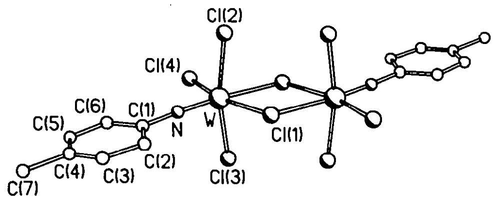

Fig. 1. Structure of the dimeric molecule, without H atoms, showing the atom-labelling scheme.

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond lengths and angles in Table 2. The molecular structure is shown in Fig. 1.

Related literature. A similar structure has been observed for the alkylimido complexes $[\{WCl_3(NR)\}_2(\mu\text{-}Cl)_2]$ with $R = C_2H_5$ (Bradley, Errington, Hursthouse & Short, 1987), $R = C_2Cl_5$ (Drew, Fowles, Rice & Rolfe, 1971), and $R = CH(CH_3)_2$ (Ashcroft, Clark, Nielson & Rickard, 1986).

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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 44157 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A *trans*-Chelating Oxybisphosphine Complex of $PtBr_2$

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Abstract. Dibromo{[3,3′-oxybis(phenylenemethylene)]bis(diphenylphosphine)}platinum(II)–acetonitrile (2/1), $[PtBr_2(C_{38}H_{32}OP_2)].\frac{1}{2}C_2H_3N$, $M_r = 942{\cdot}1$, monoclinic, $P2_1/n$, $a = 9{\cdot}923\ (8)$, $b = 16{\cdot}389\ (2)$, $c = 21{\cdot}527\ (2)$ Å, $\beta = 97{\cdot}26\ (4)°$, $V = 3472{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}802$ g cm^{-3}, $F(000) = 1828$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 62{\cdot}4$ cm^{-1}, room temperature, final $R = 0{\cdot}035$ for 5561 unique reflections with $F_o > 5\sigma(F_o)$. The coordination about the Pt atom is square planar. The Pt atom is displaced by 0·04 (1) Å towards the O atom from the best plane through atoms P_2Br_2. The phenyl rings in the *trans*-spanning 3,3′-oxydibenzyl ligand are inclined at an angle of 60 (1)°, and angle C(Ph)—O—C(Ph) is 121·6 (5)°. Angles C(Ph)—P—Pt in the equatorial plane are opened to an average value of 120·1 (1)° owing to steric hindrance involving the Br atoms.

Experimental. Yellow bipyramidal crystals grown from a CH_3CN/toluene solution. Stoe–Siemens AED2 four-circle diffractometer, monochromated Mo $K\alpha$ radiation, ω/θ scan mode, $2\theta_{\max}$ 55°, 8506 reflections $\pm h+k+l$. Four check reflections, 3% intensity variation. Crystal cut to 0·27 × 0·27 × 0·42 mm, empirical absorption correction [programs *REDU*4 and *EMPIR* (Stoe & Co., 1985)], 8 ψ scans in 2θ range 11–39°, max., min. transmission 0·253, 0·170 respectively. 7700 unique reflections ($R_{\text{int}} = 0{\cdot}033$), 5566 with $F_o > 5\sigma(F_o)$, 5561 used for all calculations, five (probably suffering from extinction) removed. Index range h −12→12, k 0→21 l 0→27. Cell constants from $\pm\omega$ values of 12 reflections and their equivalents in the 2θ range 15–28°. Structure solved by Patterson and Fourier methods. H atoms from difference syntheses, refined isotropically. A disordered CH_3CN molecule with the central C atom located on a center of symmetry was assumed to occupy two alternative orientations inclined at an angle of 5(2)°, hence $C(H_3)$ and N atoms given occupancy of 0·5. Despite large correlation coefficients between the

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Table 1. *Final positional and equivalent isotropic thermal parameters* (× 10^4)

$U_{eq} = \frac{1}{3}\sum_i\sum_j a_i^* a_i^* (\mathbf{a}_i.\mathbf{a}_j)$.

