Bindungsabstände und -winkel in Tabelle 2.* Die Struktur ist in Fig. 1, die Elementarzelle in Fig. 2 abgebildet.

Die Struktur ist aus $[TiCl_6]^{2-}$-Anionen und $[C_{16}H_{18}NO_2]^+$-Kationen aufgebaut, die durch schwache Wasserstoffbrückenbindungen $N-H\cdots Cl$ miteinander verknüpft sind. Im zentrosymmetrischen Anion ist das Ti-Atom verzerrt oktaedrisch von sechs Cl-Atomen umgeben. Die äquatorialen Ti—Cl-Abstände sind mit 2,351 (1) Å gegenüber den axialen Bindungen Ti—Cl(2) mit 2,309 (1) Å leicht aufgeweitet. In den vergleichbaren Ionen $[POCl_3.TiCl_4]$ (Branden & Lindqvist, 1960), $[Ti_2Cl_9]^{2-}$ und $[Ti_2Cl_{10}]^{2-}$ (Kistenmacher & Stucky, 1971) variieren die Abstände von 2,188 (7) Å bis 2,300 (3) Å. Im Kation, entstanden durch Protonierung der Schiffschen Base, entspricht der Bindungsabstand N—C(2) mit 1,269 Å einer Doppelbindung, der Abstand N—C(1) dagegen einer Einfachbindung [1,490 (4)]. Die weiteren Abstände sind in guter Übereinstimmung mit den Literaturwerten (Chiari, Piovesana, Tarantelli & Zanazzi, 1982; Escobar & Garland, 1983; Alyea, Dee & Ferguson, 1985). Aus der Distanz Cl(3)$\cdots$H mit einem Wert von 2,34 (3) Å und einem Winkel Cl(3)$\cdots$H—N von 164 (2)° resultiert das Vorliegen einer Wasserstoffbrückenbindung zwischen den Anionen und den H-Atomen der Iminium-Gruppe der Schiffschen Base. In Carminomycinhydrochlorid (Von Dreele & Einck, 1977) werden ebenfalls Wasserstoffbrückenbindungen beobachtet, die Abstände Cl$\cdots$

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* Listen der anisotropen Temperaturparameter, der Lageparameter der H-Atome, aller Bindungsabstände und -winkel, sowie der beobachteten und berechneten Strukturfaktoren sind bei der British Library Document Supply Centre (Supplementary Publication No. SUP 43412: 21 pp.) hinterlegt. Kopien sind erhältlich durch: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

N betragen 3,210 (5) Å und 3,238 (5) Å, in der Titelverbindung sind sie dagegen mit 3,436 (3) Å bzw. 3,331 (3) Å leicht verlängert.

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Structure of the Oxygen Adduct Bis[(ethylthioethyl)diphenylphosphine-*S,P*]-peroxoiridium(III) Tetraphenylborate(III)

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Abstract. $[Ir(O_2)\{P(C_6H_5)_2(C_4H_9S)\}_2]B(C_6H_5)_4$, $M_r = 1092\cdot2$, monoclinic, $P2_1/n$, $a = 11\cdot423$ (4), $b = 14\cdot560$ (7), $c = 30\cdot443$ (11) Å, $\beta = 98\cdot58$ (3)°, $V = 5007$ (4) Å^3, $Z = 4$, $D_m = 1\cdot44$ g cm^{-3} by flotation, $D_x = 1\cdot45$ g cm^{-3}, $\lambda(Mo\,K\alpha) = 0\cdot71069$ Å, $\mu = 28\cdot4$ cm^{-1}, $F(000) = 2216$, $T = 295$ K, $R = 0\cdot050$, 2752 unique observations. The structure consists of discrete $[Ir(O_2)(Ph_2PCH_2CH_2SEt)_2]^+$ cations and

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[BPh$_4$]$^-$ anions. The coordination around the Ir atom is a distorted octahedron, with the peroxo ligand in the equatorial plane, occupying two coordination positions [O—O distance = 1·533 (15) Å] and the chelating Ph$_2$PCH$_2$CH$_2$SEt ligands spanning axial and equatorial positions with *cis* P and S atoms.

