(6)	3(6)
C12A	34(6)	41(7)	40(7)	2(6)	4(6)	0(5)
C13A	32(8)	38(8)	39(8)	3(6)	1(7)	-1(6)
C14A	32(11)	51(14)	39(11)	4(10)	1(9)	-1(10)
C15A	31(12)	35(12)	44(11)	4(9)	1(9)	0(10)
C16A	39(8)	41(8)	48(9)	3(7)	-5(7)	2(6)
C17A	39(10)	50(15)	61(15)	2(13)	-7(9)	5(8)
C18A	46(13)	51(15)	51(11)	0(10)	-1(10)	3(12)
C19A	37(8)	52(9)	46(9)	3(8)	2(7)	3(7)
C20A	47(15)	64(16)	49(11)	4(11)	6(10)	7(13)
C21A	41(12)	56(15)	47(12)	8(11)	5(10)	13(11)
C22A	39(7)	47(6)	47(8)	-2(6)	-1(6)	-5(5)
C23A	39(8)	42(8)	49(9)	0(7)	1(7)	-2(6)
C24A	41(8)	42(8)	49(9)	-1(7)	3(7)	-1(7)
C25A	45(9)	44(8)	49(9)	2(7)	2(7)	-3(7)
C26A	43(9)	47(8)	50(10)	2(7)	0(7)	-5(7)
C27A	43(8)	48(8)	51(9)	0(7)	0(7)	-5(6)

C28A	33(7)	39(7)	40(7)	0(6)	-3(6)	-3(6)
C29A	39(8)	42(8)	46(8)	-3(7)	-2(7)	0(7)
C30A	35(9)	35(8)	42(8)	-3(7)	0(7)	1(7)
C31A	33(9)	36(8)	44(8)	-4(7)	-1(7)	1(7)
C32A	32(8)	34(8)	39(8)	-2(7)	-2(7)	0(7)
C33A	32(8)	37(8)	41(8)	-1(6)	-1(7)	-1(7)
Os1B	22(2)	166(4)	28(2)	5(2)	-1(1)	-3(2)
P1B	22(4)	61(5)	26(4)	1(4)	4(3)	5(4)
O1B	25(7)	104(8)	47(9)	3(7)	0(6)	-1(6)
O2B	26(7)	95(8)	47(9)	2(7)	0(6)	-4(6)
O3B	70(20)	137(12)	74(18)	-5(11)	12(16)	-13(11)
C45B	47(9)	131(9)	52(9)	0(7)	2(8)	-4(7)
O4B	70(20)	122(18)	49(11)	2(12)	4(11)	12(15)
C46B	38(9)	122(10)	43(8)	-1(7)	-1(7)	3(8)
C34B	32(7)	96(7)	52(8)	3(6)	1(6)	-3(5)
C35B	58(8)	91(7)	64(7)	-1(6)	-1(6)	-2(6)
C36B	66(9)	94(8)	74(8)	1(6)	0(7)	0(7)
C37B	65(9)	93(8)	74(8)	2(6)	1(7)	0(7)
C38B	64(9)	91(9)	72(9)	3(7)	1(7)	1(7)
C39B	61(8)	87(9)	70(8)	0(7)	2(7)	2(7)
C40B	62(9)	88(9)	66(9)	0(7)	1(7)	2(7)
C41B	61(9)	90(9)	70(9)	0(7)	-1(7)	-2(7)
C42B	63(9)	87(9)	75(9)	0(7)	3(7)	2(7)
C43B	64(8)	93(9)	73(9)	2(7)	-1(7)	1(7)
C44B	62(8)	88(9)	74(8)	-1(7)	-1(7)	0(7)
C1B	37(7)	55(7)	38(6)	2(5)	-1(5)	0(6)
C2B	44(9)	55(9)	46(7)	3(6)	0(6)	2(7)
C3B	45(9)	54(9)	47(8)	3(7)	-2(7)	1(8)
C4B	46(9)	54(9)	45(8)	0(7)	-4(7)	2(8)
C5B	44(9)	53(9)	46(7)	1(6)	-5(6)	3(7)
C6B	45(7)	59(7)	45(7)	0(6)	-4(6)	2(6)
C7B	50(7)	58(7)	50(8)	3(6)	1(6)	2(5)
C8B	51(7)	60(7)	52(8)	5(6)	2(6)	3(6)
C9B	55(8)	62(8)	56(9)	2(7)	5(7)	2(6)
C10B	57(8)	62(8)	59(8)	1(7)	6(7)	2(6)
C11B	57(8)	59(8)	57(9)	1(7)	3(7)	2(6)

