tively. $(\Delta/\sigma)_{max} = 0.86$, ratio of observations to parameters 9.6:1, $R = 0.055$, $wR = 0.067$, $S = 2.76$. $R = 0.062$ for all data. Final difference Fourier excursions 0.61 and -0.60 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atom numbering for Table 1, atom coordinates, and Table 2, bond distances and angles, follows that shown in Fig. 1.*

Related literature. Compounds with similar Cu bonding environment include (oxalato)-bis(aqua-*N*,*N*,*N*′,*N*′-tetramethylethylenediaminecopper(II)bis-(hexafluorophosphate) dihydrate (Sletten, 1983), and (oxalato)-bis(aqua-*N*,*N*,*N*′,*N*′-tetramethylenediaminecopper(II) diperchlorate hydrate (Julve, Verdaguer, Gleizes, Philoche-Levisalles & Kahn, 1984).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54141 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(triphenylphosphine oxide)sodium Nitrido[*N*,*N*′-*o*-phenylenebis(salicylamidato)-*N*,*N*′,*O*,*O*′]osmate(VI)

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Abstract. $[Na(C_{18}H_{15}OP)_2][OsN(C_{20}H_{12}N_2O_4)]$, $M_r = 1128.12$, triclinic, $P\bar{1}$, $a = 14.027$ (2), $b = 14.738$ (3), $c = 14.939$ (3) Å, $\alpha = 99.95$ (2), $\beta = 106.90$ (2), $\gamma = 117.11$ (1)°, $V = 2455.3$ (9) Å^3, $Z = 2$, $D_x = 1.526$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 26.75$ cm^{-1}, $F(000) = 1128$, $T = 296$ K, $R_F = 3.45\%$ for 7384 observed reflections and 551 parameters. The geometry about the anionic Os^{VI} coordination complex is square pyramidal, as found for other five-coordinate highly oxidized metal nitrides. The Os atom is displaced 0.57 Å above the basal plane of the pyramid. The Os—N(1) bond distance of 1.639 (6) Å is indicative of an Os—N triple bond. The Na^+ counterion is associated with the two Os-bound O atoms and two molecules of Ph_3PO.

Experimental. Yellow cube-shaped crystals (0.25 × 0.25 × 0.25 mm) were the gift of Terrence Collins at Carnegie Mellon University. Nicolet *R*3*m* diffractometer with graphite monochromator; ω scans; lattice parameters from least-squares fit of 25 reflections ($20 \le 2\theta \le 25°$); empirical absorption correction was applied ($T_{max}/T_{min} = 1.17$); $2\theta_{max} = 50°$ ($h = \pm 17$, $k = \pm 18$, $l = 18$); standard reflections $\bar{4}\bar{2}9$, $\bar{5}90$, $\bar{8}26$. 9382 reflections collected, 8876 unique ($R_{int} = 2.28\%$), 7384 observed with $F_o > 4.0\sigma(F_o)$, 1492 unobserved reflections. Patterson structure solution; least-squares refinement on 551 parameters; all non-H atoms anisotropic, H atoms included as idealized contributors ($d_{CH} = 0.960$ Å; $U = 1.2$ times the U value for the attached C atom), $R_F = 3.45\%$, $wR_F = 3.79\%$, $S = 1.081$, $w^{-1} = \sigma(F_o) + gF_o^2$, $g = 0.001$; $(\Delta/\sigma)_{max} = 0.139$; $\Delta\rho_{max} = 0.79$, $\Delta\rho_{min} = -0.93$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 99, 144); *SHELXTL* computer program (Sheldrick, 1985).

