Complex **1**

Crystal data. [C_{48} H_{56} N_2 Os]; formula weight = 851.15, trigonal, R 3 c, a = 36.550(5) Å, c = 17.147(3) Å, α= 90°, γ = 120°, V = 19838(5) Å^3, Z = 18, D_c = 1.282 gcm^{-3}, μ(Mo-Kα) = 2.924 mm^{-1}, F(000) = 7812, T = 301 K. A crystal of dimensions 0.20 × 0.10 ×0.05 mm mounted in a glass capillary was used for data collection at 20°C on a MAR diffractometer with a 300 mm image plate detector using graphite monochromatized Mo-K_α radiation (λ = 0.71073 Å). Data collection was made with 2° oscillation step of φ, 480 seconds exposure time and scanner distance at 120 mm. 78 images were collected. The images were interpreted and intensities integrated using program *DENZO* .[1] The structure was solved by direct methods employing *SIR-97* program [2] on PC. Os and many non-H atoms were located according to the direct methods and the successive least-square Fourier cycles. Positions of other non-hydrogen atoms were found after successful refinement by full-matrix least-squares using program SHELXL-97[3] on PC. Phenyl rings were restrained to be normal hexagon rings, with edge lengths of 1.39 Å. For convergence of least-square refinements, restrains were applied to $[NBu_4]^+$ cation, assuming that corresponding 1,2- and 1,3-bonds or distances were similar. Absolute structure was assisted by the Flack absolute structure parameter, equal to 0.007(17). 5591 independent reflections were obtained from a total 23043 reflections (R_{int} = 0.0519). 4082 reflections with $I \geq 2\ \sigma\ (I)$ were participated in the full-matrix least-square refinement against F^2. These reflections were in the range h: -40 to 39; k: -30 to 40; l: -18 to 18 with $2\theta_{max}$ = 49.24°. One crystallographic asymmetric unit consists of one formula unit, including one cation. In the final stage of least-squares refinement, atoms of cation and C atoms of phenyl rings were refined isotropically, other non-hydrogen atoms anisotropically. H atoms were generated by program SHELXL-97. The positions of H atoms were calculated based on riding mode with thermal parameters equal to 1.2 times that of the associated C atoms, and participated in the calculation of final R-indices.[4] Convergence ($(\Delta / \sigma)_{max}$ = 0.001, av. 0.001) for 227 variable parameters by full-matrix least-squares refinement on F^2 reaches to R_1 = 0.0434 and wR_2 = 0.1139 with a goodness-of-fit of 0.975, the parameters a and b for weighting scheme are 0.0773 and 0. The final difference Fourier map shows maximum rest peaks and holes of 0.404 and –0.421 e Å^{-3} respectively.

Ref:

1. *DENZO*: Otwinowski, Z. and Minor, W., "Processing of X-ray Diffraction Data Collected in Oscillation Mode", Methods in Enzymology, Volume 276: Macromolecular Crystallography, part A, p.307–326, 1997. Carter C. W., Sweet Jr. & R. M., Eds., Academic Press.
2. *SIR97*: Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *J. Appl. Crystallogr.* **1998**, *32*, 115.
3. SHELXL97: Sheldrick, G. M. (1997), A Program for Crystal Structure Analysis, University of Göttingen, Germany.
4. Since the structure refinements are against F^2, R-indices based on F^2 are larger than (more than double) those based on F. For comparison with older refinements based on F and an OMIT threshold, a conventional index R_1 based on observed F values larger than $4\sigma(F_o)$ is also given (corresponding to $I \geq 2\ \sigma\ (I)$). $wR_2 = \{\Sigma\ [w(F_o^2 - F_c^2)^2\ ] / \Sigma\ [w\ (F_o^2)^2]\}^{1/2}$, $R^1 = \Sigma\ ||F_o| - |F_c|| / \Sigma\ |F_o|$. The Goodness of Fit is always based on F^2: GooF = 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 total number of parameters refined. The weighting scheme is: $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where P is $[2\ F_c^2 + Max(F_o^2, 0)]$ /3.

Table 1. Crystal data and structure refinement for mar564.

Identification code	mar564	
Empirical formula	$C_{48} H_{56} N_2 Os$	
Formula weight	851.15	
Temperature	301(2) K	
Wavelength	0.71069 Å	
Crystal system	Trigonal	
Space group	R 3 c	
Unit cell dimensions	a = 36.550(5) Å	α= 90°.
	b = 36.550(5) Å	β= 90°.
	c = 17.147(3) Å	γ = 120°.
Volume	19838(5) Å^3	
Z	18	
Density (calculated)	1.282 Mg/m^3	
Absorption coefficient	2.924 mm^{-1}	
F(000)	7812	
Crystal size	0.2 x 0.1 x 0.05 mm^3	
Theta range for data collection	1.11 to 24.62°.	
Index ranges	-40<=h<=39, -30<=k<=40, -18<=l<=18	
Reflections collected	23043	
Independent reflections	5591 [R(int) = 0.0519]	
Completeness to theta = 24.62°	77.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5591 / 43 / 227	
Goodness-of-fit on F^2	0.975	
Final R indices [I>2sigma(I)]	R1 = 0.0434, wR2 = 0.1139	
R indices (all data)	R1 = 0.0636, wR2 = 0.1302	
Absolute structure parameter	0.007(17)	
Largest diff. peak and hole	0.404 and -0.421 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for mar564. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Os(1)	8100(1)	157(1)	-3716(1)	95(1)
N(1)	7868(4)	-182(4)	-4431(7)	121(4)
N(2)	8993(3)	1216(3)	-6228(6)	123(4)
C(1)	7625(5)	95(4)	-2991(8)	107(4)
C(2)	7371(4)	48(5)	-2521(7)	107(4)
C(3)	7065(3)	-3(4)	-1963(6)	119(4)
C(4)	6886(4)	-351(4)	-1469(8)	160(6)
C(5)	6588(4)	-393(4)	-920(7)	218(10)
C(6)	6469(4)	-87(5)	-864(8)	192(8)
C(7)	6649(5)	261(5)	-1358(9)	214(10)
C(8)	6947(4)	303(4)	-1908(7)	163(6)
C(9)	8191(4)	-185(4)	-2889(9)	100(4)
C(10)	8226(4)	-391(4)	-2372(8)	105(4)
C(11)	8239(4)	-628(3)	-1720(7)	121(4)
C(12)	8567(4)	-716(4)	-1683(8)	170(7)
C(13)	8601(5)	-931(5)	-1045(10)	229(11)
C(14)	8308(6)	-1058(4)	-443(8)	195(8)
C(15)	7980(5)	-970(5)	-480(7)	254(12)
C(16)	7945(4)	-754(4)	-1118(9)	185(8)
C(17)	8719(6)	431(5)	-3888(8)	112(4)
C(18)	9103(7)	617(5)	-3883(9)	121(5)
C(19)	9580(5)	829(5)	-3863(8)	144(6)
C(20)	9863(6)	1243(4)	-3647(7)	170(7)
C(21)	10295(5)	1388(5)	-3642(10)	217(10)
C(22)	10443(5)	1118(8)	-3851(13)	263(14)
C(23)	10160(8)	704(8)	-4067(13)	360(20)
C(24)	9729(7)	559(4)	-4072(11)	330(20)
C(25)	8125(4)	698(5)	-4033(7)	111(4)
C(26)	8135(4)	1030(5)	-4273(7)	105(4)
C(27)	8109(3)	1386(3)	-4514(5)	102(4)
C(28)	7750(3)	1312(3)	-4925(6)	147(6)

C(29)	7714(3)	1650(4)	-5204(6)	168(7)
C(30)	8037(4)	2062(3)	-5073(7)	166(7)
C(31)	8395(3)	2136(3)	-4662(7)	146(5)
C(32)	8431(3)	1798(3)	-4382(5)	132(5)
C(33)	8574(4)	825(4)	-6007(8)	131(5)
C(34)	8373(4)	506(5)	-6669(9)	146(5)
C(35)	7909(6)	114(6)	-6431(11)	170(7)
C(36)	7721(8)	-198(7)	-6970(15)	217(10)
C(37)	9180(4)	1483(4)	-5500(8)	128(4)
C(38)	9587(6)	1893(5)	-5607(12)	191(8)
C(39)	9704(10)	2178(9)	-4782(16)	295(17)
C(40)	10071(15)	2527(14)	-4770(30)	540(50)
C(41)	9295(5)	1101(6)	-6624(9)	152(6)
C(42)	9430(7)	849(7)	-6161(11)	186(8)
C(43)	9657(11)	647(10)	-6692(15)	272(14)
C(44)	9776(12)	407(11)	-6340(20)	319(18)
C(45)	8905(5)	1468(5)	-6861(9)	141(5)
C(46)	8627(8)	1636(8)	-6588(13)	235(11)
C(47)	8523(10)	1893(9)	-7243(17)	267(13)
C(48)	8620(20)	2284(12)	-7070(30)	540(50)

Table 3. Bond lengths [Å] and angles [°] for mar564.