C12B	56(7)	58(7)	55(8)	1(6)	1(6)	3(6)
C13B	50(9)	60(9)	54(9)	5(7)	1(7)	4(7)
C14B	57(14)	65(16)	55(13)	7(11)	-4(11)	4(12)
C15B	47(15)	68(14)	57(13)	11(11)	0(11)	3(12)
C16B	61(9)	64(9)	63(10)	2(7)	9(8)	2(7)
C17B	45(13)	67(13)	63(14)	10(11)	25(11)	1(11)
C18B	68(16)	63(15)	66(13)	1(11)	9(12)	1(13)
C19B	57(9)	55(10)	61(10)	0(8)	1(7)	3(7)
C20B	59(16)	54(16)	64(12)	3(11)	1(10)	2(13)
C21B	63(14)	57(16)	69(14)	-3(12)	-1(11)	6(12)
C22B	33(7)	57(6)	37(7)	-1(6)	-1(6)	1(5)
C23B	37(8)	57(8)	46(9)	-1(7)	-4(7)	-1(6)
C24B	42(9)	57(8)	51(9)	0(7)	-7(7)	-3(7)
C25B	43(9)	53(9)	51(9)	-1(8)	-7(7)	-4(7)
C26B	42(9)	53(8)	45(9)	-3(7)	-4(7)	-4(7)
C27B	39(8)	53(8)	43(9)	0(7)	-5(7)	-2(7)
C28B	32(7)	53(7)	34(7)	2(6)	3(6)	0(6)
C29B	37(9)	51(8)	42(8)	0(7)	4(7)	0(7)
C30B	43(9)	56(9)	47(9)	-1(7)	6(7)	0(7)
C31B	45(10)	54(9)	47(9)	-1(7)	8(7)	-1(8)
C32B	43(9)	53(9)	42(9)	-1(7)	8(7)	-1(7)
C33B	38(9)	51(8)	37(8)	1(7)	4(7)	0(7)

Table 11. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 1.

	x	y	z	U(eq)
H2	810	5838	2722	26
H3	967	6042	1419	33
H4	1451	7155	1102	44
H5	1728	8129	2082	37
H9	2456	8678	4338	56
H11	1576	10518	4268	54
H13	2038	6615	3256	55
H14A	2419	7832	2510	91
H14B	2596	6724	2621	91
H14C	2733	7656	3142	91
H15A	2643	6836	4285	79
H15B	2419	5819	4086	79
H15C	2244	6653	4649	79
H16	2165	11167	4499	96
H17A	2692	11403	4644	160
H17B	2720	10283	4285	160
H17C	2756	10442	5195	160
H18A	2082	10221	5918	86
H18B	1802	10967	5485	86
H18C	2183	11399	5811	86
H19	980	8764	3317	40
H20A	1290	10639	2713	66
H20B	911	10158	2420	66
H20C	1290	9575	2263	66
H21A	878	9725	4490	60
H21B	628	10117	3791	60
H21C	988	10740	4034	60
H22	1059	4723	3454	28
H23A	1728	4998	4290	37
H23B	1668	5286	3403	37

H24A	1596	3537	3066	47
H24B	1971	3636	3543	47
H25A	1611	2228	4003	46
H25B	1679	3045	4673	46
H26A	1067	2540	4681	41
H26B	1024	2852	3796	41
H27A	760	4145	4548	29
H27B	1129	4257	5046	29
H28	1596	6829	4650	29
H29A	1179	8225	4791	32
H29B	982	7640	5484	32
H30A	1395	8879	5979	43
H30B	1731	8345	5533	43
H31A	1723	7748	6829	42
H31B	1304	7396	6739	42
H32A	1679	5968	6637	37
H32B	1916	6497	5969	37
H33A	1495	5341	5400	39
H33B	1158	5882	5826	39
H36A	347	9053	2366	27
H36B	571	8057	2106	27
H37	570	9260	1085	30
H38A	565	7518	723	39
H38B	344	8177	101	39
H39	0	6669	402	29
H40A	-221	6462	1703	25
H40B	221	6462	1703	25
H41A	0	9722	484	37
H41B	0	10096	1363	37
H44A	-353	4220	2172	29
H44B	-573	3715	2878	29
H45	-571	2528	1786	35
H46A	-360	1104	2499	42
H46B	-567	1869	3073	42
H47	0	1281	3627	43
H48A	-221	2959	3995	43