Introduction. The crystal structures of dioxygen metal complexes are of interest since many of these complexes play an important role in biological and catalytic processes. We are currently interested in the chemistry of mixed-donor phosphorus–sulfur ligands and reported recently on the synthesis of some dioxygen adducts of iridium complexes containing phosphinethioether chelates (Bressan, Morandini & Rigo, 1983). In this paper we report an X-ray crystallographic investigation of the [Ir(O$_2$)(P—SEt)$_2$]BPh$_4$ (1) derivative with the ligand (ethylthioethyl)diphenylphosphine (P—SEt). The structure can be compared with those reported for other iridium dioxygen adducts and in particular with the crystal structure of the closely related [Ir(O$_2$)(dpe)$_2$]-PF$_6$ (2) [dpe = 1,2-bis(diphenylphosphino)ethane] (McGinnety, Payne & Ibers, 1969; Nolte, Singleton & Laing, 1975). The formation of both dioxygen adducts is irreversible.

Experimental. Yellow crystals of (1) were obtained by slow addition of an NaBPh$_4$ solution (*i*PrOH) to the chloride salt [Ir(O$_2$)(P—SEt)$_2$]Cl dissolved in acetone. Crystal dimensions 0·02 × 0·07 × 0·08 cm, monoclinic, space group $P2_1/n$ determined from Weissenberg and precession photographs. Final lattice parameters determined from 21 strong reflections (16 ≤ 2θ ≤ 34°) centered on an Enraf–Nonius CAD-4 diffractometer. 10 357 reflections were collected in the range −14 ≤ h ≤ 14, 0 ≤ k ≤ 17, 0 ≤ l ≤ 37 (6 ≤ 2θ ≤ 52°, ω/θ scan) using graphite-monochromated Mo $K\alpha$ radiation. Data corrected for Lorentz and polarization effects. Absorption correction according to the empirical ψ-scan method (min. transmission 77·30, max. 99·99, average 91·15%). Four standard reflections (3,5,$\overline{13}$, 3,1,$\overline{17}$, 6$\overline{4}$0, 70$\overline{5}$) checked every 4000 s exposure time. Linear decay (*ca* 29%) of I values of standard reflections throughout data collection, correction applied. Structure solution by conventional Patterson and Fourier methods followed by full-matrix least-squares refinement. H atoms located at calculated positions (C—H = 1·00 Å, B = 5 Å^2 assumed). Final refinement cycles carried out with anisotropic thermal parameters for the non-H atoms of the complex cation. The function minimized is $w(|F_o| - |F_c|)^2$, where w = $1/[\sigma(F_o) + (0·02F_o)^2 + 1·0]$ (Killean & Lawrence, 1969). Scattering factors from *International Tables for X-ray Crystallography* (1974), for all atoms in the neutral (0) oxidation state. 2752 unique reflections having $I > 3\sigma(I)$ used in the calculations (452 variables). Refinement converged to R = 0·050, wR =

0·055 and S = 1·18. Maximum least-squares Δ/σ 0·34 in last cycle. Highest peaks in final difference map of 1·4–0·6 e Å^{-3} around Ir. Calculations performed on a PDP11/44 computer using the Enraf–Nonius software package *SDP-Plus* (B. A. Frenz & Associates Inc., 1981).

Discussion. The crystal structure results from the packing of discrete [Ir(O$_2$)(P—SEt)$_2$]$^+$ cations and [BPh$_4$]$^-$ anions, held together by electrostatic and van der Waals interactions. A general view of the cation is shown in Fig. 1 together with the non-H atom numbering. Atomic positional parameters are given in Table 1, selected bond lengths and angles in Table 2.* The coordination polyhedron around the Ir atom can be described either as a distorted octahedron or as a distorted trigonal bipyramid. The latter geometry was attributed to the related cations [Ir(A_2)(dpe)$_2$]$^+$ (A_2 = O$_2$, S$_2$) (Bonds & Ibers, 1972). However, in agreement with the O—O distance of 1·533 (15) Å found in (1), the dioxygen ligand can be regarded as a peroxo group occupying two coordination sites of the metal atom (Vaška, 1976). Thus an octahedral coordination is assumed, with the dioxygen in the equatorial plane, together with a formal oxidation number III for the Ir atom (Valentine, 1973).

