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**Table S-10.** U(aniso) values for 3.

	U11	U22	U33	U23	U13	U12
Rh(1A)	35(1)	29(1)	37(1)	-1(1)	6(1)	-3(1)
C(1A)	44(4)	33(3)	49(4)	3(3)	22(3)	1(3)
C(2A)	34(3)	34(4)	43(3)	-8(3)	9(3)	3(3)
C(3A)	41(4)	45(4)	42(4)	15(3)	12(3)	13(3)
C(4A)	59(4)	32(4)	57(4)	-6(3)	12(3)	1(3)
C(5A)	50(4)	45(4)	33(3)	2(3)	13(3)	-5(3)
C(6A)	62(5)	42(4)	100(6)	21(4)	30(4)	-4(4)
C(7A)	55(5)	83(6)	67(5)	-36(5)	13(4)	-18(4)
C(8A)	73(5)	80(6)	66(5)	30(5)	26(4)	21(5)
C(9A)	113(7)	37(4)	92(6)	-23(4)	43(5)	-9(5)
C(10A)	96(6)	88(7)	35(4)	-6(4)	14(4)	4(5)
P(1A)	42(1)	40(1)	71(1)	-3(1)	16(1)	5(1)
C(11A)	82(6)	48(5)	83(6)	-22(4)	25(5)	10(4)
C(12A)	60(5)	63(6)	153(9)	-13(6)	51(6)	5(4)
C(13A)	79(6)	65(6)	94(7)	10(5)	4(5)	25(5)
S(1A)	59(1)	62(1)	50(1)	8(1)	-2(1)	-16(1)
C(17A)	34(3)	34(4)	60(4)	-2(3)	17(3)	6(3)
C(21A)	42(4)	38(4)	69(5)	-12(4)	10(3)	3(3)
C(20A)	44(4)	44(5)	109(7)	-28(5)	20(4)	-10(3)
C(19A)	73(5)	39(4)	107(7)	-11(5)	49(5)	-10(4)
C(18A)	61(5)	45(4)	65(5)	-1(4)	27(4)	6(4)
C(16A)	38(3)	33(3)	67(5)	0(3)	18(3)	0(3)
C(15A)	56(4)	58(5)	50(4)	-1(4)	7(3)	0(4)
C(14A)	44(4)	84(6)	43(4)	21(4)	20(3)	18(4)
Rh(1B)	41(1)	26(1)	41(1)	0(1)	13(1)	1(1)
C(1B)	26(3)	36(4)	100(6)	-16(4)	14(4)	-4(3)
C(2B)	55(4)	33(4)	48(4)	-6(3)	0(3)	-3(3)
C(3B)	36(3)	28(3)	54(4)	-7(3)	12(3)	-7(3)
C(4B)	59(4)	34(3)	48(4)	2(3)	12(3)	-13(3)
C(5B)	60(5)	39(4)	80(5)	-7(4)	38(4)	-17(3)
C(6B)	48(5)	63(6)	199(12)	-34(7)	17(6)	6(4)
C(7B)	128(8)	54(5)	59(5)	-7(4)	-12(5)	9(5)
C(8B)	71(5)	35(4)	100(6)	-14(4)	41(5)	2(4)
C(9B)	105(7)	48(5)	65(5)	18(4)	-4(5)	-19(5)
C(10B)	140(9)	81(7)	127(9)	-42(6)	99(8)	-46(7)
P(1B)	44(1)	32(1)	64(1)	4(1)	14(1)	-3(1)
C(11B)	62(5)	98(8)	81(6)	12(5)	6(5)	-6(5)
C(12B)	61(5)	37(4)	184(11)	-19(6)	37(6)	-22(4)
C(13B)	61(5)	77(6)	92(6)	6(5)	36(4)	-9(5)
S(1B)	88(4)	34(3)	43(3)	4(3)	28(3)	14(3)
C(17B)	51(12)	26(9)	59(12)	18(9)	8(9)	17(9)
C(21B)	57(10)	20(10)	57(10)	-7(8)	0(8)	3(7)
C(20B)	51(10)	28(11)	76(13)	6(11)	0(11)	5(8)
C(19B)	54(10)	24(8)	61(17)	-11(12)	2(13)	0(7)
C(18B)	62(11)	25(11)	59(11)	6(8)	8(9)	14(9)
C(16B)	45(10)	20(13)	50(9)	-15(7)	4(8)	-14(8)
C(15B)	101(13)	15(8)	54(9)	-16(7)	27(9)	-7(7)
C(14B)	76(19)	48(17)	36(10)	5(9)	13(10)	-19(12)
S(1C)	71(4)	48(5)	36(3)	-7(3)	11(3)	0(3)
C(17C)	50(9)	9(11)	66(11)	-7(7)	21(8)	-3(8)
C(21C)	58(10)	25(11)	65(11)	-7(8)	15(9)	3(8)
C(20C)	72(12)	55(14)	88(17)	-21(13)	7(14)	20(10)
C(19C)	64(11)	28(10)	78(20)	-7(16)	-9(17)	13(8)
C(18C)	72(14)	27(14)	70(12)	10(10)	3(11)	0(10)
C(16C)	43(11)	44(14)	45(10)	-13(10)	11(9)	-15(8)
C(15C)	78(12)	49(12)	53(10)	19(8)	1(9)	-16(9)
C(14C)	53(11)	48(13)	33(8)	-15(9)	20(7)	-25(10)