H48B	221	2959	3995	43
H49A	0	3114	1274	47
H49B	0	1900	1352	47
H36C	5561	6859	8518	73
H36D	5326	5919	8200	73
H37A	5521	5498	9542	76
H38C	5344	6776	10526	73
H38D	5588	7317	9875	73
H39A	5067	8269	10185	73
H40C	5257	8556	8926	69
H40D	4815	8538	8937	69
H41C	4462	7162	8482	73
H41D	4614	6071	8213	73
H42A	4937	5243	10088	78
H42B	4935	4920	9200	78
H43A	4652	6876	10507	74
H43B	4456	7622	9899	74
H44C	4392	5898	9480	76
H2A	4180	9002	7986	51
H3A	4012	8716	9162	51
H4A	3547	7552	9461	51
H5A	3262	6683	8512	50
H9A	2551	6049	6269	44
H11A	3444	4247	6378	47
H13A	2941	8115	7257	44
H14D	2316	6851	7430	61
H14E	2327	8017	7700	61
H14F	2593	7199	8095	61
H15D	2798	8289	5949	55
H15E	2493	8803	6488	55
H15F	2414	7723	6094	55
H16A	2814	3497	6080	51
H17D	2260	4230	6279	75
H17E	2254	3688	5454	75
H17F	2296	4899	5513	75
H18D	2952	4592	4720	74

H18E	2789	3459	4720	74
H18F	3187	3674	5073	74
H19A	4031	5912	7304	54
H20D	3770	5202	8343	80
H20E	4106	4473	8114	80
H20F	3693	4150	7905	80
H21D	4047	3989	6587	72
H21E	4388	4705	6797	72
H21F	4114	5023	6119	72
H22A	3938	9996	7142	53
H23C	3263	9751	6337	52
H23D	3330	9488	7225	52
H24C	3031	11066	7057	53
H24D	3406	11170	7531	53
H25C	3372	12484	6699	55
H25D	3293	11735	5990	55
H26C	3926	12185	5908	56
H26D	3993	11907	6796	56
H27C	3844	10473	5585	57
H27D	4220	10553	6053	57
H28A	3417	7846	5898	45
H29C	4029	6999	5062	51
H29D	3825	6422	5755	51
H30C	3580	5863	4598	45
H30D	3271	6475	5063	45
H31C	3270	6966	3786	45
H31D	3695	7279	3842	45
H32C	3103	8246	4623	42
H32D	3343	8740	3951	42
H33C	3493	9391	5202	44
H33D	3843	8938	4758	44
H36E	5290	11987	6750	93
H36F	4870	12184	6513	93
H37B	5096	13749	6993	93
H38E	4709	14031	8065	91
H38F	4487	13313	7484	91

H39B	4432	12710	8776	87
H40E	4632	10897	8381	87
H40F	4403	11412	7697	87
H41E	5300	10617	8431	88
H41F	5536	11083	7727	88
H42C	5622	13049	7724	90
H42D	5384	13705	8322	90
H43C	5063	13096	9218	92
H43D	4976	11909	9325	92
H44D	5548	12172	8817	89
H2B	5798	9528	7976	58
H3B	5956	9243	9193	59
H4B	6414	8095	9521	58
H5B	6714	7226	8635	58
H9B	7469	6759	6334	69
H11B	6613	4830	6312	69
H13B	7045	8703	7439	66
H14G	7373	7834	8306	89
H14H	7659	8618	7930	89
H14I	7663	7445	7678	89
H15G	7613	8447	6346	86
H15H	7511	9484	6780	86
H15I	7230	9000	6174	86
H16B	7522	5310	5532	75
H17G	7596	3589	5965	88
H17H	7301	3827	6624	88
H17I	7683	4434	6603	88
H18G	6873	4141	5365	99
H18H	7248	3869	4928	99
H18I	7042	4923	4753	99
H19B	5989	6380	7299	70
H20G	6253	4517	7876	89
H20H	5915	5179	8190	89
H20I	6327	5579	8301	89
H21G	5931	5521	6117	95
H21H	5673	5073	6780	95

H21I	6041	4485	6545	95
H22B	6058	10482	7131	51
H23E	6667	10042	7205	56
H23F	6723	10274	6310	56
H24E	6555	11751	7460	60
H24F	6940	11648	7023	60
H25E	6668	12160	5888	59
H25F	6613	13002	6547	59
H26E	6034	12696	5887	56
H26F	5996	12392	6779	56
H27E	5757	10975	6006	54
H27F	6135	10914	5535	54
H28B	6597	8294	5967	48
H29E	6212	6845	5798	52
H29F	5979	7427	5148	52
H30E	6409	6374	4624	59
H30F	6725	6951	5082	59
H31E	6339	7740	3813	58
H31F	6763	7390	3828	58
H32E	6918	8682	4700	55
H32F	6695	9222	4022	55
H33E	6180	9375	4788	50
H33F	6525	9842	5243	50

Figure 2. View of complex 1 of **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The hydrogen atoms have been omitted for clarity. The complex resides on a crystallographic mirror plane of symmetry at 0, y, z. Atoms with labels appended by a ‘ are related by –x, y, z.