Os(1)-N(1)	1.645(11)	C(26)-C(27)	1.416(17)
Os(1)-C(17)	1.983(17)	C(27)-C(28)	1.3900
Os(1)-C(25)	2.011(15)	C(27)-C(32)	1.3900
Os(1)-C(9)	2.024(16)	C(28)-C(29)	1.3900
Os(1)-C(1)	2.056(17)	C(29)-C(30)	1.3900
N(2)-C(37)	1.519(12)	C(30)-C(31)	1.3900
N(2)-C(41)	1.527(12)	C(31)-C(32)	1.3900
N(2)-C(33)	1.530(12)	C(33)-C(34)	1.526(15)
N(2)-C(45)	1.556(12)	C(34)-C(35)	1.632(18)
C(1)-C(2)	1.173(18)	C(35)-C(36)	1.36(2)
C(2)-C(3)	1.410(16)	C(37)-C(38)	1.504(16)
C(3)-C(4)	1.3900	C(38)-C(39)	1.68(2)
C(3)-C(8)	1.3900	C(39)-C(40)	1.31(3)
C(4)-C(5)	1.3900	C(41)-C(42)	1.475(16)
C(5)-C(6)	1.3900	C(42)-C(43)	1.63(2)
C(6)-C(7)	1.3900	C(43)-C(44)	1.31(2)
C(7)-C(8)	1.3900	C(45)-C(46)	1.501(16)
C(9)-C(10)	1.211(18)	C(46)-C(47)	1.62(2)
C(10)-C(11)	1.428(16)	C(47)-C(48)	1.32(3)
C(11)-C(12)	1.3900		
C(11)-C(16)	1.3900	N(1)-Os(1)-C(17)	107.4(6)
C(12)-C(13)	1.3900	N(1)-Os(1)-C(25)	106.9(6)
C(13)-C(14)	1.3900	C(17)-Os(1)-C(25)	89.1(5)
C(14)-C(15)	1.3900	N(1)-Os(1)-C(9)	103.9(6)
C(15)-C(16)	1.3900	C(17)-Os(1)-C(9)	85.4(5)
C(17)-C(18)	1.22(2)	C(25)-Os(1)-C(9)	148.9(5)
C(18)-C(19)	1.51(2)	N(1)-Os(1)-C(1)	106.1(6)
C(19)-C(20)	1.3900	C(17)-Os(1)-C(1)	146.4(6)
C(19)-C(24)	1.3900	C(25)-Os(1)-C(1)	83.8(5)
C(20)-C(21)	1.3900	C(9)-Os(1)-C(1)	84.2(5)
C(21)-C(22)	1.3900	C(37)-N(2)-C(41)	112.6(10)
C(22)-C(23)	1.3900	C(37)-N(2)-C(33)	108.4(9)
C(23)-C(24)	1.3900	C(41)-N(2)-C(33)	112.0(10)
C(25)-C(26)	1.262(16)	C(37)-N(2)-C(45)	110.8(10)

C(41)-N(2)-C(45)	104.2(10)	C(21)-C(20)-C(19)	120.0
C(33)-N(2)-C(45)	108.7(10)	C(20)-C(21)-C(22)	120.0
C(2)-C(1)-Os(1)	173.4(13)	C(23)-C(22)-C(21)	120.0
C(1)-C(2)-C(3)	179.0(16)	C(22)-C(23)-C(24)	120.0
C(4)-C(3)-C(8)	120.0	C(23)-C(24)-C(19)	120.0
C(4)-C(3)-C(2)	121.0(11)	C(26)-C(25)-Os(1)	176.5(12)
C(8)-C(3)-C(2)	119.0(11)	C(25)-C(26)-C(27)	174.7(13)
C(3)-C(4)-C(5)	120.0	C(28)-C(27)-C(32)	120.0
C(6)-C(5)-C(4)	120.0	C(28)-C(27)-C(26)	117.5(9)
C(7)-C(6)-C(5)	120.0	C(32)-C(27)-C(26)	122.5(9)
C(6)-C(7)-C(8)	120.0	C(27)-C(28)-C(29)	120.0
C(7)-C(8)-C(3)	120.0	C(30)-C(29)-C(28)	120.0
C(10)-C(9)-Os(1)	176.4(11)	C(31)-C(30)-C(29)	120.0
C(9)-C(10)-C(11)	174.4(14)	C(32)-C(31)-C(30)	120.0
C(12)-C(11)-C(16)	120.0	C(31)-C(32)-C(27)	120.0
C(12)-C(11)-C(10)	118.0(11)	C(34)-C(33)-N(2)	113.8(10)
C(16)-C(11)-C(10)	122.0(11)	C(33)-C(34)-C(35)	112.2(11)
C(13)-C(12)-C(11)	120.0	C(36)-C(35)-C(34)	115.8(16)
C(12)-C(13)-C(14)	120.0	C(38)-C(37)-N(2)	116.3(11)
C(15)-C(14)-C(13)	120.0	C(37)-C(38)-C(39)	109.5(13)
C(14)-C(15)-C(16)	120.0	C(40)-C(39)-C(38)	116(2)
C(15)-C(16)-C(11)	120.0	C(42)-C(41)-N(2)	116.6(12)
C(18)-C(17)-Os(1)	170.7(13)	C(41)-C(42)-C(43)	112.6(14)
C(17)-C(18)-C(19)	177.2(15)	C(44)-C(43)-C(42)	117(2)
C(20)-C(19)-C(24)	120.0	C(46)-C(45)-N(2)	113.9(11)
C(20)-C(19)-C(18)	127.1(13)	C(45)-C(46)-C(47)	115.0(15)
C(24)-C(19)-C(18)	112.9(13)	C(48)-C(47)-C(46)	117(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for mar564. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

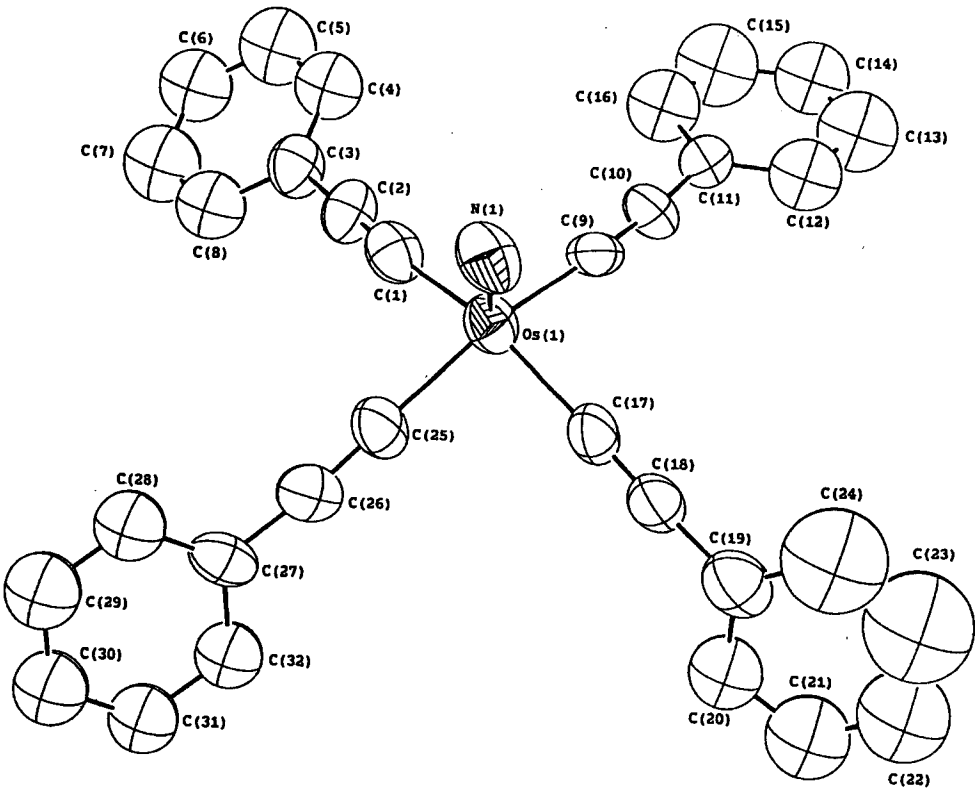
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Os(1)	116(1)	97(1)	87(1)	-2(1)	9(1)	66(1)
N(1)	165(11)	108(9)	116(8)	-38(6)	-10(7)	87(8)
C(1)	136(12)	99(9)	99(9)	-22(7)	-16(8)	68(9)
C(2)	105(10)	134(11)	95(9)	-29(7)	-3(7)	71(9)
C(3)	123(11)	130(12)	120(10)	-14(8)	9(8)	75(10)
C(9)	84(8)	63(7)	139(10)	6(7)	36(8)	24(7)
C(10)	127(11)	87(9)	110(9)	18(7)	3(7)	61(8)
C(11)	90(9)	95(9)	172(14)	1(9)	-13(9)	42(8)
C(17)	123(12)	116(11)	127(10)	39(8)	47(9)	82(10)
C(18)	139(14)	114(11)	136(11)	45(8)	63(10)	82(11)
C(19)	185(18)	135(13)	128(11)	24(10)	41(11)	92(13)
C(25)	129(11)	110(10)	120(10)	15(8)	37(8)	79(10)
C(26)	122(11)	100(10)	93(8)	-2(7)	37(7)	56(9)
C(27)	133(11)	83(8)	83(7)	10(6)	22(6)	49(8)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for mar564.