**Table S-11.** Table of Least-Squares Planes for 3.

Least-squares planes (x,y,z in crystal coordinates) and deviations from them  
 (\* indicates atom used to define plane)

$$8.303 (0.034) x + 7.761 (0.010) y + 5.046 (0.099) z = 4.627 (0.019)$$

\* 0.000 (0.000) Rh1A  
 \* 0.000 (0.000) C14A  
 \* 0.000 (0.000) S1A

Rms deviation of fitted atoms = 0.000

$$11.876 (0.040) x + 6.486 (0.018) y + 7.706 (0.048) z = 7.679 (0.040)$$

Angle to previous plane (with approximate esd) = 14.45 ( 0.32 )

\* 0.029 (0.003) S1A  
 \* -0.042 (0.004) C14A  
 \* 0.050 (0.005) C15A  
 \* -0.002 (0.004) C16A  
 \* -0.034 (0.004) C17A

Rms deviation of fitted atoms = 0.035

$$17.257 (0.072) x + 1.330 (0.091) y - 2.217 (0.384) z = 5.294 (0.045)$$

Angle to previous plane (with approximate esd) = 58.79 ( 0.56 )

\* 0.000 (0.000) Rh1B  
 \* 0.000 (0.000) C14B  
 \* 0.000 (0.000) S1B

Rms deviation of fitted atoms = 0.000

$$12.824 (0.191) x + 5.872 (0.116) y - 0.424 (0.218) z = 3.974 (0.090)$$

Angle to previous plane (with approximate esd) = 33.32 ( 1.19 )

\* -0.070 (0.016) S1B  
 \* 0.100 (0.016) C14B  
 \* -0.122 (0.021) C15B  
 \* 0.010 (0.025) C16B  
 \* 0.082 (0.029) C17B

Rms deviation of fitted atoms = 0.086

$$16.741 (0.030) x + 1.646 (0.058) y + 0.505 (0.223) z = 5.565 (0.017)$$

Angle to previous plane (with approximate esd) = 31.16 ( 1.08 )

\* 0.000 (0.000) Rh1B  
 \* 0.000 (0.000) C14C  
 \* 0.000 (0.000) S1C

Rms deviation of fitted atoms = 0.000

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$$12.474 (0.185) x + 6.062 (0.097) y - 0.365 (0.196) z = 3.937 (0.070)$$

Angle to previous plane (with approximate esd) = 32.83 ( 1.02 )

\* -0.073 (0.013) S1C  
\* 0.108 (0.012) C14C  
\* -0.132 (0.017) C15C  
\* 0.011 (0.025) C16C  
\* 0.086 (0.025) C17C

Rms deviation of fitted atoms = 0.092

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**Table S-12.** Intramolecular Distances (Å) for **4**.