The O—O distance in (1) is close to the upper limit of the range observed for related compounds (Boča, 1983, and references therein) and is equal, within experimental errors, to that found in (2) [1·533 (15) and 1·52 (1) Å, respectively]. This confirms that the O—O bond length is not significantly affected by the other ligands (Nolte,

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a full list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43448 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
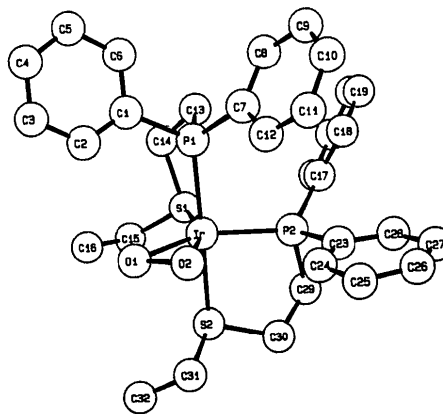

Fig. 1. A general view of the [Ir(O$_2$)(P—SEt)$_2$]$^+$ cation.

Table 1. *Positional and thermal parameters with e.s.d.'s in parentheses*

	x	y	z	$B(Å^2)$†
Ir	0.00088(6)	0.23003(5)	0.10372(2)	5.22(1)
S(1)	−0.0741(4)	0.1978(3)	0.1698(2)	6.7(1)
S(2)	0.0596(4)	0.0725(4)	0.1015(2)	7.0(1)
P(1)	−0.0632(4)	0.3810(4)	0.1133(2)	6.9(1)
P(2)	−0.1717(4)	0.1815(3)	0.0623(2)	5.7(1)
O(1)	0.1772(9)	0.2601(9)	0.1118(4)	9.1(4)
O(2)	0.1189(8)	0.2646(9)	0.0632(4)	7.9(3)
C(1)	0.043(1)	0.467(1)	0.1386(6)	7.1(5)
C(2)	0.157(2)	0.457(2)	0.1464(7)	10.0(7)
C(3)	0.233(2)	0.525(2)	0.1660(8)	10.7(7)
C(4)	0.188(2)	0.605(1)	0.1762(7)	10.1(7)
C(5)	0.070(2)	0.620(1)	0.1698(8)	10.6(7)
C(6)	−0.003(2)	0.550(1)	0.1500(7)	8.4(6)
C(7)	−0.141(1)	0.438(1)	0.0672(6)	7.0(5)
C(8)	−0.237(2)	0.497(1)	0.0680(6)	7.8(5)
C(9)	−0.292(2)	0.543(2)	0.0295(8)	11.0(8)
C(10)	−0.252(2)	0.527(2)	−0.0091(8)	10.7(7)
C(11)	−0.160(2)	0.470(2)	−0.0128(6)	9.4(7)
C(12)	−0.107(2)	0.427(1)	0.0253(6)	7.8(6)
C(13)	−0.161(1)	0.370(1)	0.1559(6)	6.2(5)
C(14)	−0.103(2)	0.310(1)	0.1925(6)	8.2(6)
C(15)	0.038(2)	0.155(2)	0.2117(6)	10.2(7)
C(16)	0.154(3)	0.194(2)	0.2131(8)	17(1)
C(17)	−0.314(1)	0.215(1)	0.0759(5)	6.5(5)
C(18)	−0.375(1)	0.289(1)	0.0568(6)	6.9(5)
C(19)	−0.478(2)	0.315(2)	0.0694(8)	11.0(7)
C(20)	−0.524(1)	0.273(2)	0.1035(7)	12.4(6)
C(21)	−0.464(2)	0.200(2)	0.1197(8)	11.2(7)
C(22)	−0.361(1)	0.170(1)	0.1086(6)	7.1(5)
C(23)	−0.179(1)	0.197(1)	0.0031(5)	5.6(5)
C(24)	−0.079(2)	0.221(1)	−0.0156(5)	7.4(5)
C(25)	−0.086(2)	0.224(1)	−0.0608(6)	8.2(5)
C(26)	−0.192(2)	0.218(2)	−0.0875(6)	10.6(7)
C(27)	−0.294(2)	0.193(2)	−0.0698(7)	9.7(7)
C(28)	−0.283(1)	0.184(1)	−0.0240(6)	6.9(5)
C(29)	−0.169(2)	0.056(1)	0.0655(6)	7.7(6)
C(30)	−0.051(2)	0.021(1)	0.0607(6)	7.9(6)
C(31)	0.187(2)	0.060(1)	0.0740(6)	9.3(6)
C(32)	0.289(2)	0.041(2)	0.1034(9)	11.9(8)
C(33)	−0.331(1)	0.322(1)	0.3156(5)	6.0(4)*
C(34)	−0.420(2)	0.357(1)	0.3364(6)	7.5(5)*
C(35)	−0.534(2)	0.377(1)	0.3132(6)	8.4(5)*
C(36)	−0.560(2)	0.359(1)	0.2697(6)	8.2(5)*
C(37)	−0.478(2)	0.322(1)	0.2462(7)	9.1(6)*
C(38)	−0.365(2)	0.306(1)	0.2696(6)	8.1(5)*
C(39)	−0.179(1)	0.333(1)	0.3930(5)	6.0(4)*
C(40)	−0.099(1)	0.401(1)	0.4112(5)	6.0(4)*
C(41)	−0.080(2)	0.418(1)	0.4568(6)	7.5(5)*
C(42)	−0.135(2)	0.367(1)	0.4838(6)	7.2(5)*
C(43)	−0.211(2)	0.307(1)	0.4699(6)	7.5(5)*
C(44)	−0.236(2)	0.286(1)	0.4235(6)	7.4(5)*
C(45)	−0.151(2)	0.193(1)	0.3391(6)	7.2(5)*
C(46)	−0.193(2)	0.131(1)	0.3058(7)	9.1(6)*
C(47)	−0.144(2)	0.045(1)	0.3050(7)	9.3(6)*
C(48)	−0.065(2)	0.011(2)	0.3364(7)	9.8(6)*
C(49)	−0.020(2)	0.068(1)	0.3721(7)	9.2(6)*
C(50)	−0.069(2)	0.155(1)	0.3717(6)	8.5(5)*
C(51)	−0.100(2)	0.363(1)	0.3141(6)	6.8(4)*
C(52)	0.014(2)	0.335(1)	0.3121(6)	7.1(5)*
C(53)	0.096(2)	0.391(1)	0.2930(6)	8.3(5)*
C(54)	0.056(2)	0.472(1)	0.2779(6)	7.8(5)*
C(55)	−0.047(2)	0.506(2)	0.2795(7)	10.3(6)*
C(56)	−0.130(2)	0.446(1)	0.2990(6)	8.1(5)*
B	−0.193(2)	0.302(1)	0.3395(6)	5.3(5)*

† Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defined as $(4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

Table 2. *Selected interatomic distances (Å) and angles (°) with e.s.d.'s in parentheses*

Ir–S(1)	2.349(5)	Ir–O(1)	2.040(10)
Ir–S(2)	2.394(5)	Ir–O(2)	2.023(10)
Ir–P(1)	2.349(6)	O(1)–O(2)	1.533(15)
Ir–P(2)	2.288(5)		
S(1)–Ir–S(2)	88.4(2)	P(1)–Ir–P(2)	95.6(2)
S(1)–Ir–P(1)	85.5(2)	P(1)–Ir–O(1)	96.0(4)
S(1)–Ir–P(2)	90.9(2)	P(1)–Ir–O(2)	95.2(4)
S(1)–Ir–O(1)	114.8(4)	P(2)–Ir–O(1)	152.5(4)
S(1)–Ir–O(2)	159.1(4)	P(2)–Ir–O(2)	109.8(4)
S(2)–Ir–P(1)	173.9(2)	O(1)–Ir–O(2)	44.3(4)
S(2)–Ir–P(2)	84.9(2)	Ir–O(1)–O(2)	67.2(5)
S(2)–Ir–O(1)	86.2(4)	Ir–O(2)–O(1)	68.4(6)
S(2)–Ir–O(2)	90.4(4)		

The P—SEt hybrid ligands form two chelate rings, with *cis* S and P atoms. The same configuration is apparently present in solution (CH_2Cl_2) at room temperature, as indicated by ^{31}P NMR (*AB* pattern for axial and equatorial P atoms). The two rings have the same twist conformation with a local pseudo-twofold axis running through the Ir atom and the middle of the opposite C—C bond. The bite angles of the two chelates are similar, averaging 85.2°, a typical value for analogous five-membered chelate rings. The S_{eq}—Ir—P_{eq} angle of 90.9(2)°, smaller than the P_{eq}—Ir—P_{eq} angle of 97.0(1)° in (2), may be explained in terms of the different steric requirements of the P—SEt and dpe ligands. The Ir—P(1) bond length [2.349(6) Å, P *trans* to S] is in agreement with those reported for other iridium—phosphine complexes (Stephan, 1984, and references therein). On the contrary, the Ir—P(2) distance [2.288(5) Å, P *trans* to O] is significantly shorter than the Ir—P equatorial distances observed in (2), 2.308(3) and 2.342(3) Å. The difference in the Ir—S bond distances [2.349(5) and 2.394(5) Å for Ir—S(1) and Ir—S(2), respectively] probably reflects the different *trans* influence of oxygen compared with phosphorus. All distances and angles within the P—SEt ligands are unexceptional, as well as in the $[BPh_4]^-$ anion, whose BC_4 core has an approximate tetrahedral geometry with a mean B—C distance of 1.67 Å and C—B—C angles in the range 106–115°.