	x	y	z	U_{eq}(Å^2)
Pt	7224·0 (2)	2415·8 (2)	6448·7 (1)	517 (1)
Br (1)	5174 (1)	2839·7 (6)	5789·1 (3)	1025 (2)
Br (2)	9289 (1)	2083·5 (4)	7131·0 (3)	659 (2)
P(1)	8583 (1)	2446 (1)	5646 (1)	537 (3)
P(2)	5884 (1)	2393 (1)	7255 (1)	561 (4)
O(1)	6434 (6)	−651 (3)	6024 (2)	1098 (17)
C(11)	7843 (6)	2588 (3)	4836 (2)	570 (14)
C(12)	8499 (7)	3047 (4)	4434 (3)	762 (17)
C(13)	7971 (9)	3137 (5)	3817 (3)	1008 (24)
C(14)	6745 (9)	2773 (6)	3602 (3)	1077 (26)
C(15)	6096 (8)	2304 (6)	4003 (4)	1077 (25)
C(16)	6635 (6)	2198 (4)	4610 (3)	808 (18)
C(21)	9609 (6)	1517 (4)	5585 (3)	690 (16)
C(22)	8694 (6)	808 (3)	5381 (3)	670 (16)
C(23)	8013 (7)	406 (3)	5813 (3)	711 (17)
C(24)	7110 (7)	−205 (4)	5620 (3)	787 (19)
C(25)	6913 (7)	−453 (4)	5006 (3)	810 (19)
C(26)	7622 (8)	−61 (4)	4570 (3)	861 (20)
C(27)	8493 (7)	561 (4)	4760 (3)	823 (19)
C(31)	9756 (5)	3292 (3)	5778 (2)	530 (13)
C(32)	9219 (6)	4069 (4)	5739 (3)	675 (17)
C(33)	10068 (7)	4749 (4)	5828 (3)	759 (18)
C(34)	11431 (7)	4642 (4)	5976 (3)	872 (21)
C(35)	11969 (6)	3883 (5)	6030 (4)	926 (22)
C(36)	11126 (6)	3202 (4)	5934 (3)	741 (17)
C(41)	5099 (6)	3372 (3)	7376 (3)	637 (15)
C(42)	3953 (7)	3406 (4)	7690 (3)	866 (20)
C(43)	3412 (7)	4166 (5)	7833 (4)	976 (23)
C(44)	4002 (8)	4866 (4)	7658 (4)	1036 (25)
C(45)	5102 (8)	4825 (4)	7336 (4)	1029 (25)
C(46)	5670 (7)	4079 (4)	7203 (3)	814 (19)
C(51)	4454 (6)	1671 (4)	7071 (3)	689 (16)
C(52)	4881 (6)	795 (4)	7039 (3)	620 (15)
C(53)	5483 (7)	503 (4)	6530 (3)	690 (16)
C(54)	5920 (6)	−292 (3)	6523 (3)	683 (16)
C(55)	5796 (7)	−811 (4)	7023 (3)	776 (18)
C(56)	5195 (7)	−526 (4)	7519 (3)	818 (19)
C(57)	4718 (7)	263 (4)	7522 (3)	766 (18)
C(61)	6645 (6)	2110 (3)	8041 (2)	591 (14)
C(62)	7235 (6)	1337 (4)	8140 (3)	708 (17)
C(63)	7773 (7)	1095 (5)	8730 (3)	872 (21)
C(64)	7774 (8)	1628 (5)	9228 (3)	1014 (24)
C(65)	7223 (9)	2390 (5)	9133 (3)	953 (23)
C(66)	6669 (7)	2631 (4)	8550 (3)	756 (18)
C(*X*)*	0	0	0	1572 (69)
C(*Y*)*	1011 (36)	342 (25)	−481 (21)	3630 (186)
N(*Y*)*	−776 (23)	−261 (16)	303 (12)	1901 (94)

* Occupancy 0·5.