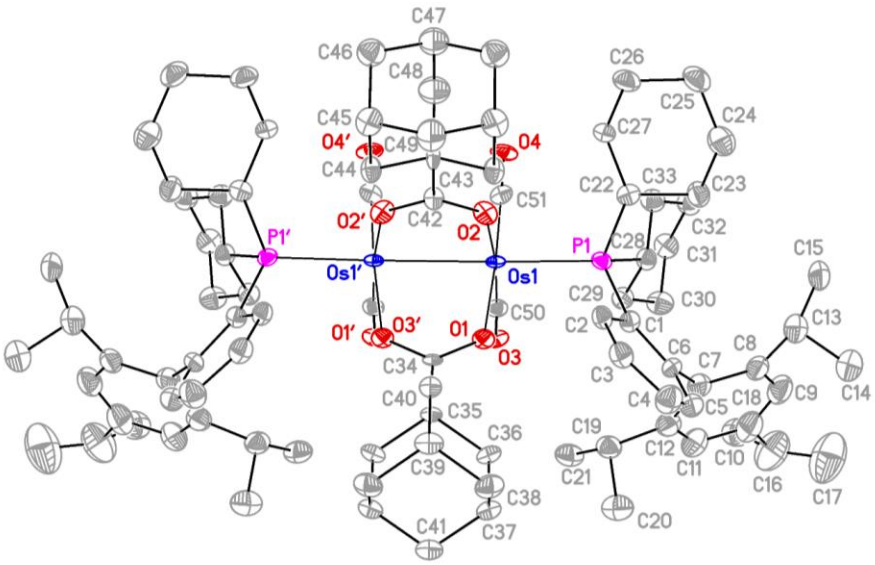
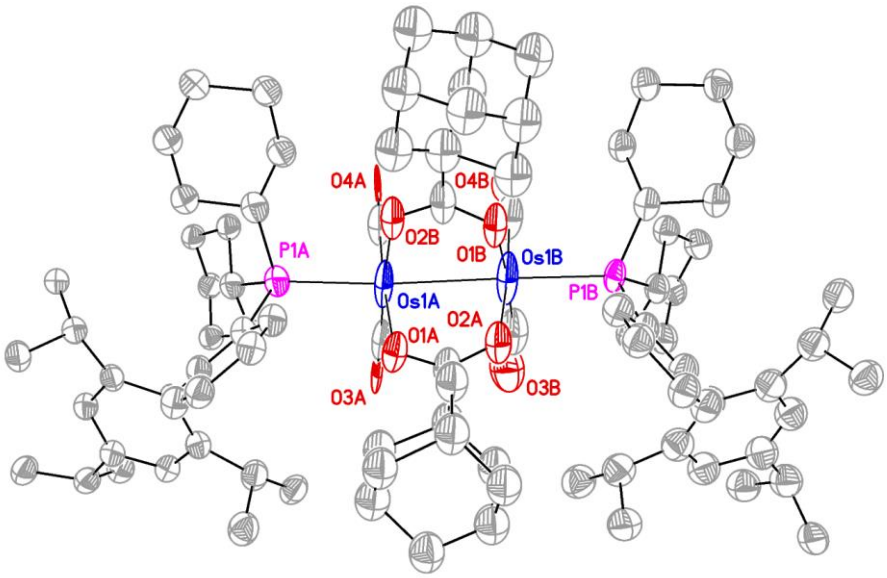


Figure 3. View of complex 2 of **$Os_2(CO)_4(O_2CR)_2(XPhos)_2$** showing the heteroatom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The hydrogen atoms have been omitted for clarity. The complex is disordered near a crystallographic mirror plane of symmetry at ½, y, z.



X-ray Experimental for complex $\mathbf{C_{22}H_{28}SO_4Br_2}$:

Crystals grew as very thin, colorless needles by vapor diffusion with chloroform and hexanes. The data crystal was cut from a longer needle and had approximate dimensions; 0.24 x 0.023 x 0.017 mm. The data were collected using a μ-focus Cu Kα radiation source (λ = 1.5418Å) with collimating mirror monochromators. A total of 575 frames of data were collected using ω-scans with a scan range of 1° and a counting time of 5 seconds per frame with a detector offset of +/- 42.2° and 20 seconds per frame with a detector offset of +/- 107.9°. The data were collected at 100 K. Details of crystal data, data collection and structure refinement are listed in Table 1. Data collection, unit cell refinement and data reduction were performed.The structure was solved by direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms The hydrogen atoms on carbon were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms). The hydrogen atom attached to O1 was observed in a ΔF map and refined with an isotropic displacement parameter.