Bonds	Distance	Bonds	Distance
Rh(1)-C(14)	2.051(5)	C(3)-C(8)	1.500(6)
Rh(1)-C(3)	2.228(4)	C(4)-C(5)	1.418(6)
Rh(1)-C(4)	2.233(4)	C(4)-C(9)	1.501(6)
Rh(1)-C(5)	2.233(4)	C(5)-C(10)	1.492(6)
Rh(1)-C(2)	2.265(5)	C(14)-C(15)	1.419(6)
Rh(1)-P(1)	2.2718(11)	C(14)-C(18)	1.420(6)
Rh(1)-C(1)	2.276(5)	C(15)-C(21)	1.423(6)
Rh(1)-S(1)	2.3349(14)	C(15)-C(16)	1.487(6)
S(1)-C(17)	1.769(5)	C(16)-C(17)	1.413(6)
P(1)-C(11)	1.816(5)	C(16)-C(22)	1.414(6)
P(1)-C(12)	1.822(5)	C(17)-C(25)	1.386(6)
P(1)-C(13)	1.835(5)	C(18)-C(19)	1.388(6)
C(1)-C(2)	1.417(7)	C(19)-C(20)	1.376(7)
C(1)-C(5)	1.452(7)	C(20)-C(21)	1.373(7)
C(1)-C(6)	1.496(6)	C(22)-C(23)	1.376(7)
C(2)-C(3)	1.461(7)	C(23)-C(24)	1.387(8)
C(2)-C(7)	1.503(6)	C(24)-C(25)	1.383(8)
C(3)-C(4)	1.423(6)		

**Table S-13.** Intramolecular Bond Angles (°) for **4**.

Bonds	Angle	Bonds	Angle
C(14)-Rh(1)-C(3)	107.2(2)	C(3)-C(2)-C(7)	125.7(4)
C(14)-Rh(1)-C(4)	92.7(2)	C(1)-C(2)-Rh(1)	72.2(3)
C(3)-Rh(1)-C(4)	37.2(2)	C(3)-C(2)-Rh(1)	69.6(2)
C(14)-Rh(1)-C(5)	113.0(2)	C(7)-C(2)-Rh(1)	128.4(3)
C(3)-Rh(1)-C(5)	62.6(2)	C(4)-C(3)-C(2)	107.1(4)
C(4)-Rh(1)-C(5)	37.0(2)	C(4)-C(3)-C(8)	126.9(4)
C(14)-Rh(1)-C(2)	144.8(2)	C(2)-C(3)-C(8)	125.5(4)
C(3)-Rh(1)-C(2)	37.9(2)	C(4)-C(3)-Rh(1)	71.6(3)
C(4)-Rh(1)-C(2)	62.1(2)	C(2)-C(3)-Rh(1)	72.4(3)
C(5)-Rh(1)-C(2)	62.2(2)	C(8)-C(3)-Rh(1)	127.9(3)
C(14)-Rh(1)-P(1)	87.76(13)	C(5)-C(4)-C(3)	109.3(4)
C(3)-Rh(1)-P(1)	109.81(12)	C(5)-C(4)-C(9)	125.6(4)
C(4)-Rh(1)-P(1)	145.02(13)	C(3)-C(4)-C(9)	125.1(4)
C(5)-Rh(1)-P(1)	159.03(13)	C(5)-C(4)-Rh(1)	71.5(3)
C(2)-Rh(1)-P(1)	99.37(13)	C(3)-C(4)-Rh(1)	71.2(2)
C(14)-Rh(1)-C(1)	150.5(2)	C(9)-C(4)-Rh(1)	125.5(3)
C(3)-Rh(1)-C(1)	62.2(2)	C(4)-C(5)-C(1)	107.5(4)
C(4)-Rh(1)-C(1)	61.8(2)	C(4)-C(5)-C(10)	127.1(5)
C(5)-Rh(1)-C(1)	37.6(2)	C(1)-C(5)-C(10)	125.0(4)
C(2)-Rh(1)-C(1)	36.4(2)	C(4)-C(5)-Rh(1)	71.5(3)
P(1)-Rh(1)-C(1)	121.54(13)	C(1)-C(5)-Rh(1)	72.8(3)
C(14)-Rh(1)-S(1)	89.21(13)	C(10)-C(5)-Rh(1)	126.4(3)
C(3)-Rh(1)-S(1)	158.07(12)	C(15)-C(14)-C(18)	117.4(4)
C(4)-Rh(1)-S(1)	130.25(13)	C(15)-C(14)-Rh(1)	126.1(3)
C(5)-Rh(1)-S(1)	97.93(12)	C(18)-C(14)-Rh(1)	116.4(3)
C(2)-Rh(1)-S(1)	125.66(12)	C(14)-C(15)-C(21)	117.9(4)
P(1)-Rh(1)-S(1)	84.72(4)	C(14)-C(15)-C(16)	124.7(4)
C(1)-Rh(1)-S(1)	96.36(12)	C(21)-C(15)-C(16)	117.4(4)
C(17)-S(1)-Rh(1)	104.6(2)	C(17)-C(16)-C(22)	116.0(4)
C(11)-P(1)-C(12)	102.4(3)	C(17)-C(16)-C(15)	124.5(4)
C(11)-P(1)-C(13)	102.3(3)	C(22)-C(16)-C(15)	119.5(4)
C(12)-P(1)-C(13)	101.6(3)	C(25)-C(17)-C(16)	119.9(4)
C(11)-P(1)-Rh(1)	112.7(2)	C(25)-C(17)-S(1)	117.1(4)
C(12)-P(1)-Rh(1)	118.1(2)	C(16)-C(17)-S(1)	123.0(3)
C(13)-P(1)-Rh(1)	117.4(2)	C(19)-C(18)-C(14)	122.4(5)
C(2)-C(1)-C(5)	108.1(4)	C(20)-C(19)-C(18)	119.8(5)
C(2)-C(1)-C(6)	126.6(5)	C(21)-C(20)-C(19)	119.5(5)
C(5)-C(1)-C(6)	125.3(5)	C(20)-C(21)-C(15)	122.7(5)
C(2)-C(1)-Rh(1)	71.4(3)	C(23)-C(22)-C(16)	123.6(5)
C(5)-C(1)-Rh(1)	69.6(3)	C(22)-C(23)-C(24)	119.0(6)
C(6)-C(1)-Rh(1)	125.5(4)	C(25)-C(24)-C(23)	119.0(5)
C(1)-C(2)-C(3)	107.9(4)	C(24)-C(25)-C(17)	122.4(5)
C(1)-C(2)-C(7)	126.2(5)		