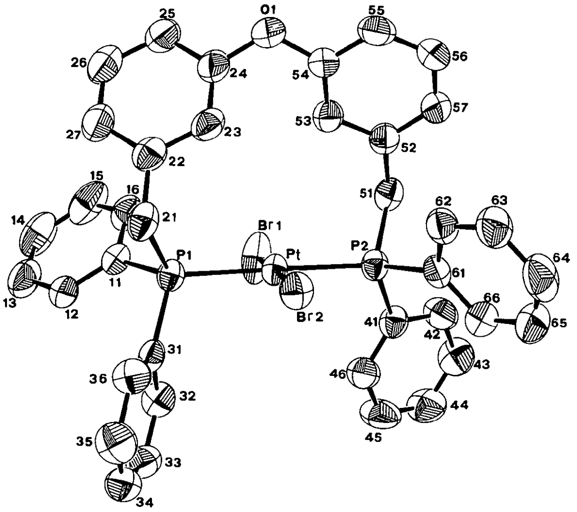

Fig. 1. View of the molecule showing the numbering scheme.

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

Pt–Br(1)	2·430 (1)	P(1)–C(11)	1·820 (5)
Pt–Br(2)	2·427 (1)	P(1)–C(21)	1·845 (6)
Pt–P(1)	2·322 (1)	P(1)–C(31)	1·811 (5)
Pt–P(2)	2·315 (2)	P(2)–C(41)	1·815 (6)
O(1)–C(24)	1·375 (9)	P(2)–C(51)	1·852 (6)
O(1)–C(54)	1·378 (8)	P(2)–C(61)	1·825 (5)
Br(1)–Pt–Br(2)	176·27 (2)	C(11)–P(1)–C(21)	101·3 (2)
P(1)–Pt–P(2)	179·46 (5)	C(11)–P(1)–C(31)	103·0 (2)
Br(1)–Pt–P(1)	94·64 (3)	C(21)–P(1)–C(31)	107·1 (3)
Br(1)–Pt–P(2)	85·70 (3)	Pt–P(2)–C(41)	113·0 (2)
Br(2)–Pt–P(1)	85·99 (3)	Pt–P(2)–C(51)	110·3 (2)
Br(2)–Pt–P(2)	93·64 (3)	Pt–P(2)–C(61)	119·3 (2)
C(24)–O(1)–C(54)	121·6 (5)	C(41)–P(2)–C(51)	105·3 (3)
Pt–P(1)–C(11)	120·9 (2)	C(41)–P(2)–C(61)	103·1 (3)
Pt–P(1)–C(21)	114·4 (2)	C(51)–P(2)–C(61)	104·6 (3)
Pt–P(1)–C(31)	108·8 (2)		

parameters of the three atoms this model appeared to be the best possible and no further attempt was made to improve it. Weighted anisotropic blocked (421 + 129 parameters) full-matrix least-squares refinement on F, $R = 0{\cdot}035$, $wR = 0{\cdot}037$; weighting scheme $w^{-1} = \sigma^2(F_o) + 0{\cdot}00082(F_o^2)$. Max. Δ/σ 0·39, mean $\Delta/\sigma <$ 0·08 (excluding CH_3CN). Max. features in final difference map +1·02 [0·92 Å from Br(2)], −1·26 e Å^{-3}. Neutral complex-atom scattering factors from *International Tables for X-ray Crystallography* (1974). Program: *SHELX*76 (Sheldrick, 1976). Final positional and equivalent isotropic thermal parameters are given in Table 1,* and selected bond distances and angles in Table 2. The numbering scheme used is apparent from Fig. 1 prepared using *ORTEP* (Johnson, 1976).

