anisotropically. Phenyl rings refined as rigid polygons (C—C = 1·35 Å, C—C—C = 120°) using H atoms placed in idealized positions with fixed isotropic thermal parameters [U(H) = 0·08 Å^2]. Scattering factors, including terms for anomalous dispersion, taken from *International Tables for X-ray Crystallography* (1974). Refinement based on F with weights of the form $w^{-1} = [\sigma^2(F) + 0{\cdot}001402(F^2)]$. Convergence to conventional R values of $R = 0{\cdot}0452$ and $wR = 0{\cdot}0491$ obtained using 256 variable parameters and 2657 unique reflections with $F^2 > 3\sigma(F^2)$. One intense low-angle reflection ($\bar{2}02$) suffered from severe extinction and was therefore omitted during the refinement process. For final cycle maximum shift/σ = 0·013 with maximum and minimum residual electron densities of +0·97 and −0·91 e Å^{-3} in the vicinities of Au(1) and Au(2). Slope of normal probability plot equal to 1·08 with a goodness-of-fit indicator of 1·273. Two crystallographically independent molecules are present in the asymmetric unit. A perspective view of one is shown in Fig. 1. Atomic positional and equivalent isotropic thermal parameters for both molecules are presented in Table 1.* Bond angles and distances are summarized in Table 2. Fig. 2 shows the packing.

Related literature. The structure of the gold(I) bromide derivative has been previously reported (Porter, Knachel & Fackler, 1986). The preparation and X-ray crystal structures of mononuclear trimethylgold(III) complexes containing sulfoxonium and phosphonium ylide ligands have also been described (Fackler & Paparizos, 1977; Stein, Fackler, Paparizos & Chen, 1981).

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* Lists of structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44002 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (Methanesulfonato)(*meso*-tetraphenylporphinato)iron(III)–Chloroform (1/1)

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Abstract. $[\mathrm{Fe(C_{44}H_{26}N_4)(CH_3O_3S)].CHCl_3}$, M_r = 883·06, orthorhombic, *Pbca*, a = 15·035 (5), b = 25·888 (5), c = 21·007 (4) Å, V = 8176 (6) Å^3, Z = 8, D_x = 1·435 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 6·599 cm^{-1}, F(000) = 3624, T = 293 K, R = 0·075 for 1510 unique observed reflections. One O atom from $\mathrm{CH_3SO_3^-}$ and the four pyrrole N atoms coordinate to Fe to form the five-coordinated complex. The Fe atom is displaced 0·412 (2) Å from the porphinato plane. The average Fe—N bond distance is 2·05 (2) Å, while the Fe—O distance is 1·95 (1) Å. The Fe—N bond lengths indicate that Fe is in a high-spin state.

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Experimental. Approximately 1 g of [Fe(TPP)]$_2$O (TPP = tetraphenylporphinato) was dissolved in 100 ml chloroform, washed with 100 ml aqueous (2 *M*) methanesulfonic acid and the chloroform layer separated. Slow diffusion of pentane into the chloroform solution resulted in large needles.

Black platelet crystal 0·45 × 0·25 × 0·05 mm mounted on a glass fiber. Density not measured. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using variable-speed (0·58–3·4° min^{-1}) ω–2θ scans. Unit cell determined from least squares of angle data for 25 reflections with $12 < 2\theta < 22°$. Numerical absorption correction (Coppens, Leiserowitz & Rabinovich, 1965) based on crystal shape varied from 0·854 to 0·967. Data collected to $(\sin\theta)/\lambda$ of 0·595 Å^{-1}, $0 < h < 17$, $0 < k < 30$, $0 < l < 25$. Three standard reflections (600; $0\overline{64}$; $1\overline{55}$) varied 5·1% over 196 h of data collection, no drift correction applied. 8104 reflections measured. 7950 unique, 6440 reflections with $I < 3\sigma(I)$ considered unobserved. Structure solved by Patterson and Fourier methods. Full-matrix least squares minimized $\sum w(\Delta F)^2$, where $w(F) = 1/\sigma^2(F) = 4F^2/\sigma^2(F^2)$; $\sigma^2(F^2) = (0{\cdot}05F^2)^2 + \sigma^2_{\rm counting}(F^2)$. The H atoms were positioned according to idealized geometry (C—H = 0·95 Å) with isotropic B = 5 Å^2. Fe atom was refined anisotropically, and all other non-H atoms refined isotropically for 238 variables. H atoms were not included in the refinement but used for F_c