**Table S-14.** Positional parameters and B(eq) for **4**.

	x	y	z	U (eq)
Rh (1)	-3528 (1)	-2544 (1)	-197 (1)	23 (1)
S (1)	-3713 (1)	-3685 (1)	803 (1)	32 (1)
P (1)	-1281 (1)	-2481 (1)	180 (1)	31 (1)
C (1)	-4240 (5)	-3455 (3)	-1241 (3)	32 (1)
C (2)	-3256 (5)	-2811 (3)	-1536 (3)	34 (1)
C (3)	-3793 (5)	-1874 (3)	-1401 (3)	31 (1)
C (4)	-5114 (5)	-1974 (3)	-1039 (3)	30 (1)
C (5)	-5391 (5)	-2932 (3)	-912 (3)	31 (1)
C (6)	-4141 (7)	-4494 (3)	-1272 (3)	56 (2)
C (7)	-1958 (6)	-3046 (4)	-1986 (3)	50 (1)
C (8)	-3151 (5)	-989 (3)	-1701 (3)	43 (1)
C (9)	-6082 (5)	-1188 (4)	-853 (4)	50 (1)
C (10)	-6684 (5)	-3350 (4)	-589 (3)	49 (1)
C (11)	-403 (5)	-3588 (3)	58 (3)	49 (2)
C (12)	-887 (5)	-2179 (4)	1224 (3)	50 (1)
C (13)	-155 (5)	-1680 (3)	-377 (4)	51 (2)
C (14)	-3920 (5)	-1547 (3)	661 (3)	27 (1)
C (15)	-4784 (4)	-1650 (3)	1347 (3)	28 (1)
C (16)	-5632 (4)	-2487 (4)	1523 (3)	32 (1)
C (17)	-5249 (5)	-3409 (3)	1324 (3)	31 (1)
C (18)	-3293 (5)	-665 (3)	538 (3)	35 (1)
C (19)	-3432 (5)	61 (3)	1086 (3)	41 (1)
C (20)	-4198 (5)	-64 (4)	1777 (3)	43 (1)
C (21)	-4868 (5)	-893 (3)	1900 (3)	38 (1)
C (22)	-6907 (5)	-2384 (4)	1925 (3)	42 (1)
C (23)	-7724 (6)	-3121 (5)	2153 (3)	54 (2)
C (24)	-7309 (6)	-4018 (5)	1963 (3)	55 (2)
C (25)	-6083 (5)	-4146 (4)	1553 (3)	42 (1)

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**Table S-15.** U(aniso) values for 4.