	x	y	z	U(eq)
H(4)	6965	-556	-1507	192
H(5)	6467	-626	-589	262
H(6)	6270	-115	-496	231
H(7)	6570	465	-1321	257
H(8)	7067	536	-2238	195
H(12)	8763	-631	-2086	204
H(13)	8821	-990	-1020	275
H(14)	8331	-1202	-16	234
H(15)	7784	-1054	-77	305
H(16)	7726	-695	-1143	222
H(20)	9764	1424	-3507	204
H(21)	10484	1665	-3498	261
H(22)	10732	1214	-3847	316
H(23)	10259	523	-4207	433
H(24)	9540	282	-4216	399
H(28)	7534	1037	-5013	177
H(29)	7474	1600	-5479	201
H(30)	8013	2288	-5259	199
H(31)	8611	2411	-4573	175
H(32)	8671	1847	-4107	158
H(33A)	8623	686	-5569	157
H(33B)	8378	914	-5838	157
H(34A)	8557	397	-6808	175
H(34B)	8344	649	-7123	175
H(35A)	7938	-9	-5951	203
H(35B)	7724	226	-6324	203
H(36A)	7452	-413	-6774	325
H(36B)	7897	-317	-7073	325
H(36C)	7679	-83	-7443	325
H(37A)	8973	1545	-5281	153

H(37B)	9228	1314	-5120	153
H(38A)	9560	2050	-6036	229
H(38B)	9814	1836	-5730	229
H(39A)	9485	2251	-4695	354
H(39B)	9693	2001	-4351	354
H(40A)	10113	2658	-4263	809
H(40B)	10078	2717	-5160	809
H(40C)	10291	2463	-4863	809
H(41A)	9162	945	-7097	182
H(41B)	9546	1360	-6779	182
H(42A)	9186	622	-5903	223
H(42B)	9626	1027	-5761	223
H(43A)	9463	483	-7107	326
H(43B)	9904	879	-6936	326
H(44A)	9895	301	-6714	478
H(44B)	9536	174	-6096	478
H(44C)	9984	568	-5951	478
H(45A)	9172	1703	-7034	170
H(45B)	8773	1285	-7307	170
H(46A)	8762	1822	-6147	282
H(46B)	8362	1401	-6405	282
H(47A)	8673	1903	-7716	321
H(47B)	8224	1731	-7359	321
H(48A)	8588	2418	-7529	816
H(48B)	8904	2440	-6888	816
H(48C)	8430	2277	-6672	816

Perspective view of [$^{n}Bu_4N$][OsN(C≡CPh)$_4$], [$^{n}Bu_4N$]**1** (30 % thermal ellipsoids)



Complex **2**

Crystal data. [C_{56} H_{72} N_2 Os]; formula weight = 963.36, orthorhombic, *P nma*, *a* = 15.860(3) Å, *b* = 26.132(5) Å, *c* = 12.774(3) Å, *V* = 5294.2(18) Å^3, *Z* = 4, D_c = 1.209 gcm^{-3}, μ(Mo-Kα) = 2.443 mm^{-1}, *F*(000) = 1992, *T* = 301 K. A crystal of dimensions 0.25 × 0.25 × 0.15 mm mounted on a glass fibre was used for data collection at 20°C on a MAR diffractometer with a 300 mm image plate detector using graphite monochromatized Mo-K_α radiation (λ = 0.71073 Å). Data collection was made with 2° oscillation step of φ, 480 seconds exposure time and scanner distance at 120 mm. 85 images were collected. The images were interpreted and intensities integrated using program *DENZO* .[1] The structure was solved by direct methods employing *SIR-97* program [2] on PC. Os and many non-H atoms were located according to the direct methods and the successive least-square Fourier cycles. Positions of other non-hydrogen atoms were found after successful refinement by full-matrix least-squares using program SHELXL-97[3] on PC. Two phenyl rings were restrained to be normal hexagon rings with edge lengths of 1.39 Å. For the n-butyl groups, restrains were applied to assume similar 1,2 and 1,3-distances, respectively. 3761 independent reflections were obtained from a total 21249 reflections (R_{int} = 0.0393). 2624 reflections with $I \geq 2\ \sigma\ (I)$ were participated in the full-matrix least-square refinement against F^2. These reflections were in the range *h*: -19 to 18; *k*: -27 to 27; *l*: -14 to 14 with $2\theta_{max}$ = 50.62°. One crystallographic asymmetric unit consists of half of formula unit. In the final stage of least-squares refinement, atoms of cation and Et-groups were refined isotropically, other non-hydrogen atoms anisotropically. H atoms were generated by program SHELXL-97. The positions of H atoms were calculated based on riding mode with thermal parameters equal to 1.2 times that of the associated C atoms, and participated in the calculation of final R-indices.[4] Convergence $((\Delta / \sigma)_{max}$ = 0.001, av. 0.001) for 193 variable parameters by full-matrix least-squares refinement on F^2 reaches to R_1 = 0.0515 and wR_2 = 0.1477 with a goodness-of-fit of 0.987, the parameters a and b for weighting scheme are 0.1124 and 0. The final difference Fourier map shows maximum rest peaks and holes of 0.781 and –0.834 e Å^{-3} respectively.

Ref:

1. *DENZO*: Otwinowski, Z. and Minor, W., "Processing of X-ray Diffraction Data Collected in Oscillation Mode", Methods in Enzymology, Volume 276: Macromolecular Crystallography, part A, p.307–326, 1997. Carter C. W., Sweet Jr. & R. M., Eds., Academic Press.
2. *SIR97*: Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *J. Appl. Crystallogr.* **1998**, *32*, 115.
3. SHELXL97: Sheldrick, G. M. (1997), A Program for Crystal Structure Analysis, University of Göttingen, Germany.
4. Since the structure refinements are against F^2, R-indices based on F^2 are larger than (more than double) those based on *F*. For comparison with older refinements based on *F* and an OMIT threshold, a conventional index R_1 based on observed *F* values larger than $4\sigma(F_o)$ is also given (corresponding to $I \geq 2\ \sigma\ (I)$). $wR_2 = \{\Sigma\ [w(F_o^2 - F_c^2)^2\ ] / \Sigma\ [w\ (F_o^2)^2]\}^{1/2}$, $R^1 = \Sigma\ ||F_o| - |F_c|| / \Sigma\ |F_o|$. The Goodness of Fit is always based on F^2: GooF = 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 total number of parameters refined. The weighting scheme is: $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ where P is $[2\ F_c^2 + Max(F_o^2, 0)]$ /3.

Table 1. Crystal data and structure refinement for mar554a.