The function, $\Sigma w(|F_O|^2 - |F_C|^2)^2$, was minimized, where $w = 1/[(\sigma (F_O))^2 + (0.0476*P)^2]$ and $P = (|F_O|^2 + 2|F_C|^2)/3$. $R_W(F^2)$ refined to 0.165, with R(F) equal to 0.0666 and a goodness of fit, S, = 1.01. Definitions used for calculating R(F), $R_W(F^2)$ and the goodness of fit, S, are given below.[1] The data were checked for secondary extinction effects but no correction was necessary. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[2] Tables of positional and thermal parameters, bond lengths and angles, torsion angles and figures are found elsewhere.

Crystallographic Material for **$C_{22}H_{28}SO_4Br_2$**

X-Ray Experimental

Table 12. Crystallographic Material for **$C_{22}H_{28}SO_4Br_2$**

Table 13. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of **$C_{22}H_{28}SO_4Br_2$**

Table 14. Bond Lengths (Å) and Angles (^{o}) for the non-hydrogen atoms of **$C_{22}H_{28}SO_4Br_2$**

Table 15. Anisotropic thermal parameters for the non-hydrogen atoms of **$C_{22}H_{28}SO_4Br_2$**.

Table 16. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of **$C_{22}H_{28}SO_4Br_2$**

Table 17. Torsion Angles (^{o}) for the non-hydrogen atoms of **$C_{22}H_{28}SO_4Br_2$**

Table 18. H-Bond Lengths (Å) and Angles (^{o}) for **$C_{22}H_{28}SO_4Br_2$**.

Figure 4 View of **$C_{22}H_{28}SO_4Br_2$**showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level.

Table 12. Crystal data and structure refinement for **$C_{22}H_{28}SO_4Br_2$**.

Empirical formula	C22 H28 Br2 O4 S	
Formula weight	548.32	
Temperature	100(2) K	
Wavelength	1.54184 Å	
Crystal system	monoclinic	
Space group	P 21/n	
Unit cell dimensions	a = 5.8411(7) Å	α = 90°.
	b = 15.530(3) Å	β = 95.069(9)°.
	c = 25.512(2) Å	γ = 90°.
Volume	2305.2(6) $Å^3$	
Z	4	
Density (calculated)	1.580 Mg/m^3	
Absorption coefficient	5.515 mm^{-1}	
F(000)	1112	
Crystal size	0.240 x 0.023 x 0.017 mm^3	
Theta range for data collection	3.335 to 74.479°.	
Index ranges	-6<=h<=7, -17<=k<=19, -31<=l<=20	
Reflections collected	7883	
Independent reflections	4466 [R(int) = 0.0818]	
Completeness to theta = 67.684°	99.2 %	
Absorption correction	Gaussian	
Max. and min. transmission	1.00 and 0.464	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4466 / 0 / 268	
Goodness-of-fit on F^2	1.009	
Final R indices [I>2sigma(I)]	R1 = 0.0666, wR2 = 0.1391	
R indices (all data)	R1 = 0.1218, wR2 = 0.1651	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.859 and -1.012 e.$Å^{-3}$	

Table 13. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 1. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C1	5008(12)	9438(5)	3352(3)	26(2)
C2	6762(12)	9415(5)	3016(3)	29(2)
C3	6546(13)	9844(5)	2534(3)	32(2)
C4	4623(12)	10334(5)	2393(3)	28(2)
C5	2898(12)	10389(5)	2731(3)	31(2)
C6	3065(12)	9950(5)	3202(3)	26(2)
C7	5137(12)	8996(5)	3889(3)	27(2)
C8	6693(13)	8196(5)	3916(3)	29(2)
C9	6942(13)	7767(5)	4462(3)	32(2)
C10	8885(13)	7114(5)	4539(3)	33(2)
C11	9181(12)	6735(5)	5091(3)	29(2)
C12	11335(13)	6177(5)	5189(3)	33(2)
C13	11610(15)	5806(6)	5745(3)	40(2)
C14	13771(16)	5253(7)	5841(4)	53(2)
C15	5770(15)	7540(6)	3500(3)	39(2)
C16	5850(12)	9686(5)	4307(3)	26(2)
C17	7420(11)	11579(5)	3950(2)	23(1)
C18	8258(12)	11662(5)	3462(3)	27(2)
C19	7068(13)	12173(5)	3079(3)	31(2)
C20	5060(12)	12582(5)	3204(3)	29(2)
C21	4263(13)	12508(5)	3689(3)	31(2)
C22	5402(12)	11999(5)	4074(3)	28(2)
Br1	4294(2)	10916(1)	1742(1)	44(1)
Br2	3412(2)	13246(1)	2674(1)	44(1)
O1	2811(10)	8770(4)	3975(2)	35(1)
O2	8203(8)	9968(3)	4224(2)	24(1)
O3	11280(8)	10958(4)	4386(2)	28(1)
O4	7963(8)	11045(4)	4921(2)	28(1)
S1	8859(3)	10901(1)	4424(1)	21(1)

Table14. Bond lengths [Å] and angles [°] for 1.