	U11	U22	U33	U23	U13	U12
Rh(1)	24(1)	24(1)	21(1)	-2(1)	2(1)	0(1)
S(1)	38(1)	29(1)	29(1)	3(1)	6(1)	2(1)
P(1)	26(1)	35(1)	34(1)	2(1)	1(1)	1(1)
C(1)	38(3)	35(3)	24(3)	-3(2)	1(2)	-6(2)
C(2)	43(3)	41(3)	19(2)	0(2)	3(2)	5(2)
C(3)	37(3)	32(3)	22(2)	1(2)	-3(2)	-8(2)
C(4)	32(3)	34(3)	23(2)	-1(2)	-8(2)	3(2)
C(5)	32(3)	38(3)	23(2)	-1(2)	-4(2)	-11(2)
C(6)	90(5)	31(3)	49(3)	-11(3)	0(3)	-6(3)
C(7)	52(3)	65(4)	33(3)	-4(3)	13(3)	14(3)
C(8)	45(3)	43(3)	42(3)	15(2)	0(2)	-9(2)
C(9)	40(3)	56(3)	53(4)	-1(3)	-4(3)	18(3)
C(10)	40(3)	67(4)	40(3)	4(3)	-6(2)	-19(3)
C(11)	39(3)	52(3)	56(4)	6(2)	3(3)	14(2)
C(12)	34(3)	67(4)	48(3)	-6(3)	-11(3)	-4(3)
C(13)	33(3)	53(3)	67(5)	10(3)	3(3)	-11(2)
C(14)	27(2)	29(2)	26(3)	2(2)	5(2)	2(2)
C(15)	23(2)	34(2)	27(2)	-3(2)	-4(2)	6(2)
C(16)	27(2)	45(3)	22(2)	-1(2)	-1(2)	4(2)
C(17)	32(3)	41(3)	18(2)	5(2)	-1(2)	-1(2)
C(18)	40(3)	30(2)	35(3)	-3(2)	0(2)	4(2)
C(19)	48(3)	32(3)	42(3)	-8(2)	-10(2)	6(2)
C(20)	59(3)	39(3)	32(3)	-12(2)	-11(2)	19(3)
C(21)	48(3)	40(3)	24(2)	-3(2)	0(2)	13(2)
C(22)	35(3)	58(3)	33(3)	7(3)	8(2)	12(3)
C(23)	30(3)	89(4)	43(4)	21(3)	8(2)	-3(3)
C(24)	47(3)	73(4)	44(3)	22(3)	-4(3)	-15(3)
C(25)	42(3)	54(3)	30(3)	7(2)	0(2)	-13(2)



**Table S-16.** Table of Least-Squares Planes for 4.

Least-squares planes (x,y,z in crystal coordinates) and deviations from them  
 (\* indicates atom used to define plane)

$$- 9.519 (0.004) x + 1.122 (0.023) y + 3.043 (0.027) z = 3.704 (0.005)$$

\* 0.000 (0.000) Rh1  
 \* 0.000 (0.000) S1  
 \* 0.000 (0.000) C14

Rms deviation of fitted atoms = 0.000

$$- 5.883 (0.015) x + 0.156 (0.019) y + 13.166 (0.020) z = 1.291 (0.012)$$

Angle to previous plane (with approximate esd) = 42.22 ( 0.16 )

\* -0.106 (0.002) S1  
 \* 0.107 (0.003) C17  
 \* 0.056 (0.003) C16  
 \* -0.225 (0.003) C15  
 \* 0.169 (0.002) C14

Rms deviation of fitted atoms = 0.145

$$- 7.764 (0.011) x - 4.818 (0.024) y + 8.254 (0.027) z = 1.782 (0.008)$$

Angle to previous plane (with approximate esd) = 28.69 ( 0.20 )

\* -0.030 (0.003) C14  
 \* 0.025 (0.003) C15  
 \* 0.010 (0.003) C18  
 \* 0.015 (0.003) C19  
 \* -0.020 (0.004) C20  
 \* -0.001 (0.003) C21