Identification code	mar554a	
Empirical formula	$C_{56} H_{72} N_2 Os$	
Formula weight	963.36	
Temperature	301(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P n m a	
Unit cell dimensions	a = 15.860(3) Å	α= 90°.
	b = 26.132(5) Å	β= 90°.
	c = 12.774(3) Å	γ = 90°.
Volume	5294.2(18) $Å^3$	
Z	4	
Density (calculated)	1.209 Mg/m^3	
Absorption coefficient	2.443 mm^{-1}	
F(000)	1992	
Crystal size	0.25 x 0.25 x 0.15 mm^3	
Theta range for data collection	1.77 to 25.31°.	
Index ranges	-19<=h<=18, -27<=k<=27, -14<=l<=14	
Reflections collected	21249	
Independent reflections	3761 [R(int) = 0.0393]	
Completeness to theta = 25.31°	76.2 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3761 / 21 / 193	
Goodness-of-fit on F^2	0.987	
Final R indices [I>2sigma(I)]	R1 = 0.0515, wR2 = 0.1477	
R indices (all data)	R1 = 0.0696, wR2 = 0.1561	
Largest diff. peak and hole	0.781 and -0.834 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for mar554a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Os(1)	7784(1)	2500	231(1)	99(1)
N(1)	8723(5)	2500	732(8)	123(3)
N(2)	4859(5)	2500	-1158(7)	123(3)
C(1)	7089(5)	3007(3)	1050(7)	109(2)
C(2)	6607(5)	3283(3)	1517(6)	116(2)
C(3)	6018(4)	3572(2)	2136(4)	116(2)
C(4)	5440(5)	3314(2)	2760(6)	176(4)
C(5)	4861(4)	3588(3)	3354(6)	188(5)
C(6)	4859(4)	4120(3)	3323(6)	163(4)
C(7)	5437(6)	4378(2)	2698(7)	195(5)
C(8)	6017(4)	4104(2)	2105(6)	169(4)
C(9)	4158(10)	4402(5)	3972(12)	239(6)
C(10)	4430(30)	4293(11)	5104(19)	377(15)
C(11)	7800(4)	1981(3)	-951(7)	106(2)
C(12)	7739(4)	1695(3)	-1670(8)	113(2)
C(13)	7675(3)	1371(2)	-2612(5)	114(2)
C(14)	7921(4)	861(3)	-2564(6)	152(3)
C(15)	7864(5)	554(2)	-3450(8)	171(5)
C(16)	7562(5)	757(4)	-4383(7)	168(5)
C(17)	7317(5)	1267(4)	-4431(5)	171(4)
C(18)	7374(4)	1574(2)	-3545(6)	142(3)
C(19)	7497(16)	445(10)	-5336(15)	239(7)
C(20)	8062(14)	591(7)	-6254(18)	299(10)
C(21)	5391(5)	2056(3)	-984(8)	143(3)
C(22)	5008(7)	1525(4)	-1111(11)	196(4)
C(23)	5585(8)	1103(4)	-997(12)	209(5)
C(24)	5267(12)	621(5)	-1145(16)	297(8)
C(25)	4110(8)	2500	-478(9)	132(4)
C(26)	4315(12)	2500	696(12)	229(8)
C(27)	3752(19)	2500	1508(16)	720(50)
C(28)	2912(15)	2500	1360(30)	348(17)

C(29)	4523(8)	2500	-2242(9)	147(4)
C(30)	5167(13)	2500	-3104(13)	237(8)
C(31)	5268(16)	2781(8)	-3974(16)	221(11)
C(32)	5700(20)	2500	-4808(17)	277(13)

Table 3. Bond lengths [Å] and angles [°] for mar554a.

Os(1)-N(1)	1.620(8)	C(27)-C(28)	1.35(2)
Os(1)-C(1)	2.016(9)	C(29)-C(30)	1.501(15)
Os(1)-C(1)#1	2.016(9)	C(30)-C(31)#1	1.341(18)
Os(1)-C(11)	2.030(9)	C(30)-C(31)	1.341(18)
Os(1)-C(11)#1	2.030(9)	C(31)-C(32)	1.461(19)
N(2)-C(21)#1	1.451(8)	C(31)-C(31)#1	1.47(4)
N(2)-C(21)	1.451(8)	C(32)-C(31)#1	1.461(19)
N(2)-C(25)	1.471(11)		
N(2)-C(29)	1.484(11)	N(1)-Os(1)-C(1)	107.3(3)
C(1)-C(2)	1.210(10)	N(1)-Os(1)-C(1)#1	107.3(3)
C(2)-C(3)	1.437(10)	C(1)-Os(1)-C(1)#1	82.1(5)
C(3)-C(4)	1.3900	N(1)-Os(1)-C(11)	106.4(3)
C(3)-C(8)	1.3900	C(1)-Os(1)-C(11)	146.3(3)
C(4)-C(5)	1.3900	C(1)#1-Os(1)-C(11)	87.4(3)
C(5)-C(6)	1.3900	N(1)-Os(1)-C(11)#1	106.4(3)
C(6)-C(7)	1.3900	C(1)-Os(1)-C(11)#1	87.4(3)
C(6)-C(9)	1.571(15)	C(1)#1-Os(1)-C(11)#1	146.3(3)
C(7)-C(8)	1.3900	C(11)-Os(1)-C(11)#1	83.9(5)
C(9)-C(10)	1.54(3)	C(21)#1-N(2)-C(21)	106.1(8)
C(11)-C(12)	1.188(11)	C(21)#1-N(2)-C(25)	112.3(6)
C(12)-C(13)	1.474(11)	C(21)-N(2)-C(25)	112.3(6)
C(13)-C(14)	1.3900	C(21)#1-N(2)-C(29)	110.5(6)
C(13)-C(18)	1.3900	C(21)-N(2)-C(29)	110.5(6)
C(14)-C(15)	1.3900	C(25)-N(2)-C(29)	105.1(9)
C(15)-C(16)	1.3900	C(2)-C(1)-Os(1)	173.8(7)
C(16)-C(17)	1.3900	C(1)-C(2)-C(3)	174.6(8)
C(16)-C(19)	1.47(2)	C(4)-C(3)-C(8)	120.0
C(17)-C(18)	1.3900	C(4)-C(3)-C(2)	119.3(5)
C(19)-C(20)	1.52(2)	C(8)-C(3)-C(2)	120.7(5)
C(21)-C(22)	1.523(11)	C(3)-C(4)-C(5)	120.0
C(22)-C(23)	1.442(13)	C(6)-C(5)-C(4)	120.0
C(23)-C(24)	1.369(16)	C(5)-C(6)-C(7)	120.0
C(25)-C(26)	1.535(15)	C(5)-C(6)-C(9)	117.2(7)
C(26)-C(27)	1.369(19)	C(7)-C(6)-C(9)	122.8(7)

C(6)-C(7)-C(8)	120.0	C(16)-C(19)-C(20)	117.2(19)
C(7)-C(8)-C(3)	120.0	N(2)-C(21)-C(22)	118.7(7)
C(10)-C(9)-C(6)	102.1(17)	C(23)-C(22)-C(21)	115.7(9)
C(12)-C(11)-Os(1)	174.0(7)	C(24)-C(23)-C(22)	117.1(12)
C(11)-C(12)-C(13)	175.9(9)	N(2)-C(25)-C(26)	113.9(10)
C(14)-C(13)-C(18)	120.0	C(27)-C(26)-C(25)	127.0(15)
C(14)-C(13)-C(12)	119.7(6)	C(28)-C(27)-C(26)	122(2)
C(18)-C(13)-C(12)	120.3(6)	N(2)-C(29)-C(30)	116.2(11)
C(13)-C(14)-C(15)	120.0	C(31)#1-C(30)-C(31)	66(2)
C(16)-C(15)-C(14)	120.0	C(31)#1-C(30)-C(29)	133.6(16)
C(15)-C(16)-C(17)	120.0	C(31)-C(30)-C(29)	133.6(16)
C(15)-C(16)-C(19)	121.5(13)	C(30)-C(31)-C(32)	112.7(17)
C(17)-C(16)-C(19)	118.5(13)	C(30)-C(31)-C(31)#1	56.8(10)
C(18)-C(17)-C(16)	120.0	C(32)-C(31)-C(31)#1	59.9(9)
C(17)-C(18)-C(13)	120.0	C(31)#1-C(32)-C(31)	60.3(18)

Symmetry transformations used to generate equivalent atoms:

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for mar554a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Os(1)	77(1)	122(1)	98(1)	0	-2(1)	0
N(1)	79(5)	154(6)	136(7)	0	-23(5)	0
C(1)	102(5)	130(6)	96(6)	-4(4)	-8(4)	-5(4)
C(2)	118(6)	136(5)	94(5)	-5(4)	-7(5)	4(5)
C(3)	123(6)	120(5)	105(5)	-15(4)	-2(5)	26(5)
C(4)	224(11)	141(6)	163(9)	-7(6)	92(9)	-10(7)
C(5)	198(10)	164(8)	203(12)	-20(7)	97(10)	-17(7)
C(6)	165(8)	141(7)	183(10)	-49(7)	42(8)	34(6)
C(7)	250(14)	135(7)	200(12)	-8(7)	59(11)	1(8)
C(8)	217(11)	112(6)	179(10)	-14(5)	56(9)	4(6)
C(11)	93(5)	122(5)	105(7)	1(5)	17(4)	-4(4)
C(12)	81(5)	145(6)	113(7)	-8(6)	14(4)	-4(4)
C(13)	90(5)	121(5)	132(8)	-15(5)	25(5)	-13(4)
C(14)	178(8)	138(7)	141(9)	-6(7)	-6(7)	1(6)
C(15)	202(12)	136(7)	175(12)	-42(8)	2(9)	-9(6)
C(16)	177(10)	174(11)	153(11)	-60(10)	19(10)	-51(9)
C(17)	174(11)	223(15)	116(8)	-21(10)	-17(7)	-26(9)
C(18)	149(7)	169(7)	109(8)	0(7)	12(6)	6(6)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for mar554a.