C1-C2	1.393(10)	C12-H12B	0.99
C1-C6	1.411(10)	C13-C14	1.528(12)
C1-C7	1.528(10)	C13-H13A	0.99
C2-C3	1.395(10)	C13-H13B	0.99
C2-H2	0.95	C14-H14A	0.98
C3-C4	1.378(11)	C14-H14B	0.98
C3-H3	0.95	C14-H14C	0.98
C4-C5	1.385(10)	C15-H15A	0.98
C4-Br1	1.885(7)	C15-H15B	0.98
C5-C6	1.377(10)	C15-H15C	0.98
C5-H5	0.95	C16-O2	1.476(8)
C6-H6	0.95	C16-H16A	0.99
C7-O1	1.439(9)	C16-H16B	0.99
C7-C8	1.537(11)	C17-C18	1.383(9)
C7-C16	1.543(9)	C17-C22	1.408(10)
C8-C15	1.535(10)	C17-S1	1.759(7)
C8-C9	1.540(10)	C18-C19	1.397(10)
C8-H8	1.00	C18-H18	0.95
C9-C10	1.521(11)	C19-C20	1.395(11)
C9-H9A	0.99	C19-H19	0.95
C9-H9B	0.99	C20-C21	1.364(11)
C10-C11	1.523(10)	C20-Br2	1.894(7)
C10-H10A	0.99	C21-C22	1.385(10)
C10-H10B	0.99	C21-H21	0.95
C11-C12	1.531(11)	C22-H22	0.95
C11-H11A	0.99	O1-H1O	0.64(10)
C11-H11B	0.99	O2-S1	1.572(5)
C12-C13	1.526(10)	O3-S1	1.429(5)
C12-H12A	0.99	O4-S1	1.433(5)

C2-C1-C6	117.7(7)	C3-C2-H2	119.4
C2-C1-C7	124.0(7)	C1-C2-H2	119.5
C6-C1-C7	118.1(6)	C4-C3-C2	120.1(7)
C3-C2-C1	121.1(7)	C4-C3-H3	120.0

C2-C3-H3	120.0	C12-C11-H11A	108.9
C3-C4-C5	119.6(7)	C10-C11-H11B	108.9
C3-C4-Br1	120.7(6)	C12-C11-H11B	108.9
C5-C4-Br1	119.7(6)	H11A-C11-H11B	107.7
C6-C5-C4	120.9(7)	C13-C12-C11	112.5(7)
C6-C5-H5	119.6	C13-C12-H12A	109.1
C4-C5-H5	119.6	C11-C12-H12A	109.1
C5-C6-C1	120.6(7)	C13-C12-H12B	109.1
C5-C6-H6	119.7	C11-C12-H12B	109.1
C1-C6-H6	119.7	H12A-C12-H12B	107.8
O1-C7-C1	105.9(6)	C12-C13-C14	112.2(8)
O1-C7-C8	111.0(6)	C12-C13-H13A	109.2
C1-C7-C8	112.6(6)	C14-C13-H13A	109.2
O1-C7-C16	105.4(6)	C12-C13-H13B	109.2
C1-C7-C16	107.2(6)	C14-C13-H13B	109.2
C8-C7-C16	114.1(6)	H13A-C13-H13B	107.9
C15-C8-C7	109.7(6)	C13-C14-H14A	109.5
C15-C8-C9	109.7(7)	C13-C14-H14B	109.5
C7-C8-C9	113.5(6)	H14A-C14-H14B	109.5
C15-C8-H8	107.9	C13-C14-H14C	109.5
C7-C8-H8	107.9	H14A-C14-H14C	109.5
C9-C8-H8	107.9	H14B-C14-H14C	109.5
C10-C9-C8	114.5(6)	C8-C15-H15A	109.5
C10-C9-H9A	108.6	C8-C15-H15B	109.5
C8-C9-H9A	108.6	H15A-C15-H15B	109.5
C10-C9-H9B	108.6	C8-C15-H15C	109.5
C8-C9-H9B	108.6	H15A-C15-H15C	109.5
H9A-C9-H9B	107.6	H15B-C15-H15C	109.5
C9-C10-C11	113.5(7)	O2-C16-C7	107.7(5)
C9-C10-H10A	108.9	O2-C16-H16A	110.2
C11-C10-H10A	108.9	C7-C16-H16A	110.2
C9-C10-H10B	108.9	O2-C16-H16B	110.2
C11-C10-H10B	108.9	C7-C16-H16B	110.2
H10A-C10-H10B	107.7	H16A-C16-H16B	108.5
C10-C11-C12	113.2(6)	C18-C17-C22	121.9(6)
C10-C11-H11A	108.9	C18-C17-S1	119.2(5)