Rms deviation of fitted atoms = 0.019

$$- 4.692 (0.017) x - 0.920 (0.030) y + 14.449 (0.016) z = 0.228 (0.016)$$

Angle to previous plane (with approximate esd) = 32.53 ( 0.20 )

\* -0.014 (0.003) C16  
 \* 0.008 (0.003) C17  
 \* 0.012 (0.004) C22  
 \* -0.002 (0.004) C23  
 \* -0.004 (0.004) C24  
 \* 0.001 (0.003) C25

Rms deviation of fitted atoms = 0.009

## Table to be deposited

**Table 1.** Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement coefficients ( $\text{\AA}^2 \times 10^3$ ) for the compound  $\text{OsH}\{\text{OC}(\text{OCH}_3)\text{CH}(\text{CO}_2\text{CH}_3)\text{CO}\}(\text{CO})(\text{P}^i\text{Pr}_3)_2$  (**3**).

Atom	X/a	Y/b	Z/c	$U_{eq}^a$
Os	1675 (1)	4789 (1)	2761 (1)	17 (1)
P(1)	235 (1)	2409 (1)	2025 (1)	20 (1)
P(2)	2712 (1)	7007 (1)	3755 (1)	20 (1)
O(1)	-385 (2)	5244 (2)	2312 (1)	23 (1)
O(2)	-2002 (2)	5866 (2)	1540 (1)	37 (1)
O(3)	2677 (2)	5512 (2)	1784 (1)	24 (1)
O(4)	4385 (3)	6293 (2)	766 (1)	43 (1)
O(5)	2711 (4)	7135 (5)	291 (3)	124 (2)
O(6)	4452 (2)	4012 (2)	3297 (1)	38 (1)
C(1)	-3197 (4)	5438 (4)	2016 (2)	45 (1)
C(2)	-571 (3)	5717 (3)	1738 (2)	28 (1)
C(3)	531 (4)	6132 (3)	1248 (2)	36 (1)
C(4)	2026 (3)	6006 (3)	1311 (2)	28 (1)
C(5)	3054 (4)	6549 (3)	729 (2)	38 (1)
C(6)	5429 (5)	6770 (3)	235 (2)	47 (1)
C(7)	3370 (3)	4340 (3)	3105 (2)	24 (1)
C(8)	-1972 (3)	2033 (3)	1691 (2)	32 (1)
C(9)	-2993 (4)	640 (3)	1051 (2)	57 (1)
C(10)	-2795 (4)	2257 (3)	2415 (2)	46 (1)
C(11)	489 (4)	1239 (3)	2622 (2)	27 (1)
C(12)	-421 (5)	-317 (3)	2200 (2)	45 (1)
C(13)	205 (5)	1599 (3)	3486 (2)	44 (1)
C(14)	834 (4)	1661 (3)	1081 (2)	29 (1)
C(15)	588 (5)	2368 (3)	442 (2)	44 (1)
C(16)	2610 (4)	1781 (4)	1252 (2)	47 (1)
C(17)	4279 (3)	8503 (3)	3567 (2)	29 (1)
C(18)	5555 (4)	8044 (3)	3177 (2)	41 (1)
C(19)	3564 (4)	9201 (3)	3033 (2)	41 (1)
C(20)	3687 (3)	7089 (3)	4784 (2)	30 (1)
C(21)	2638 (4)	5959 (4)	5106 (2)	44 (1)
C(22)	5407 (4)	7076 (3)	4858 (2)	40 (1)
C(23)	1091 (3)	7708 (3)	3964 (2)	25 (1)
C(24)	-440 (3)	6698 (3)	4125 (2)	33 (1)
C(25)	1681 (4)	9100 (3)	4637 (2)	38 (1)

<sup>a</sup> Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

## Table to be deposited

**Table 2.** Anisotropic displacement coefficients  $U_{ij}^*$  ( $\text{\AA}^2 \times 10^3$ ) for the non-hydrogen atoms for the compound  $\text{OsH}\{\text{OC}(\text{OCH}_3)\text{CH}(\text{CO}_2\text{CH}_3)\text{CO}\}(\text{CO})(\text{P}^i\text{Pr}_3)_2$  (**3**).