Singleton & Laing, 1975). The two Ir—O distances are equal within experimental errors and fall within the normal ranges (Boča, 1983). The peroxo ligand does not lie in the plane passing through Ir, S(1) and P(2), atoms O(1) and O(2) being displaced from this plane towards S(2) by 0.33 (1) and 0.10 (1) Å, respectively (Fig. 1).

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Structure of (1,10-Phenanthroline)(salicylaldehydato)copper(II) Nitrate

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Abstract. $[Cu(C_7H_5O_2)(C_{12}H_8N_2)]NO_3$, $M_r = 426.9$, triclinic, $P\bar{1}$, $a = 11.739\,(3)$, $b = 9.062\,(2)$, $c = 8.773\,(2)$ Å, $\alpha = 95.74\,(2)$, $\beta = 112.78\,(2)$, $\gamma = 79.87\,(1)°$, $V = 846.5\,(6)$ Å^3, $Z = 2$, $D_x = 1.67$ Mg m^{-3}, $\lambda(Mo\,K\alpha) = 0.71069$ Å, $\mu = 1.379$ mm^{-1}, $F(000) = 434$, room temperature. $R = 0.043$ for 2380 observed reflections. The Cu ion displays a distorted square-pyramidal coordination, being linked to two O atoms of the salicylaldehydato and two N atoms of the 1,10-phenanthroline ligand and an O atom of the nitrate group. There is a weak interaction between Cu and an O atom of another salicylaldehydato ligand. The (1,10-phenanthroline)-(salicylaldehydato)copper(II) ion is roughly planar, the phenyl group of the salicylaldehydato ligand deviating most from this plane. Deviations in metal–ligand lengths are due to the different characters of the C–O bonds.

Introduction. The behaviour of mixed-chelate complexes (Griesser & Sigel, 1970) has been of great interest for inorganic and bioinorganic chemists in the last few years because their synthesis is a challenge itself, as is their characterization. A series of compounds of formula $M(N–N)(O–O)$ (M = Cu, N–N = *o*-phenanthroline, bipyridine or substituted phenanthroline, O–O = salicylaldehydato or acetylacetonato) have been synthesized, characterized and their stability constants determined by potentiometric methods (Gasque, 1984; Ruiz-Ramírez & Gasque,

1986). The title compound is a member of this series of complexes and its crystal structure is determined in order to obtain the ligand arrangement around the Cu ion.

Experimental. Dark-green pinacoids (0.5 × 0.4 × 0.1 mm), Philips PW 1100 diffractometer, Mo $K\alpha$, graphite monochromator, cell parameters from 25 reflections ($4 \leq \theta \leq 12°$), ω-scan technique, scan width 1°, scan speed 0.03° s^{-1}. 2982 independent reflections with $\theta \leq 25°$; 2380 with $I \geq 2.5\sigma(I)$, *hkl* range −12 to 12, 0 to 10, and 0 to 10. Three standard reflections were measured every 2 h, significant intensity decay was not observed, Lp correction, absorption ignored.

The structure was solved by direct methods (*MULTAN*80, Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinements (*SHELX*76, Sheldrick, 1976), $\sum w(|F_o| - |F_c|)^2$ minimized, $w = [\sigma^2(F_o) + 0.004|F_o|^2]^{-1}$, f, f' and f'' from *International Tables for X-ray Crystallography* (1974). An O atom of the NO_3 group was located in a disordered position, an occupancy factor of 0.5 was assigned according to peak heights of Fourier synthesis. All H atoms from $\Delta\rho$ map and refined with an overall isotropic temperature factor, remaining atoms refined anisotropically, final $R = 0.043$ ($wR = 0.048$) for all observed reflections, max. shift/e.s.d. = 0.27 in U_{12} of Cu, max. and min. peaks in final $\Delta\rho$ map 0.4 [1.05 Å away from N(32)] and −0.3 e Å^{-3}, respectively; IBM-3082 computer.