C22-C17-S1	118.8(5)	C22-C21-H21	119.7
C17-C18-C19	119.3(6)	C21-C22-C17	117.7(7)
C17-C18-H18	120.4	C21-C22-H22	121.2
C19-C18-H18	120.4	C17-C22-H22	121.2
C20-C19-C18	118.4(7)	C7-O1-H1O	118(10)
C20-C19-H19	120.8	C16-O2-S1	115.5(4)
C18-C19-H19	120.8	O3-S1-O4	119.5(3)
C21-C20-C19	122.0(7)	O3-S1-O2	104.4(3)
C21-C20-Br2	120.0(6)	O4-S1-O2	109.5(3)
C19-C20-Br2	118.0(5)	O3-S1-C17	109.4(3)
C20-C21-C22	120.7(7)	O4-S1-C17	109.0(3)
C20-C21-H21	119.6	O2-S1-C17	103.9(3)

Table 15. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 1. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C1	26(3)	29(4)	22(3)	-8(3)	3(3)	-5(3)
C2	25(4)	32(4)	30(4)	-1(3)	5(3)	1(3)
C3	30(4)	39(4)	28(3)	2(3)	8(3)	4(3)
C4	25(4)	28(4)	31(4)	0(3)	5(3)	-4(3)
C5	24(4)	33(4)	37(4)	-4(3)	3(3)	0(3)
C6	27(4)	31(4)	22(3)	-1(3)	5(3)	-3(3)
C7	23(3)	26(4)	32(3)	-2(3)	5(3)	-7(3)
C8	32(4)	28(4)	27(3)	-3(3)	1(3)	-3(3)
C9	34(4)	36(4)	24(3)	-7(3)	-5(3)	6(3)
C10	34(4)	28(4)	37(4)	6(3)	4(3)	4(3)
C11	33(4)	24(4)	30(3)	-4(3)	6(3)	-4(3)
C12	36(4)	27(4)	37(4)	1(3)	6(3)	-3(3)
C13	45(5)	34(5)	40(4)	12(4)	4(3)	-8(4)
C14	47(5)	50(6)	60(6)	16(5)	-3(4)	2(5)
C15	46(5)	40(5)	28(4)	-5(4)	-5(3)	-1(4)
C16	23(3)	27(4)	28(3)	-3(3)	3(3)	-3(3)
C17	21(3)	25(4)	23(3)	0(3)	2(2)	-2(3)
C18	21(3)	32(4)	30(3)	4(3)	4(3)	4(3)
C19	34(4)	35(4)	23(3)	2(3)	6(3)	-3(3)
C20	27(4)	33(4)	25(3)	5(3)	-7(3)	-2(3)
C21	29(4)	25(4)	37(4)	4(3)	-1(3)	-3(3)
C22	21(3)	28(4)	35(4)	4(3)	4(3)	-2(3)
Br1	53(1)	47(1)	32(1)	11(1)	8(1)	6(1)
Br2	38(1)	45(1)	48(1)	21(1)	-8(1)	2(1)
O1	26(3)	47(4)	33(3)	-1(3)	4(2)	-7(2)
O2	23(2)	21(2)	27(2)	-1(2)	0(2)	-1(2)
O3	20(2)	35(3)	30(2)	1(2)	2(2)	0(2)
O4	31(3)	37(3)	16(2)	-1(2)	8(2)	0(2)
S1	18(1)	25(1)	21(1)	-1(1)	1(1)	-2(1)

Table 16. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for 1.

	x	y	z	U(eq)
H2	8127	9102	3117	35
H3	7727	9799	2302	38
H5	1582	10733	2637	37
H6	1862	9993	3428	32
H8	8256	8380	3829	35
H9A	7201	8221	4733	38
H9B	5479	7475	4520	38
H10A	10339	7396	4462	40
H10B	8583	6640	4282	40
H11A	7813	6383	5149	35
H11B	9261	7211	5350	35
H12A	11254	5698	4932	40
H12B	12705	6528	5131	40
H13A	10246	5452	5803	48
H13B	11685	6285	6002	48
H14A	13721	4786	5582	79
H14B	13844	5009	6196	79
H14C	15133	5610	5806	79
H15A	4204	7372	3566	58
H15B	6761	7029	3519	58
H15C	5755	7798	3149	58
H16A	4780	10181	4270	31
H16B	5808	9443	4665	31
H18	9629	11374	3389	33
H19	7612	12241	2742	37
H21	2912	12809	3763	37
H22	4841	11937	4411	34
H1O	2580(180)	8680(80)	4210(40)	50(40)

Table 17. Torsion angles [°] for 1.