	x	y	z	U(eq)
H(4)	5442	2959	2781	211
H(5)	4474	3415	3772	226
H(7)	5435	4734	2677	234
H(8)	6403	4277	1687	203
H(9A)	4156	4767	3827	287
H(9B)	3604	4263	3827	287
H(10A)	4044	4455	5578	565
H(10B)	4987	4425	5217	565
H(10C)	4429	3930	5223	565
H(14)	8123	725	-1940	183
H(15)	8028	213	-3418	205
H(17)	7115	1403	-5055	206
H(18)	7209	1916	-3577	171
H(19A)	6917	457	-5574	286
H(19B)	7619	93	-5150	286
H(20A)	7932	379	-6845	448
H(20B)	8642	544	-6061	448
H(20C)	7968	944	-6434	448
H(21A)	5615	2080	-279	171
H(21B)	5866	2081	-1460	171
H(22A)	4562	1486	-599	235
H(22B)	4752	1504	-1800	235
H(23A)	6044	1152	-1488	251
H(23B)	5824	1118	-298	251
H(24A)	5714	375	-1090	446
H(24B)	5016	599	-1828	446
H(24C)	4847	552	-623	446
H(25A)	3772	2200	-638	158
H(25B)	3772	2800	-638	158
H(26A)	4671	2203	808	275

H(26B)	4671	2797	808	275
H(27A)	3880	2202	1933	865
H(27B)	3880	2798	1933	865
H(28A)	2630	2500	2021	521
H(28B)	2754	2200	969	521
H(28C)	2754	2800	969	521
H(29A)	4168	2799	-2327	177
H(29B)	4168	2201	-2327	177

Complex **4**

Crystal data. [C_{72} H_{72} N_2 Os]; formula weight = 1155.52, monoclinic, $P\ 2_1/n$, a = 11.134(2) Å, b = 16.242(3) Å, c = 33.898(7) Å, β = 92.78(3)°, V = 6123(2) Å^3, Z = 4, D_c = 1.254 gcm^{-3}, μ(Mo-Kα) = 2.124 mm^{-1}, F(000) = 2376, T = 301 K. A crystal of dimensions 0.40 × 0.30 × 0.15 mm mounted in a glass capillary was used for data collection at 20°C on a MAR diffractometer with a 300 mm image plate detector using graphite monochromatized Mo-K$_\alpha$ radiation (λ = 0.71073 Å). Data collection was made with 3° oscillation step of φ, 300 seconds exposure time and scanner distance at 120 mm. 60 images were collected. The images were interpreted and intensities integrated using program *DENZO* .[1] The structure was solved by direct methods employing *SIR-97* program [2] on PC. Os and many non-H atoms were located according to the direct methods and the successive least-square Fourier cycles. Positions of other non-hydrogen atoms were found after successful refinement by full-matrix least-squares using program SHELXL-97[3] on PC. Cation ^{n}Bu$_4$N$^+$ were restrained to have similar 1,2- and 1,3-bond lengths or distances, respectively. Three of four terminal phenyl rings were disordered and were treated with two or three sets of positions. All phenyl rings were restrained to be normal hexagon rings. 6673 independent reflections were obtained from a total 16197 reflections (R_{int} = 0.0397). 4021 reflections with $I \geq 2\ \sigma\ (I)$ were participated in the full-matrix least-square refinement against F^2. These reflections were in the range h: -10 to 10; k: -19 to 17; l: -38 to 38 with $2\theta_{max}$ = 50.68°. One crystallographic asymmetric unit consists of one formula unit, including one cation. In the final stage of least-squares refinement, atoms of disordered fragments were refined isotropically, other non-hydrogen atoms anisotropically. H atoms were generated by program SHELXL-97. The positions of H atoms were calculated based on riding mode with thermal parameters equal to 1.2 times that of the associated C atoms, and participated in the calculation of final R-indices.[4] Convergence ((Δ / σ)$_{max}$ = 0.001, av. 0.001) for 532 variable parameters by full-matrix least-squares refinement on F^2 reaches to R_1 = 0.0452 and wR_2 = 0.1177 with a goodness-of-fit of 0.975, the parameters a and b for weighting scheme are 0.0744 and 0. The final difference Fourier map shows maximum rest peaks and holes of 0.512 and –0.529 e Å^{-3} respectively.

Ref:

1. *DENZO*: Otwinowski, Z. and Minor, W., "Processing of X-ray Diffraction Data Collected in Oscillation Mode", Methods in Enzymology, Volume 276: Macromolecular Crystallography, part A, p.307–326, 1997. Carter C. W., Sweet Jr. & R. M., Eds., Academic Press.
2. *SIR97*: Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *J. Appl. Crystallogr.* **1998**, *32*, 115.
3. SHELXL97: Sheldrick, G. M. (1997), A Program for Crystal Structure Analysis, University of Göttingen, Germany.
4. Since the structure refinements are against F^2, R-indices based on F^2 are larger than (more than double) those based on F. For comparison with older refinements based on F and an OMIT threshold, a conventional index R_1 based on observed F values larger than $4\sigma(F_o)$ is also given (corresponding to $I \geq 2\ \sigma\ (I)$). wR_2 = {Σ [$w(F_o^2 - F_c^2)^2$] / Σ [$w\ (F_o^2)^2$]}$^{1/2}$, R^1 = Σ ||F_o| – |F_c|| / Σ |F_o|. The Goodness of Fit is always based on F^2: GooF = S= {Σ[$w(F_o^2 - F_c^2)^2$] / (n – p)}$^{1/2}$ where n is the number of reflections and p is the total number of parameters refined. The weighting scheme is: w = 1/[$\sigma^2(F_o^2)$ + $(aP)^2$ + bP] where P is [2 F_c^2 + Max(F_o^2, 0)] /3.

Table 1. Crystal data and structure refinement for mar616.

Identification code	mar616	
Empirical formula	C_{72} H_{72} N_2 Os	
Formula weight	1155.52	
Temperature	301(2) K	
Wavelength	0.71069 Å	
Crystal system	Monoclinic	
Space group	P $2_1/n$	
Unit cell dimensions	a = 11.134(2) Å	α= 90°.
	b = 16.242(3) Å	β= 92.78(3)°.
	c = 33.898(7) Å	γ = 90°.
Volume	6123(2) Å^3	
Z	4	
Density (calculated)	1.254 Mg/m^3	
Absorption coefficient	2.124 mm^{-1}	
F(000)	2376	
Crystal size	0.4 x 0.3 x 0.15 mm^3	
Theta range for data collection	1.39 to 25.34°.	
Index ranges	-10<=h<=10, -19<=k<=17, -38<=l<=38	
Reflections collected	16197	
Independent reflections	6673 [R(int) = 0.0397]	
Completeness to theta = 25.34°	59.5 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6673 / 49 / 532	
Goodness-of-fit on F^2	0.975	
Final R indices [I>2sigma(I)]	R1 = 0.0452, wR2 = 0.1177	
R indices (all data)	R1 = 0.0807, wR2 = 0.1424	
Largest diff. peak and hole	0.512 and -0.529 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for mar616. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Os(1)	10337(1)	-3243(1)	7107(1)	74(1)
N(1)	9833(8)	-4178(4)	7106(3)	105(3)
N(2)	10167(7)	98(4)	7506(2)	92(3)
C(1)	11613(10)	-3178(5)	7571(4)	80(3)
C(2)	12267(10)	-3113(5)	7852(3)	77(3)
C(3)	12998(7)	-3091(4)	8206(2)	74(3)
C(4)	14147(8)	-2756(3)	8212(2)	89(3)
C(5)	14853(5)	-2747(3)	8562(3)	92(4)
C(6)	14410(7)	-3074(4)	8905(2)	89(4)
C(7)	13261(7)	-3410(3)	8898(2)	90(3)
C(8)	12555(5)	-3418(3)	8548(2)	88(3)
C(9)	15195(14)	-3037(4)	9268(3)	101(4)
C(10)	16425(15)	-3167(5)	9249(4)	155(7)
C(11)	17166(8)	-3124(6)	9590(6)	211(11)
C(12)	16677(12)	-2951(5)	9951(4)	212(13)
C(13)	15447(14)	-2821(5)	9970(3)	179(8)
C(14)	14706(8)	-2864(5)	9628(4)	136(6)
C(15)	9299(9)	-2641(5)	7496(4)	78(3)
C(16)	8754(9)	-2292(5)	7753(4)	76(3)
C(17)	8180(6)	-1890(3)	8050(2)	72(3)
C(18)	8683(5)	-1916(3)	8433(2)	85(3)
C(19)	8194(7)	-1444(4)	8728(2)	98(4)
C(20)	7202(7)	-946(3)	8639(2)	88(4)
C(21)	6699(5)	-921(3)	8256(3)	91(4)
C(22)	7188(6)	-1393(4)	7961(2)	81(3)
C(23)	6375(14)	-466(8)	8895(4)	91(8)
C(24)	5995(15)	332(9)	8809(4)	101(9)
C(25)	5361(12)	773(6)	9082(5)	80(6)
C(26)	5107(13)	416(9)	9441(4)	132(9)
C(27)	5487(14)	-382(9)	9527(4)	127(9)
C(28)	6121(13)	-823(6)	9254(5)	84(6)