Related literature. Transition-metal complexes of ditertiary phosphines spanning the *trans* positions and containing flexible and rigid backbones have been studied extensively (Pryde, Shaw & Weeks, 1976; Camalli, Caruso, Chaloupka, Kapoor, Pregosin & Venanzi, 1984; Shaw, 1980, 1982). For example, the ligand (2,11-benzo[*c*]phenanthrenediyl)bismethylenebis(diphenylphosphine), which has a rigid backbone, forms square-planar *trans* chelate complexes with a variety of metal centers and they have found applications in mechanistic as well as structural studies (Bracher, Pregosin & Venanzi, 1975; Barrow, Burgi, Johnson & Venanzi, 1976; Barrow, Burgi, Camalli, Caruso, Fischer, Venanzi & Zambonelli, 1983). The title compound is a complex of a similar type of *trans*-spanning ligand, but with a more flexible backbone. For the synthesis and properties of the title compound and the structure of the AgCl complex of the same ligand see Marty, Kapoor, Burgi & Fischer (1987) and

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* Lists of observed and calculated structure factors, anisotropic thermal parameters, H-atom positions and bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44172 (37 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1-Amino-4-phenylamino-9,10-anthracenedione

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Abstract. $C_{20}H_{14}N_2O_2$, $M_r = 314{\cdot}3$, monoclinic, $P2_1/c$, $a = 12{\cdot}251\ (2)$, $b = 16{\cdot}746\ (3)$, $c = 7{\cdot}347\ (1)$ Å, $\beta = 91{\cdot}69\ (1)°$, $U = 1507\ (2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}40$, $D_x = 1{\cdot}385$ Mg m^{-3}, Mo $K\bar{\alpha}$ radiation, $\lambda = 0{\cdot}7107$ Å, $\mu = 0{\cdot}052$ mm^{-1}, $F(000) = 654$, $T = 295\ (2)$ K, $R = 0{\cdot}050$ for 1573 reflections with $I \geq 2{\cdot}5\sigma(I)$. The anthraquinone nucleus is nearly planar as exemplified by the dihedral angle of 5·3° formed between the outer six-membered rings; the nitrogen-bound phenyl group is not coplanar with the remaining molecule but forms a dihedral angle of 44·6°. Within the crystal lattice there are no significant intermolecular contacts less than those expected from normal van der Waals radii separations; the closest contact of 2·51 (5) Å occurs between O(1) and H(3′)($1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$). Within the individual molecules however there are two close intramolecular hydrogen-bonding contacts of 1·76 (4) Å for N(1)–H(n1)···O(1) and 1·89 (4) Å for N(2)–H(n2)···O(2).

Experimental. $C_{20}H_{14}N_2O_2$ was obtained as well formed deep-coloured crystals from the recrystallization of a solution (benzene/80–120°-petroleum spirit) of technical-grade (Fluka A.G.) 1,4-diaminoanthraquinone. The compound is registered as C. I. Disperse Blue-19. Density by flotation. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω:2θ scan technique. Cell parameters on crystal $0{\cdot}44 \times 0{\cdot}31 \times 0{\cdot}11$ mm from least-squares procedure (de Boer & Duisenberg, 1984) on 25 reflections ($8 \leq \theta \leq 12°$). No absorption correction. Total of 3779 reflections ($1 \leq \theta \leq 27{\cdot}5°$) measured in the range $-15 \leq h \leq 1$, $-21 \leq k \leq 0$, $-9 \leq l \leq 9$. No significant variation in the intensities of three standards ($2\bar{4}\bar{2}$, $\bar{1}15$, $\bar{1}72$) monitored every 7200 s. 3456 unique reflections ($R_{\rm int} = 0{\cdot}025$), 1573 satisfied $I \geq 2{\cdot}5\sigma(I)$. Structure solved by direct methods with *SHELXS*86 (Sheldrick, 1986), full-matrix least-squares refinement of 273 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms, H atoms located from difference map and refined. At convergence $R = 0{\cdot}050$, $wR = 0{\cdot}054$, $w = 3{\cdot}62/[\sigma^2(F) + 0{\cdot}0015F^2]$, $S = 2{\cdot}3$, $(\Delta/\sigma)_{\rm max} \leq 0{\cdot}001$, $(\Delta\rho)_{\rm max} = 0{\cdot}17$, $(\Delta\rho)_{\rm min} = -0{\cdot}28$ e Å^{-3}; no extinction correction. Scattering factors for all atoms given in *SHELX*76 (Sheldrick, 1976), all calculations on laboratory μ-VAX computer system. Atomic parameters given in Table 1, selected bond

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