C6-C1-C2-C3	-3.6(11)	C10-C11-C12-C13	179.7(7)
C7-C1-C2-C3	-179.0(7)	C11-C12-C13-C14	-179.7(7)
C1-C2-C3-C4	3.1(12)	O1-C7-C16-O2	-177.9(6)
C2-C3-C4-C5	-0.7(12)	C1-C7-C16-O2	-65.3(7)
C2-C3-C4-Br1	-179.8(6)	C8-C7-C16-O2	60.1(8)
C3-C4-C5-C6	-1.0(11)	C22-C17-C18-C19	-0.3(11)
Br1-C4-C5-C6	178.2(6)	S1-C17-C18-C19	177.5(6)
C4-C5-C6-C1	0.3(11)	C17-C18-C19-C20	-0.2(11)
C2-C1-C6-C5	1.9(10)	C18-C19-C20-C21	1.2(12)
C7-C1-C6-C5	177.6(7)	C18-C19-C20-Br2	-178.1(6)
C2-C1-C7-O1	-149.9(7)	C19-C20-C21-C22	-1.8(12)
C6-C1-C7-O1	34.6(9)	Br2-C20-C21-C22	177.6(6)
C2-C1-C7-C8	-28.4(10)	C20-C21-C22-C17	1.2(11)
C6-C1-C7-C8	156.2(6)	C18-C17-C22-C21	-0.1(11)
C2-C1-C7-C16	97.9(8)	S1-C17-C22-C21	-178.0(5)
C6-C1-C7-C16	-77.5(8)	C7-C16-O2-S1	154.6(4)
O1-C7-C8-C15	58.8(8)	C16-O2-S1-O3	170.1(4)
C1-C7-C8-C15	-59.8(8)	C16-O2-S1-O4	41.0(5)
C16-C7-C8-C15	177.6(6)	C16-O2-S1-C17	-75.3(5)
O1-C7-C8-C9	-64.3(8)	C18-C17-S1-O3	36.7(7)
C1-C7-C8-C9	177.1(6)	C22-C17-S1-O3	-145.4(6)
C16-C7-C8-C9	54.6(8)	C18-C17-S1-O4	169.0(6)
C15-C8-C9-C10	70.8(9)	C22-C17-S1-O4	-13.1(7)
C7-C8-C9-C10	-166.2(6)	C18-C17-S1-O2	-74.3(6)
C8-C9-C10-C11	176.8(6)	C22-C17-S1-O2	103.6(6)
C9-C10-C11-C12	-172.0(7)		

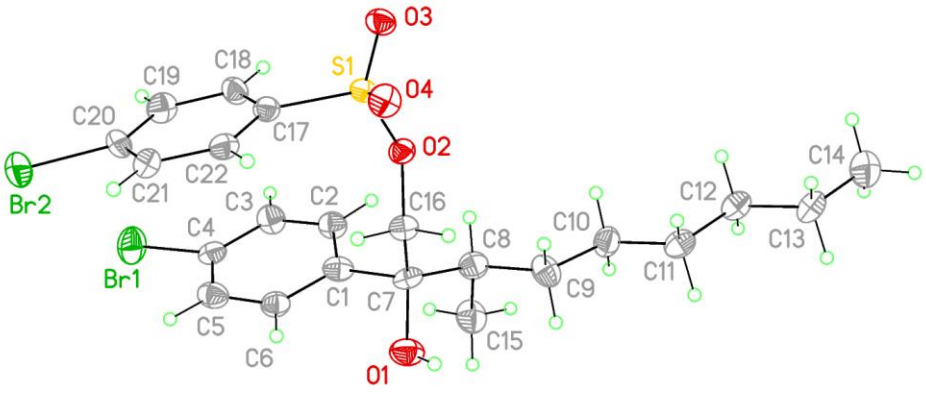
Table 18. Hydrogen bonds for 1 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O1-H1O...O4#1	0.64(10)	2.31(10)	2.904(8)	156(13)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+2,-z+1

Figure 4. View of $C_{22}H_{28}SO_4Br_2$ showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level.



References

[1] $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ where w is the weight given each reflection. $R(F) = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|\}$ for reflections with $F_o > 4(\sigma(F_o))$.

$S = [\Sigma w(|F_o|^2 - |F_c|^2)^2/(n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.

[2] International Tables for X-ray Crystallography (**1992**). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.