Atomic and equivalent isotropic thermal parameters are given in Table 1, and selected bond lengths and angles are listed in Table 2. Fig. 1 shows the molecular structure of the compound. An isolated view of the anion displaying the coordination sphere of Os is shown in Fig. 2.†

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† Lists of structure factors, anisotropic thermal parameters, H-atom parameters, and full lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54448 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Os	92 (1)	2248 (1)	1047 (1)	41 (1)
Na	−1137 (2)	1236 (2)	−1704 (1)	46 (1)
P(1)	103 (1)	2623 (1)	−3333 (1)	52 (1)
P(2)	−4321 (1)	197 (1)	−2525 (1)	53 (1)
O(1)	−939 (3)	838 (3)	−100 (3)	47 (2)
O(2)	865 (4)	166 (3)	2214 (3)	62 (2)
O(3)	3471 (4)	5025 (4)	2152 (4)	108 (3)
O(4)	40 (3)	2722 (3)	−113 (3)	54 (2)
O(5)	−3055 (3)	625 (4)	−2296 (3)	68 (2)
O(6)	−442 (4)	1968 (3)	−2772 (3)	69 (3)
N(1)	−738 (4)	2497 (4)	1493 (3)	54 (2)
N(2)	692 (3)	1567 (3)	1931 (3)	44 (2)
N(3)	1755 (4)	3513 (4)	1897 (4)	56 (2)
C(1)	−1392 (4)	−147 (4)	12 (4)	43 (2)
C(2)	−2428 (4)	−1021 (4)	−794 (4)	52 (3)
C(3)	−2953 (5)	−2071 (4)	−768 (5)	64 (3)
C(4)	−2441 (5)	−2264 (4)	55 (5)	63 (3)
C(5)	−1409 (5)	−1405 (4)	858 (5)	56 (3)
C(6)	−872 (4)	−344 (4)	847 (4)	46 (3)
C(7)	288 (4)	482 (4)	1707 (4)	45 (3)
C(8)	1709 (4)	2365 (4)	2844 (4)	51 (3)
C(9)	2050 (5)	2172 (6)	3730 (4)	69 (4)
C(10)	2958 (6)	3039 (7)	4595 (5)	85 (5)
C(11)	3530 (6)	4091 (7)	4579 (5)	87 (4)
C(12)	3213 (5)	4292 (5)	3713 (5)	73 (3)
C(13)	2276 (4)	3421 (5)	2828 (4)	57 (3)
C(14)	2413 (5)	4390 (4)	1644 (5)	68 (3)
C(15)	1785 (6)	4561 (4)	760 (5)	70 (4)
C(16)	2387 (8)	5616 (6)	715 (7)	98 (5)
C(17)	1893 (10)	5897 (7)	−33 (8)	112 (7)
C(18)	768 (8)	5136 (6)	−800 (6)	100 (6)
C(19)	182 (7)	4085 (5)	−788 (5)	74 (5)
C(20)	679 (6)	3795 (4)	−20 (4)	59 (3)
C(21)	−3660 (3)	1025 (3)	−493 (3)	59 (3)
C(22)	−3771	1441	358	75 (4)
C(23)	−4733	1545	257	78 (4)
C(24)	−5584	1234	−695	97 (6)
C(25)	−5473	819	−1545	82 (5)
C(26)	−4511	715	−1444	56 (3)
C(31)	−4619 (4)	−1823 (4)	−3133 (3)	75 (4)
C(32)	−5228	−2957	−3419	100 (6)
C(33)	−6382	−3541	−3508	152 (9)
C(34)	−6928	−2990	−3312	173 (9)
C(35)	−6320	−1856	−3026	121 (7)
C(36)	−5165	−1273	−2936	64 (3)
C(41)	−5939 (3)	−200 (3)	−4409 (3)	71 (4)
C(42)	−6434	142	−5123	88 (5)
C(43)	−5999	1250	−4917	97 (7)
C(44)	−5069	2018	−3997	103 (7)
C(45)	−4574	1677	−3283	83 (4)
C(46)	−5009	568	−3489	55 (3)
C(51)	−1441 (3)	3312 (3)	−3532 (4)	86 (5)
C(52)	−1814	4033	−3641	113 (6)
C(53)	−1063	5041	−3672	101 (6)
C(54)	62	5329	−3593	123 (7)
C(55)	435	4609	−3483	104 (6)
C(56)	−316	3600	−3452	63 (3)
C(61)	2382 (4)	3284 (4)	−3231 (3)	103 (5)
C(62)	3610	3873	−2724	128 (7)
C(63)	4165	4562	−1724	97 (5)
C(64)	3492	4664	−1233	112 (6)
C(65)	2264	4075	−1741	108 (6)
C(66)	1709	3386	−2740	55 (3)
C(71)	−322 (4)	2217 (4)	−5363 (4)	89 (5)
C(72)	−762	1531	−6345	110 (6)
C(73)	−1265	410	−6572	125 (6)
C(74)	−1327	−24	−5816	124 (6)
C(75)	−887	662	−4835	86 (5)
C(76)	−384	1782	−4608	58 (3)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Os—N(1)	1.639 (6)	Os—O(1)	1.984 (3)
Os—N(2)	2.010 (5)	Os—O(4)	1.972 (4)
Os—N(3)	2.007 (3)	Na—O(1)	2.532 (5)
Na—O(5)	2.227 (5)	Na—O(4)	2.407 (3)
Na—O(6)	2.281 (6)		