Table 1. *Fractional atomic coordinates and isotropic thermal parameters* (Å^2) *for the non-*H *atoms*

	x	y	z	$B_{\rm iso}$
Fe	0·0644 (1)	0·1993 (1)	0·0885 (1)	2·7 (1)*
Cl(1)	0·3943 (4)	0·0132 (2)	0·0382 (3)	7·4 (2)
Cl(2)	0·2800 (4)	−0·0317 (3)	0·1300 (3)	8·5 (2)
Cl(3)	0·3830 (5)	0·0570 (3)	0·1626 (4)	9·2 (2)
S	0·0920 (3)	0·0966 (2)	0·1682 (3)	4·2 (1)
O(1)	0·0740 (8)	0·1525 (4)	0·1606 (5)	3·6 (2)
O(2)	0·1316 (10)	0·0871 (5)	0·2278 (8)	7·7 (4)
O(3)	0·1379 (9)	0·0757 (6)	0·1149 (7)	7·2 (4)
N(1)	−0·0534 (8)	0·2386 (4)	0·1043 (6)	2·3 (3)
N(2)	−0·0024 (9)	0·1551 (4)	0·0227 (6)	2·6 (3)
N(3)	0·1269 (9)	0·2619 (5)	0·1264 (7)	2·8 (3)
N(4)	0·1789 (8)	0·1833 (5)	0·0405 (6)	2·6 (3)
C(Cl)	0·3239 (12)	0·0271 (7)	0·1046 (9)	5·0 (5)
C(S)	0·0155 (17)	0·0701 (11)	0·1686 (13)	9·5 (8)
C(1)	−0·2035 (12)	0·2515 (7)	0·1168 (9)	4·0 (4)
C(2)	−0·1597 (11)	0·2922 (6)	0·1431 (8)	3·4 (4)
C(3)	−0·1114 (11)	0·1013 (6)	−0·0128 (9)	3·8 (4)
C(4)	−0·0342 (11)	0·0883 (7)	−0·0413 (9)	3·7 (4)
C(5)	0·2367 (11)	0·3083 (6)	0·1776 (8)	3·3 (3)
C(6)	0·1602 (12)	0·3325 (6)	0·1878 (9)	3·8 (4)
C(7)	0·3267 (11)	0·1669 (6)	0·0287 (8)	3·0 (3)
C(8)	0·2843 (11)	0·1366 (6)	−0·0135 (8)	3·6 (4)
C(9)	−0·1353 (10)	0·2179 (5)	0·0942 (8)	2·8 (3)
C(10)	−0·0647 (12)	0·2852 (5)	0·1344 (8)	3·3 (4)
C(11)	−0·0916 (10)	0·1453 (6)	0·0266 (8)	2·7 (3)
C(12)	0·0344 (9)	0·1206 (6)	−0·0190 (7)	2·4 (3)
C(13)	0·2167 (11)	0·2646 (6)	0·1393 (8)	3·1 (3)
C(14)	0·0920 (10)	0·3041 (6)	0·1555 (7)	3·0 (3)
C(15)	0·2637 (9)	0·1958 (6)	0·0640 (7)	2·5 (3)
C(16)	0·1911 (10)	0·1457 (6)	−0·0043 (8)	2·9 (3)
C(17)	−0·1543 (9)	0·1723 (6)	0·0601 (7)	2·3 (3)
C(18)	0·0009 (11)	0·3173 (6)	0·1578 (7)	2·8 (3)
C(19)	0·1258 (11)	0·1184 (7)	−0·0334 (9)	3·8 (4)
C(20)	0·2812 (9)	0·2305 (6)	0·1115 (7)	2·2 (3)
C(21)	−0·2494 (11)	0·1552 (6)	0·0614 (8)	3·2 (4)
C(22)	−0·2899 (11)	0·1370 (6)	0·1173 (8)	3·4 (4)
C(23)	−0·3767 (13)	0·1210 (7)	0·1178 (9)	4·8 (4)
C(24)	−0·4263 (14)	0·1216 (8)	0·0627 (10)	5·8 (5)
C(25)	−0·3883 (16)	0·1374 (9)	0·0076 (12)	7·3 (6)
C(26)	−0·3011 (12)	0·1573 (6)	0·0066 (9)	4·3 (4)
C(27)	−0·0260 (11)	0·3667 (7)	0·1880 (8)	3·4 (4)
C(28)	−0·0496 (13)	0·3670 (7)	0·2516 (10)	5·3 (5)
C(29)	−0·0835 (14)	0·4132 (8)	0·2790 (11)	6·5 (6)
C(30)	−0·0913 (13)	0·4543 (7)	0·2436 (10)	5·3 (5)
C(31)	−0·0656 (16)	0·4553 (8)	0·1847 (11)	6·9 (6)
C(32)	−0·0336 (12)	0·4097 (7)	0·1532 (9)	4·6 (5)
C(33)	0·1509 (10)	0·0784 (6)	−0·0802 (9)	3·7 (4)
C(34)	0·1819 (13)	0·0331 (7)	−0·0595 (10)	5·5 (5)
C(35)	0·2166 (13)	−0·0076 (7)	−0·0984 (10)	5·6 (5)
C(36)	0·2094 (13)	−0·0033 (8)	−0·1676 (11)	6·2 (5)
C(37)	0·1714 (16)	0·0505 (9)	−0·1918 (12)	7·5 (6)
C(38)	0·1452 (13)	0·0842 (7)	−0·1444 (9)	5·0 (5)
C(39)	0·3714 (12)	0·2354 (6)	0·1349 (9)	3·2 (4)
C(40)	0·3965 (12)	0·2156 (6)	0·1924 (9)	3·9 (4)
C(41)	0·4797 (15)	0·2146 (7)	0·2162 (11)	6·4 (6)
C(42)	0·5513 (12)	0·2437 (7)	0·1772 (9)	4·8 (5)
C(43)	0·5296 (12)	0·2643 (7)	0·1234 (10)	4·5 (4)
C(44)	0·4350 (12)	0·2586 (6)	0·1008 (8)	3·7 (4)