C(23')	6832(14)	-402(8)	8979(5)	71(6)
C(24')	6373(15)	380(9)	8899(5)	96(8)
C(25')	6053(15)	886(8)	9207(8)	156(11)
C(26')	6192(19)	612(13)	9595(6)	245(19)
C(27')	6651(19)	-170(15)	9675(5)	260(20)
C(28')	6971(14)	-677(9)	9367(6)	114(8)
C(29)	9476(9)	-2568(6)	6665(4)	88(3)
C(30)	9084(10)	-2145(6)	6384(3)	84(3)
C(31)	8610(7)	-1713(5)	6032(2)	93(3)
C(32)	9058(7)	-938(5)	5946(2)	127(5)
C(33)	8652(8)	-532(3)	5605(3)	130(5)
C(34)	7798(8)	-900(5)	5349(2)	106(4)
C(35)	7351(7)	-1675(6)	5435(2)	136(5)
C(36)	7757(7)	-2082(4)	5777(3)	140(6)
C(37)	7393(16)	-414(9)	4983(4)	85(7)
C(38)	8216(11)	-31(9)	4748(5)	93(8)
C(39)	7822(13)	354(9)	4399(5)	102(8)
C(40)	6605(15)	356(10)	4285(4)	106(8)
C(41)	5783(11)	-27(10)	4520(5)	113(8)
C(42)	6177(14)	-412(9)	4869(5)	117(8)
C(37')	7168(17)	-638(11)	4966(5)	104(9)
C(38')	7782(14)	10(12)	4799(7)	135(11)
C(39')	7337(17)	366(10)	4449(6)	142(11)
C(40')	6277(18)	74(11)	4265(5)	122(9)
C(41')	5663(13)	-574(10)	4432(6)	123(9)
C(42')	6108(16)	-930(8)	4782(6)	117(8)
C(43)	11742(10)	-3246(6)	6717(4)	83(3)
C(44)	12422(11)	-3254(5)	6453(4)	88(3)
C(45)	13185(8)	-3277(6)	6124(2)	100(4)
C(46)	13549(8)	-2543(4)	5957(3)	125(5)
C(47)	14236(8)	-2556(5)	5625(3)	136(5)
C(48)	14559(8)	-3304(7)	5461(2)	131(5)
C(49)	14195(10)	-4038(5)	5629(3)	227(11)
C(50)	13508(10)	-4025(4)	5960(3)	229(12)
C(51)	15252(15)	-3280(9)	5066(4)	88(7)
C(52)	16177(15)	-3833(9)	5004(5)	169(13)

C(53)	16820(14)	-3781(10)	4664(5)	110(8)
C(54)	16538(17)	-3176(11)	4385(5)	131(10)
C(55)	15613(18)	-2622(9)	4446(4)	205(17)
C(56)	14970(15)	-2674(8)	4787(5)	169(13)
C(51')	14970(30)	-2953(13)	5051(6)	75(8)
C(52')	15430(30)	-3641(10)	4865(7)	71(8)
C(53')	15990(30)	-3554(14)	4510(7)	102(11)
C(54')	16100(30)	-2780(17)	4341(6)	115(13)
C(55')	15640(30)	-2092(13)	4527(7)	103(10)
C(56')	15080(20)	-2178(11)	4882(7)	103(10)
C(51")	15430(20)	-3625(15)	5148(6)	75(8)
C(52")	16500(20)	-4068(15)	5170(7)	103(10)
C(53")	17231(19)	-4082(17)	4849(9)	103(10)
C(54")	16890(30)	-3654(19)	4506(8)	115(13)
C(55")	15820(30)	-3211(17)	4485(6)	102(11)
C(56")	15090(20)	-3197(14)	4806(7)	71(8)
C(57)	10176(9)	1003(5)	7632(4)	101(4)
C(58)	9351(11)	1263(7)	7951(4)	134(5)
C(59)	9479(13)	2218(8)	7994(5)	158(7)
C(60)	8838(16)	2593(14)	8299(7)	330(17)
C(61)	10509(9)	-458(5)	7863(3)	87(3)
C(62)	11755(9)	-322(6)	8036(3)	98(3)
C(63)	11989(10)	-891(6)	8385(4)	107(4)
C(64)	13237(10)	-792(6)	8560(4)	130(5)
C(65)	11106(10)	21(5)	7190(3)	94(3)
C(66)	11308(11)	-827(5)	7025(4)	131(5)
C(67)	12339(14)	-801(8)	6744(4)	161(7)
C(68)	13531(13)	-641(11)	6919(6)	217(11)
C(69)	8909(9)	-161(6)	7338(4)	118(5)
C(70)	8341(12)	350(7)	6998(4)	139(5)
C(71)	7057(12)	78(9)	6878(5)	186(8)
C(72)	6466(14)	511(10)	6534(6)	223(10)

Table 3. Bond lengths [Å] and angles [°] for mar616.

Os(1)-N(1)	1.619(6)	C(23)-C(24)	1.3900
Os(1)-C(15)	2.046(12)	C(23)-C(28)	1.3900
Os(1)-C(29)	2.054(13)	C(24)-C(25)	1.3900
Os(1)-C(1)	2.072(12)	C(25)-C(26)	1.3900
Os(1)-C(43)	2.096(12)	C(26)-C(27)	1.3900
N(2)-C(57)	1.531(8)	C(27)-C(28)	1.3900
N(2)-C(65)	1.538(9)	C(23')-C(24')	1.3900
N(2)-C(61)	1.541(9)	C(23')-C(28')	1.3900
N(2)-C(69)	1.544(9)	C(24')-C(25')	1.3900
C(1)-C(2)	1.175(13)	C(25')-C(26')	1.3900
C(2)-C(3)	1.416(13)	C(26')-C(27')	1.3900
C(3)-C(4)	1.3900	C(27')-C(28')	1.3900
C(3)-C(8)	1.3900	C(29)-C(30)	1.237(14)
C(4)-C(5)	1.3900	C(30)-C(31)	1.460(12)
C(5)-C(6)	1.3900	C(31)-C(32)	1.3900
C(6)-C(7)	1.3900	C(31)-C(36)	1.3900
C(6)-C(9)	1.476(9)	C(32)-C(33)	1.3900
C(7)-C(8)	1.3900	C(33)-C(34)	1.3900
C(9)-C(10)	1.3900	C(34)-C(35)	1.3900
C(9)-C(14)	1.3900	C(34)-C(37')	1.508(16)
C(10)-C(11)	1.3900	C(34)-C(37)	1.520(14)
C(11)-C(12)	1.3900	C(35)-C(36)	1.3900
C(12)-C(13)	1.3900	C(37)-C(38)	1.3900
C(13)-C(14)	1.3900	C(37)-C(42)	1.3900
C(15)-C(16)	1.222(14)	C(38)-C(39)	1.3900
C(16)-C(17)	1.383(12)	C(39)-C(40)	1.3900
C(17)-C(18)	1.3900	C(40)-C(41)	1.3900
C(17)-C(22)	1.3900	C(41)-C(42)	1.3900
C(18)-C(19)	1.3900	C(37')-C(38')	1.3900
C(19)-C(20)	1.3900	C(37')-C(42')	1.3900
C(20)-C(21)	1.3900	C(38')-C(39')	1.3900
C(20)-C(23)	1.513(12)	C(39')-C(40')	1.3900
C(20)-C(23')	1.525(14)	C(40')-C(41')	1.3900
C(21)-C(22)	1.3900	C(41')-C(42')	1.3900