N(2)—Os—N(3)	79.9 (2)	O(1)—Os—O(4)	77.7 (1)
O(1)—Os—N(2)	91.5 (2)	O(4)—Os—N(3)	92.0 (2)
N(1)—Os—N(2)	104.6 (2)	N(1)—Os—N(3)	106.0 (2)
N(1)—Os—O(1)	108.5 (2)	N(1)—Os—O(4)	107.5 (2)
Na—O(6)—P(1)	170.4 (3)	Na—O(5)—P(2)	170.3 (3)
O(1)—Na—O(4)	60.2 (1)	O(1)—Na—O(5)	97.3 (2)
O(1)—Na—O(6)	154.7 (2)	O(4)—Na—O(5)	113.8 (2)
O(4)—Na—O(6)	104.3 (1)	O(5)—Na—O(6)	107.7 (2)

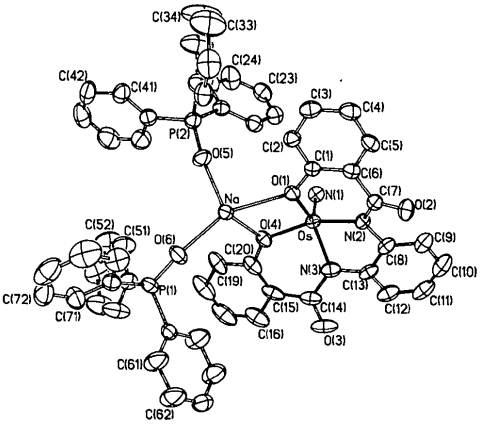

Fig. 1. Molecular structure and labeling scheme for the title compound.

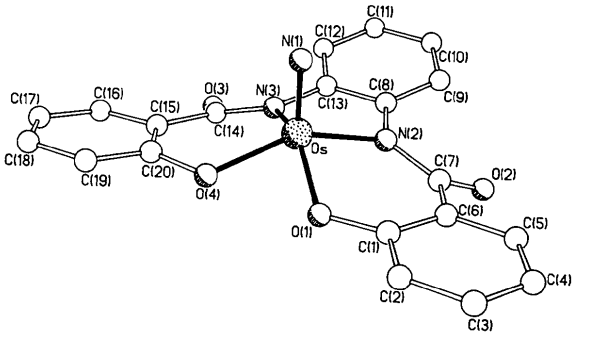

Fig. 2. View of anion showing the coordination about the Os atom.

Related literature. The title compound was obtained by the treatment of $[(Ph_3PO)_2Os(\eta^4\text{-HBA-B})]$ [where HBA-B = *N,N′-o*-phenylenebis(salicylamidato)-*N,N′,O,O′*] with Me_3SiN_3 followed by NaOH. An analogous compound, $[Ph_3PNH_2][OsN(\eta^4\text{-HBA-B})]$, and its crystal structure have been reported (Barner, Collins, Mapes & Santarsiero, 1986).

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