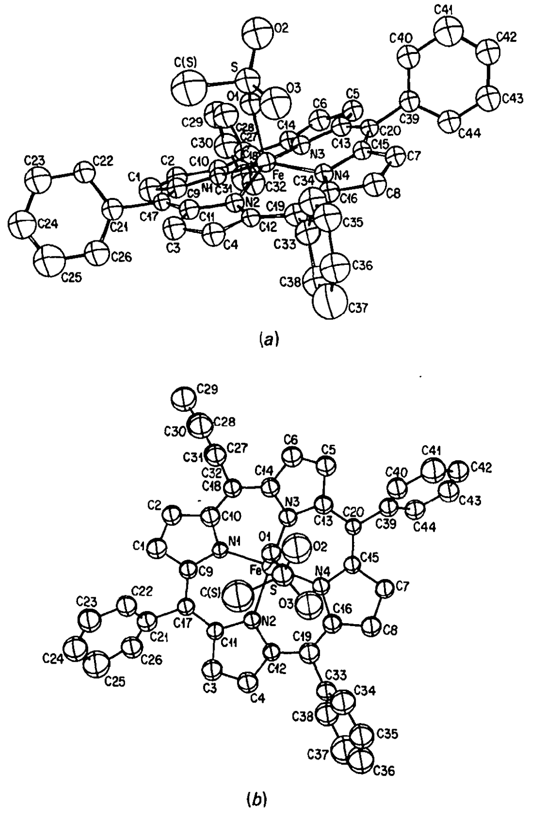
* Anisotropically refined atom. The equivalent isotropic thermal parameter is computed as: $\tfrac{4}{3}[a^2 \times 0{\cdot}00281 + b^2 \times 0{\cdot}00102 + c^2 \times 0{\cdot}00157 + ab(\cos\gamma) \times 0{\cdot}00010 + ac(\cos\beta) \times 0{\cdot}00032 + bc(\cos\alpha) \times -0{\cdot}00033]$.