Atom	$U_{11}$	$U_{22}$	$U_{33}$	$U_{23}$	$U_{13}$	$U_{12}$
Os	17 (1)	18 (1)	17 (1)	6 (1)	4 (1)	7 (1)
P (1)	22 (1)	19 (1)	20 (1)	5 (1)	5 (1)	8 (1)
P (2)	20 (1)	19 (1)	21 (1)	5 (1)	5 (1)	7 (1)
O (1)	21 (1)	27 (1)	24 (1)	7 (1)	4 (1)	12 (1)
O (2)	29 (1)	47 (1)	44 (1)	19 (1)	4 (1)	23 (1)
O (3)	24 (1)	28 (1)	23 (1)	11 (1)	6 (1)	10 (1)
O (4)	54 (1)	50 (1)	43 (1)	27 (1)	30 (1)	26 (1)
O (5)	71 (2)	231 (5)	176 (4)	185 (4)	71 (2)	87 (3)
O (6)	33 (1)	49 (1)	40 (1)	12 (1)	2 (1)	26 (1)
C (1)	33 (2)	68 (2)	41 (2)	15 (2)	9 (1)	30 (2)
C (2)	25 (1)	24 (1)	32 (2)	6 (1)	0 (1)	12 (1)
C (3)	32 (2)	44 (2)	43 (2)	29 (2)	6 (1)	17 (1)
C (4)	29 (2)	27 (1)	27 (1)	10 (1)	4 (1)	7 (1)
C (5)	32 (2)	49 (2)	40 (2)	30 (2)	8 (1)	11 (1)
C (6)	61 (2)	43 (2)	44 (2)	19 (2)	35 (2)	17 (2)
C (7)	26 (1)	24 (1)	19 (1)	3 (1)	5 (1)	7 (1)
C (8)	21 (1)	25 (1)	43 (2)	5 (1)	1 (1)	5 (1)
C (9)	36 (2)	37 (2)	72 (3)	-8 (2)	-16 (2)	5 (2)
C (10)	25 (2)	43 (2)	70 (2)	17 (2)	18 (2)	9 (1)
C (11)	33 (2)	23 (1)	29 (1)	12 (1)	9 (1)	11 (1)
C (12)	69 (2)	25 (2)	43 (2)	15 (1)	11 (2)	16 (2)
C (13)	69 (2)	35 (2)	33 (2)	19 (1)	17 (2)	19 (2)
C (14)	40 (2)	24 (1)	21 (1)	4 (1)	8 (1)	14 (1)
C (15)	72 (2)	42 (2)	24 (2)	12 (1)	14 (2)	26 (2)
C (16)	48 (2)	67 (2)	38 (2)	14 (2)	24 (2)	32 (2)
C (17)	25 (1)	22 (1)	32 (2)	4 (1)	7 (1)	1 (1)
C (18)	26 (2)	39 (2)	49 (2)	7 (2)	15 (1)	2 (1)
C (19)	47 (2)	34 (2)	46 (2)	23 (1)	18 (2)	9 (2)
C (20)	33 (2)	30 (1)	22 (1)	3 (1)	-1 (1)	11 (1)
C (21)	43 (2)	62 (2)	29 (2)	24 (2)	4 (1)	17 (2)
C (22)	35 (2)	37 (2)	42 (2)	10 (1)	-6 (1)	10 (1)
C (23)	27 (1)	28 (1)	25 (1)	7 (1)	9 (1)	14 (1)
C (24)	29 (2)	42 (2)	33 (2)	11 (1)	13 (1)	16 (1)
C (25)	43 (2)	31 (2)	40 (2)	3 (1)	14 (1)	19 (1)

\* The anisotropic displacement exponent takes the form:  $-2\pi^2(h^2a^{*2}U_{11} + \dots + 2hka^*b^*U_{12})$ .

## Table to be deposited

**Table 3.** Hydrogen atom coordinates\* ( $\times 10^4$ ) and isotropic displacement coefficients ( $\text{\AA}^2 \times 10^3$ ) for the compound  $\text{OsH}\{\text{OC}(\text{OCH}_3)\text{CH}(\text{CO}_2\text{CH}_3)\text{CO}\}(\text{CO})(\text{P}i\text{Pr}_3)_2$  (**3**).