C(43)-C(44)	1.199(14)	C(66)-C(67)	1.528(12)
C(44)-C(45)	1.435(15)	C(67)-C(68)	1.452(14)
C(45)-C(46)	1.3900	C(69)-C(70)	1.530(11)
C(45)-C(50)	1.3900	C(70)-C(71)	1.533(11)
C(46)-C(47)	1.3900	C(71)-C(72)	1.488(14)
C(47)-C(48)	1.3900		
C(48)-C(49)	1.3900	N(1)-Os(1)-C(15)	104.1(4)
C(48)-C(51")	1.562(15)	N(1)-Os(1)-C(29)	110.5(4)
C(48)-C(51)	1.580(14)	C(15)-Os(1)-C(29)	87.5(4)
C(48)-C(51')	1.592(15)	N(1)-Os(1)-C(1)	105.9(4)
C(49)-C(50)	1.3900	C(15)-Os(1)-C(1)	82.7(4)
C(51)-C(52)	1.3900	C(29)-Os(1)-C(1)	143.6(3)
C(51)-C(56)	1.3900	N(1)-Os(1)-C(43)	105.4(4)
C(52)-C(53)	1.3900	C(15)-Os(1)-C(43)	150.4(4)
C(53)-C(54)	1.3900	C(29)-Os(1)-C(43)	83.0(4)
C(54)-C(55)	1.3900	C(1)-Os(1)-C(43)	88.5(4)
C(55)-C(56)	1.3900	C(57)-N(2)-C(65)	106.1(7)
C(51')-C(52')	1.3900	C(57)-N(2)-C(61)	110.2(8)
C(51')-C(56')	1.3900	C(65)-N(2)-C(61)	110.4(7)
C(52')-C(53')	1.3900	C(57)-N(2)-C(69)	111.0(7)
C(53')-C(54')	1.3900	C(65)-N(2)-C(69)	110.8(8)
C(54')-C(55')	1.3900	C(61)-N(2)-C(69)	108.4(7)
C(55')-C(56')	1.3900	C(2)-C(1)-Os(1)	174.6(10)
C(51")-C(52")	1.3900	C(1)-C(2)-C(3)	175.0(11)
C(51")-C(56")	1.3900	C(4)-C(3)-C(8)	120.0
C(52")-C(53")	1.3900	C(4)-C(3)-C(2)	121.0(7)
C(53")-C(54")	1.3900	C(8)-C(3)-C(2)	119.0(7)
C(54")-C(55")	1.3900	C(3)-C(4)-C(5)	120.0
C(55")-C(56")	1.3900	C(6)-C(5)-C(4)	120.0
C(57)-C(58)	1.513(12)	C(7)-C(6)-C(5)	120.0
C(58)-C(59)	1.564(13)	C(7)-C(6)-C(9)	122.5(8)
C(59)-C(60)	1.423(14)	C(5)-C(6)-C(9)	117.5(8)
C(61)-C(62)	1.497(10)	C(6)-C(7)-C(8)	120.0
C(62)-C(63)	1.515(11)	C(7)-C(8)-C(3)	120.0
C(63)-C(64)	1.492(11)	C(10)-C(9)-C(14)	120.0
C(65)-C(66)	1.508(10)	C(10)-C(9)-C(6)	120.0(12)

C(14)-C(9)-C(6)	120.0(12)	C(27')-C(28')-C(23')	120.0
C(9)-C(10)-C(11)	120.0	C(30)-C(29)-Os(1)	172.8(10)
C(12)-C(11)-C(10)	120.0	C(29)-C(30)-C(31)	174.9(11)
C(13)-C(12)-C(11)	120.0	C(32)-C(31)-C(36)	120.0
C(12)-C(13)-C(14)	120.0	C(32)-C(31)-C(30)	119.2(7)
C(13)-C(14)-C(9)	120.0	C(36)-C(31)-C(30)	120.8(7)
C(16)-C(15)-Os(1)	174.6(10)	C(33)-C(32)-C(31)	120.0
C(15)-C(16)-C(17)	177.8(12)	C(32)-C(33)-C(34)	120.0
C(16)-C(17)-C(18)	119.2(7)	C(35)-C(34)-C(33)	120.0
C(16)-C(17)-C(22)	120.4(7)	C(35)-C(34)-C(37')	106.3(10)
C(18)-C(17)-C(22)	120.0	C(33)-C(34)-C(37')	133.7(10)
C(19)-C(18)-C(17)	120.0	C(35)-C(34)-C(37)	123.2(9)
C(18)-C(19)-C(20)	120.0	C(33)-C(34)-C(37)	116.8(9)
C(19)-C(20)-C(21)	120.0	C(37')-C(34)-C(37)	16.9(10)
C(19)-C(20)-C(23)	132.5(8)	C(36)-C(35)-C(34)	120.0
C(21)-C(20)-C(23)	107.0(8)	C(35)-C(36)-C(31)	120.0
C(19)-C(20)-C(23')	114.5(9)	C(38)-C(37)-C(42)	120.0
C(21)-C(20)-C(23')	125.3(9)	C(38)-C(37)-C(34)	121.5(12)
C(23)-C(20)-C(23')	22.0(8)	C(42)-C(37)-C(34)	118.3(12)
C(22)-C(21)-C(20)	120.0	C(39)-C(38)-C(37)	120.0
C(21)-C(22)-C(17)	120.0	C(38)-C(39)-C(40)	120.0
C(24)-C(23)-C(28)	120.0	C(41)-C(40)-C(39)	120.0
C(24)-C(23)-C(20)	123.2(11)	C(42)-C(41)-C(40)	120.0
C(28)-C(23)-C(20)	116.3(11)	C(41)-C(42)-C(37)	120.0
C(23)-C(24)-C(25)	120.0	C(38')-C(37')-C(42')	120.0
C(26)-C(25)-C(24)	120.0	C(38')-C(37')-C(34)	110.2(14)
C(25)-C(26)-C(27)	120.0	C(42')-C(37')-C(34)	129.7(14)
C(28)-C(27)-C(26)	120.0	C(37')-C(38')-C(39')	120.0
C(27)-C(28)-C(23)	120.0	C(40')-C(39')-C(38')	120.0
C(24')-C(23')-C(28')	120.0	C(39')-C(40')-C(41')	120.0
C(24')-C(23')-C(20)	119.5(12)	C(40')-C(41')-C(42')	120.0
C(28')-C(23')-C(20)	120.5(12)	C(41')-C(42')-C(37')	120.0
C(25')-C(24')-C(23')	120.0	C(44)-C(43)-Os(1)	170.9(11)
C(26')-C(25')-C(24')	120.0	C(43)-C(44)-C(45)	177.0(13)
C(25')-C(26')-C(27')	120.0	C(46)-C(45)-C(50)	120.0
C(28')-C(27')-C(26')	120.0	C(46)-C(45)-C(44)	119.3(7)

C(50)-C(45)-C(44)	120.6(7)	C(51')-C(52')-C(53')	120.0
C(45)-C(46)-C(47)	120.0	C(54')-C(53')-C(52')	120.0
C(46)-C(47)-C(48)	120.0	C(55')-C(54')-C(53')	120.0
C(49)-C(48)-C(47)	120.0	C(54')-C(55')-C(56')	120.0
C(49)-C(48)-C(51")	101.2(11)	C(55')-C(56')-C(51')	120.0
C(47)-C(48)-C(51")	138.2(12)	C(52")-C(51")-C(56")	120.0
C(49)-C(48)-C(51)	122.2(8)	C(52")-C(51")-C(48)	134.1(14)
C(47)-C(48)-C(51)	117.7(8)	C(56")-C(51")-C(48)	104.3(14)
C(51")-C(48)-C(51)	24.0(9)	C(53")-C(52")-C(51")	120.0
C(49)-C(48)-C(51')	140.1(11)	C(52")-C(53")-C(54")	120.0
C(47)-C(48)-C(51')	97.4(10)	C(55")-C(54")-C(53")	120.0
C(51")-C(48)-C(51')	46.3(14)	C(56")-C(55")-C(54")	120.0
C(51)-C(48)-C(51')	22.3(9)	C(55")-C(56")-C(51")	120.0
C(50)-C(49)-C(48)	120.0	C(58)-C(57)-N(2)	118.2(8)
C(49)-C(50)-C(45)	120.0	C(57)-C(58)-C(59)	106.7(9)
C(52)-C(51)-C(56)	120.0	C(60)-C(59)-C(58)	116.4(14)
C(52)-C(51)-C(48)	120.5(6)	C(62)-C(61)-N(2)	114.3(7)
C(56)-C(51)-C(48)	119.5(6)	C(61)-C(62)-C(63)	109.7(8)
C(51)-C(52)-C(53)	120.0	C(64)-C(63)-C(62)	111.4(9)
C(54)-C(53)-C(52)	120.0	C(66)-C(65)-N(2)	116.8(7)
C(55)-C(54)-C(53)	120.0	C(65)-C(66)-C(67)	109.7(8)
C(54)-C(55)-C(56)	120.0	C(68)-C(67)-C(66)	116.7(13)
C(55)-C(56)-C(51)	120.0	C(70)-C(69)-N(2)	117.7(8)
C(52')-C(51')-C(56')	120.0	C(69)-C(70)-C(71)	113.0(9)
C(52')-C(51')-C(48)	103.7(14)	C(72)-C(71)-C(70)	116.4(11)
C(56')-C(51')-C(48)	135.9(14)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for mar616. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Os(1)	92(1)	62(1)	67(1)	-1(1)	4(1)	-5(1)
N(1)	158(8)	61(4)	97(7)	-8(4)	-1(7)	-31(5)
N(2)	107(7)	64(4)	106(8)	1(4)	2(7)	-22(4)
C(1)	98(9)	59(5)	83(8)	-1(5)	-1(7)	15(5)
C(2)	90(10)	79(6)	63(7)	0(5)	2(7)	21(5)
C(3)	68(9)	63(5)	92(9)	-16(5)	-7(7)	10(5)
C(4)	119(11)	80(6)	68(8)	0(5)	15(8)	-7(6)
C(5)	86(9)	68(6)	123(11)	1(6)	7(9)	-17(5)
C(6)	110(11)	65(6)	92(9)	9(5)	12(9)	9(5)
C(7)	89(9)	97(7)	84(9)	5(6)	10(8)	-19(6)
C(8)	85(9)	119(7)	62(7)	1(6)	6(7)	-7(6)
C(9)	132(14)	75(6)	93(11)	6(6)	-6(10)	-7(7)
C(10)	133(16)	144(11)	180(20)	30(11)	-69(15)	-16(11)
C(11)	210(20)	220(20)	200(30)	-10(20)	0(20)	-23(14)
C(12)	172(19)	177(15)	270(30)	66(16)	-160(20)	-77(15)
C(13)	250(20)	174(13)	110(14)	45(11)	-30(17)	-31(16)
C(14)	149(13)	122(9)	130(15)	16(10)	-64(14)	-18(9)
C(15)	61(8)	77(6)	94(9)	15(6)	-12(7)	-11(5)
C(16)	57(8)	76(6)	97(9)	-2(6)	23(7)	-2(5)
C(17)	54(8)	67(5)	97(9)	4(5)	30(7)	5(5)
C(18)	83(8)	89(6)	84(8)	7(6)	11(8)	14(5)
C(19)	103(10)	94(6)	97(10)	17(6)	1(8)	23(6)
C(20)	86(9)	73(6)	109(11)	16(6)	50(8)	2(5)
C(21)	72(8)	89(6)	112(11)	29(7)	15(9)	11(5)
C(22)	76(8)	86(6)	83(8)	2(6)	18(7)	-5(6)
C(29)	68(8)	102(7)	93(9)	-25(6)	6(8)	-7(6)
C(30)	83(9)	94(6)	73(8)	2(6)	-15(7)	-5(6)
C(31)	85(9)	121(8)	72(7)	15(7)	-11(7)	10(7)
C(32)	202(14)	81(7)	92(10)	-2(6)	-30(10)	-20(8)
C(33)	188(14)	109(8)	89(10)	3(7)	-27(10)	13(8)
C(34)	107(10)	121(9)	88(9)	6(7)	-4(9)	15(7)