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

Fe	O(1)	1·94 (1)	C(2)	C(10)	1·45 (2)
Fe	N(1)	2·07 (1)	C(3)	C(4)	1·35 (2)
Fe	N(2)	2·06 (2)	C(3)	C(11)	1·44 (2)
Fe	N(3)	2·04 (2)	C(4)	C(12)	1·41 (2)
Fe	N(4)	2·04 (1)	C(5)	C(6)	1·33 (2)
Cl(1)	C(Cl)	1·79 (2)	C(5)	C(13)	1·42 (2)
Cl(2)	C(Cl)	1·74 (2)	C(6)	C(14)	1·43 (2)
Cl(3)	C(Cl)	1·70 (2)	C(7)	C(8)	1·34 (2)
S	O(1)	1·48 (1)	C(7)	C(15)	1·42 (2)
S	O(2)	1·41 (1)	C(8)	C(16)	1·43 (2)
S	O(3)	1·42 (1)	C(9)	C(17)	1·41 (2)
S	C(S)	1·34 (1)	C(10)	C(18)	1·38 (2)
N(1)	C(9)	1·36 (1)	C(11)	C(17)	1·37 (2)
N(1)	C(10)	1·37 (2)	C(12)	C(19)	1·41 (2)
N(2)	C(11)	1·37 (2)	C(13)	C(20)	1·44 (2)
N(2)	C(12)	1·37 (1)	C(14)	C(18)	1·41 (2)
N(3)	C(13)	1·38 (2)	C(15)	C(20)	1·37 (2)
N(3)	C(14)	1·36 (2)	C(16)	C(19)	1·36 (2)
N(4)	C(15)	1·41 (1)	C(17)	C(21)	1·50 (2)
N(4)	C(16)	1·37 (2)	C(18)	C(27)	1·48 (2)
C(1)	C(2)	1·36 (2)	C(19)	C(33)	1·48 (2)
C(1)	C(9)	1·43 (2)	C(20)	C(39)	1·45 (2)
			C—C phenyl av.		1·37

O(1)	Fe	N(1)	104·2 (4)	C(3)	C(4)	C(12)	110 (1)
O(1)	Fe	N(2)	102·3 (4)	C(6)	C(5)	C(13)	106 (1)
O(1)	Fe	N(3)	99·0 (4)	C(5)	C(6)	C(14)	108 (1)
O(1)	Fe	N(4)	101·3 (5)	C(8)	C(7)	C(15)	110 (1)
N(1)	Fe	N(2)	87·9 (4)	C(7)	C(8)	C(16)	106 (1)
N(1)	Fe	N(3)	86·6 (5)	N(1)	C(9)	C(1)	111 (1)
N(1)	Fe	N(4)	154·5 (4)	N(1)	C(9)	C(17)	126 (1)
N(2)	Fe	N(3)	158·7 (4)	C(1)	C(9)	C(17)	122 (1)
N(2)	Fe	N(4)	88·1 (4)	N(1)	C(10)	C(2)	107 (1)
N(3)	Fe	N(4)	88·0 (4)	N(1)	C(10)	C(18)	127 (1)
Cl(1)	C(Cl)	Cl(2)	106·7 (7)	C(2)	C(10)	C(18)	126 (1)
Cl(1)	C(Cl)	Cl(3)	110·0 (8)	N(2)	C(11)	C(3)	108 (1)
Cl(2)	C(Cl)	Cl(3)	112·2 (7)	N(2)	C(11)	C(17)	128 (1)
O(1)	S	O(2)	110·1 (7)	C(3)	C(11)	C(17)	124 (1)
O(1)	S	O(3)	112·1 (7)	N(2)	C(12)	C(4)	108 (2)
O(2)	S	O(3)	115·4 (8)	N(2)	C(12)	C(19)	124 (1)
Fe	O(1)	S	134·9 (7)	C(4)	C(12)	C(19)	128 (1)
Fe	N(1)	C(9)	123·8 (8)	N(3)	C(13)	C(5)	111 (1)
Fe	N(1)	C(10)	127·7 (9)	N(3)	C(13)	C(20)	123 (1)
C(9)	N(1)	C(10)	108 (1)	C(5)	C(13)	C(20)	125 (1)
Fe	N(2)	C(11)	122·8 (9)	N(3)	C(14)	C(6)	110 (1)
Fe	N(2)	C(12)	126·6 (9)	N(3)	C(14)	C(18)	126 (1)
C(11)	N(2)	C(12)	108 (2)	C(6)	C(14)	C(18)	124 (1)
Fe	N(3)	C(13)	124·7 (9)	N(4)	C(15)	C(7)	108 (2)
Fe	N(3)	C(14)	129·7 (9)	N(4)	C(15)	C(20)	126 (1)
C(13)	N(3)	C(14)	104·4 (1)	C(7)	C(15)	C(20)	127 (1)
Fe	N(4)	C(15)	123·1 (9)	N(4)	C(16)	C(8)	110 (1)
Fe	N(4)	C(16)	126·8 (8)	N(4)	C(16)	C(19)	126 (1)
C(15)	N(4)	C(16)	107 (2)	C(8)	C(16)	C(19)	124 (1)
C(2)	C(1)	C(9)	105 (1)	C(13)	C(20)	C(15)	125 (1)
C(1)	C(2)	C(10)	109 (1)	C—C—C phenyl		range	114–129
C(4)	C(3)	C(11)	106 (1)				