Atom	X/a	Y/b	Z/c	U
H**	822 (37)	4433 (31)	3425 (18)	40 (9)
H(1A)	-4187 (10)	5576 (21)	1818 (8)	53
H(1B)	-2755 (10)	5995 (15)	2587 (3)	53
H(1C)	-3459 (19)	4452 (7)	1966 (10)	53
H(3)**	222 (38)	6520 (32)	883 (20)	40 (9)
H(6A)	6494 (10)	6721 (21)	396 (8)	57
H(6B)	5568 (22)	7734 (8)	276 (10)	57
H(6C)	4934 (13)	6179 (14)	-326 (3)	57
H(8)	-2019 (3)	2760 (3)	1431 (2)	38
H(9A)	-2446 (15)	501 (12)	596 (7)	69
H(9B)	-4068 (11)	640 (10)	854 (11)	69
H(9C)	-3119 (25)	-115 (4)	1294 (4)	69
H(10A)	-2116 (13)	3140 (11)	2827 (6)	55
H(10B)	-2932 (24)	1492 (12)	2650 (8)	55
H(10C)	-3865 (12)	2281 (22)	2232 (3)	55
H(11)	1669 (4)	1374 (3)	2690 (2)	33
H(12A)	-170 (21)	-565 (5)	1661 (6)	54
H(12B)	-1596 (5)	-539 (5)	2143 (12)	54
H(12C)	-82 (20)	-844 (3)	2524 (7)	54
H(13A)	836 (20)	2586 (6)	3769 (4)	52
H(13B)	553 (24)	1033 (16)	3777 (4)	52
H(13C)	-956 (6)	1410 (20)	3467 (2)	52
H(14)	127 (4)	652 (3)	856 (2)	34
H(15A)	-533 (8)	2334 (19)	354 (9)	53
H(15B)	795 (25)	1885 (14)	-69 (4)	53
H(15C)	1346 (18)	3340 (7)	630 (6)	53
H(16A)	2744 (7)	1258 (19)	1627 (10)	57
H(16B)	3320 (5)	2762 (4)	1497 (12)	57
H(16C)	2902 (9)	1402 (21)	743 (3)	57
H(17)	4854 (3)	9218 (3)	4104 (2)	35
H(18A)	5895 (18)	7470 (17)	3465 (8)	49
H(18B)	6502 (11)	8866 (3)	3212 (11)	49
H(18C)	5080 (8)	7501 (18)	2606 (4)	49
H(19A)	2929 (21)	9672 (18)	3329 (5)	49
H(19B)	2855 (20)	8494 (4)	2534 (6)	49
H(19C)	4450 (4)	9883 (15)	2895 (10)	49
H(20)	3785 (3)	8000 (3)	5161 (2)	36

\* Hydrogen atoms were included in calculated positions and refined riding on their respective carbon atoms. Their displacement parameters were restricted to 1.2 times those of the carbon atoms to which they are bonded.

\*\* These hydrogens were refined as free isotropic atoms.

**Table 3 (cont.).** Hydrogen atom coordinates\* ( $\times 10^4$ ) and isotropic displacement coefficients ( $\text{\AA}^2 \times 10^3$ ) for the compound  $\text{OsH}\{\text{OC}(\text{OCH}_3)\text{CH}(\text{CO}_2\text{CH}_3)\text{CO}\}(\text{CO})_2$  (**3**).

<i>Atom</i>	<i>X/a</i>	<i>Y/b</i>	<i>Z/c</i>	<i>U</i>
H(21A)	1540 (8)	5980 (15)	5070 (12)	52
H(21B)	3128 (14)	6131 (13)	5673 (4)	52
H(21C)	2575 (21)	5046 (4)	4779 (8)	52
H(22A)	6113 (7)	7865 (12)	4713 (12)	48
H(22B)	5378 (5)	6205 (10)	4490 (9)	48
H(22C)	5834 (10)	7148 (21)	5418 (3)	48
H(23)	719 (3)	7898 (3)	3456 (2)	30
H(24A)	-757 (13)	5775 (6)	3731 (7)	40
H(24B)	-1331 (7)	7038 (10)	4072 (11)	40
H(24C)	-207 (7)	6637 (15)	4676 (4)	40
H(25A)	2699 (13)	9728 (8)	4559 (7)	45
H(25B)	1871 (23)	8947 (4)	5166 (2)	45
H(25C)	853 (11)	9516 (10)	4617 (8)	45

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\* Hydrogen atoms were included in calculated positions and refined riding on their respective carbon atoms. Their displacement parameters were restricted to 1.2 times those of the carbon atoms to which they are bonded.