C(35)	121(11)	190(13)	92(10)	39(10)	-48(10)	-47(9)
C(36)	118(12)	181(11)	116(12)	24(10)	-43(11)	-74(9)
C(43)	82(9)	82(6)	87(8)	4(6)	20(7)	-9(6)
C(44)	97(11)	77(6)	92(9)	0(7)	15(8)	-13(6)
C(45)	111(11)	104(7)	90(9)	4(7)	36(8)	-9(7)
C(46)	111(11)	149(11)	119(13)	4(9)	46(10)	-2(8)
C(47)	138(13)	168(12)	106(12)	-17(10)	40(11)	-4(10)
C(48)	107(11)	189(14)	100(10)	-42(10)	31(9)	-77(10)
C(49)	260(20)	184(15)	250(30)	-135(16)	190(20)	-84(14)
C(50)	300(20)	118(10)	280(30)	-94(14)	180(20)	-74(12)
C(57)	95(9)	87(6)	118(11)	-6(7)	-6(9)	-13(6)
C(58)	126(12)	132(10)	143(16)	-22(9)	9(11)	5(8)
C(59)	136(15)	188(15)	153(18)	-48(12)	26(13)	4(11)
C(60)	124(17)	550(40)	320(50)	30(40)	40(20)	70(20)
C(61)	91(9)	90(6)	82(8)	11(6)	13(7)	-21(6)
C(62)	96(10)	100(7)	98(9)	-3(7)	13(8)	-6(6)
C(63)	81(9)	118(8)	121(12)	7(8)	6(9)	6(7)
C(64)	128(12)	123(9)	141(14)	7(9)	31(11)	34(8)
C(65)	121(9)	76(6)	89(8)	0(5)	34(8)	-13(6)
C(66)	191(14)	70(6)	130(12)	-23(7)	-5(11)	-37(7)
C(67)	226(19)	124(9)	143(15)	-20(9)	132(16)	-22(12)
C(68)	152(16)	228(18)	280(30)	-49(18)	71(18)	29(14)
C(69)	99(10)	120(8)	135(12)	-26(8)	14(9)	-64(7)
C(70)	186(16)	124(9)	103(11)	31(8)	-24(11)	-18(9)
C(71)	119(13)	233(17)	200(20)	38(15)	-53(14)	-102(12)
C(72)	200(18)	232(18)	230(30)	60(18)	-97(18)	-15(13)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for mar616.

	x	y	z	U(eq)
H(4)	14443	-2537	7983	106
H(5)	15622	-2523	8566	111
H(7)	12964	-3629	9127	108
H(8)	11785	-3642	8544	106
H(10)	16752	-3283	9008	187
H(11)	17988	-3212	9577	253
H(12)	17173	-2923	10179	254
H(13)	15120	-2705	10211	214
H(14)	13884	-2777	9641	163
H(18)	9347	-2249	8493	102
H(19)	8531	-1461	8984	118
H(21)	6035	-587	8196	109
H(22)	6851	-1375	7705	97
H(24)	6165	571	8568	121
H(25)	5107	1307	9024	96
H(26)	4682	712	9624	158
H(27)	5317	-620	9767	153
H(28)	6375	-1357	9312	101
H(24')	6280	563	8640	116
H(25')	5746	1409	9154	187
H(26')	5978	951	9801	294
H(27')	6744	-354	9934	312
H(28')	7278	-1200	9420	137
H(32)	9629	-692	6117	152
H(33)	8951	-13	5547	156
H(35)	6780	-1922	5264	163
H(36)	7458	-2600	5835	168
H(38)	9029	-32	4824	112
H(39)	8372	610	4241	122
H(40)	6342	614	4052	128

H(41)	4969	-25	4444	136
H(42)	5626	-668	5027	141
H(38')	8490	205	4922	162
H(39')	7747	800	4338	171
H(40')	5980	313	4031	147
H(41')	4955	-769	4309	148
H(42')	5698	-1364	4893	141
H(46)	13333	-2043	6067	150
H(47)	14479	-2065	5513	164
H(49)	14411	-4538	5519	273
H(50)	13265	-4516	6073	274
H(52)	16366	-4238	5191	203
H(53)	17439	-4151	4623	132
H(54)	16968	-3141	4157	157
H(55)	15424	-2217	4259	246
H(56)	14351	-2304	4828	203
H(52')	15355	-4159	4978	86
H(53')	16291	-4015	4385	122
H(54')	16472	-2721	4103	138
H(55')	15716	-1573	4414	124
H(56')	14780	-1718	5007	124
H(52")	16728	-4354	5399	124
H(53")	17947	-4378	4863	124
H(54")	17380	-3663	4291	138
H(55")	15594	-2925	4256	122
H(56")	14375	-2901	4791	86
H(57A)	10992	1143	7721	121
H(57B)	9978	1333	7400	121
H(58A)	9582	994	8199	160
H(58B)	8526	1116	7878	160
H(59A)	10325	2347	8038	190
H(59B)	9213	2468	7745	190
H(60A)	9054	3164	8320	495
H(60B)	9038	2322	8545	495
H(60C)	7989	2545	8239	495
H(61A)	9941	-363	8066	105

H(61B)	10432	-1028	7781	105
H(62A)	11845	246	8121	117
H(62B)	12336	-428	7838	117
H(63A)	11418	-773	8585	128
H(63B)	11869	-1457	8301	128
H(64A)	13358	-1160	8780	195
H(64B)	13353	-235	8648	195
H(64C)	13803	-918	8364	195
H(65A)	10866	382	6972	113
H(65B)	11868	223	7302	113
H(66A)	10580	-1018	6885	157
H(66B)	11502	-1208	7240	157
H(67A)	12359	-1325	6607	193
H(67B)	12157	-380	6547	193
H(68A)	14101	-627	6715	325
H(68B)	13748	-1070	7104	325
H(68C)	13534	-121	7053	325
H(69A)	8960	-727	7249	141
H(69B)	8365	-152	7553	141
H(70A)	8836	304	6772	167
H(70B)	8330	924	7077	167
H(71A)	7071	-506	6819	223
H(71B)	6563	152	7103	223
H(72A)	5699	261	6469	334
H(72B)	6964	471	6311	334
H(72C)	6352	1080	6599	334

Perspective view of $[^nBu_4N][OsN(C{\equiv}CC_6H_4Ph)_4]$, $[^nBu_4N]$**4** (30 % thermal ellipsoids)