calculation. $R = 0{\cdot}075$, $wR = 0{\cdot}089$ and $S = 1{\cdot}79$. Final $(\Delta/\sigma)_{\max} = 0{\cdot}01$, $\Delta\rho_{\max} = 0{\cdot}53\,(4)\,\text{e}\,\text{Å}^{-3}$ on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); programs used those of Enraf–Nonius (1982) *SDP* package. Table 1 gives atom coordinates and Table 2 gives selected bond distances and angles.* Figs. 1(*a*) and 1(*b*) give the atom numbering and a perspective drawing of the molecule. Fe atom is displaced 0·412 (2) and 0·415 (2) Å from the porphinato core and N_4 plane, respectively. There are no contacts shorter than 4·3 Å between the Fe atom and non-H atoms in neighboring molecules. The average Fe—N bond length is 2·05 (2) Å, while the Fe—O distance is 1·95 (1) Å. Fe—N bond lengths indicate that Fe is in a high-spin state (Scheidt & Reed, 1981). The Fe—O—S angle is 134·9 (7)°. Cl atoms of chloroform and C, O(2), O(3) atoms of $CH_3SO_3^-$ have large temperature factors and may be disordered; this led to the high *R* values quoted.



(*a*)

(*b*)

Fig. 1. *ORTEP* diagram (Johnson, 1976) and numbering scheme for non-H atoms. Chloroform not included. 50% probability surfaces. (*a*) Side view. (*b*) View from above.

Related literature. The structure analysis of the title compound is part of a program of charge density studies on iron porphyrins (Lecomte, Blessing, Coppens & Tabard, 1986).

A number of related five-coordinate oxygen-ligated ferric porphyrinates have been studied: methoxyiron(III) *meso*-porphyrin IX DME (dimethyl ester) [Fe—O = 1·842 (4) Å; Hoard, Hamor, Hamor & Caughey (1965)]; *μ*-oxo-bis[*meso*-tetraphenylporphinato)iron(III)] [Fe—O = 1·763 (1) Å; Hoffman, Collins, Day, Fleischer, Srivastava & Hoard (1972)]; perchlorato(*meso*-tetraphenylporphinato)iron(III) [Fe—O = 2·029 (4) Å; Reed, Mashiko, Bently, Kastner, Scheidt, Spartalian & Lang (1979)]; (octaethylporphinato)perchloratoiron(III) [Fe—O = 2·067 (9) Å; Masuda, Taga, Osaki, Sugimoto, Yoshida & Ogoshi (1980)]; *μ*-oxo-FTFFe$_2$ [FTF = *N,N'*-bis(10,15,20-triphenyl-5-porphinyl-*o*-phenyl)urea] [Fe—O = 1·78 (2) Å, Landrum, Grimmet, Haller, Scheidt & Reed (1981)]; methoxy(*meso*-tetraphenylporphinato)iron(III) [Fe—O = 1·816 (2) Å; Lecomte, Chadwick, Coppens & Stevens (1983); (TTOPFe)$_2$ [TTOP = 5-(2-hydroxyphenyl)-10,15,20 tritolylporphine] [Fe—O-(phenolate) = 1·847 (2) Å; Goff, Shiomura, Lee & Scheidt (1984)]; benzenesulfinato(*meso*-tetraphenylporphinato)iron(III) [Fe—O = 1·92 (1) Å; Cocolios, LaGrange, Guilard, Oumous & Lecomte (1984)]; acetato(*meso*-tetraphenylporphinato)iron(III) [Fe—O = 1·898 (4) Å; Oumous, Lecomte, Cocolios & Guilard (1984)]; *μ*-sulfato-bis(*meso*-tetraphenylporphinato)-iron(III) [Fe—O = 1·894 (4) Å; Scheidt, Lee, Bartczak & Hatano (1984)]; and *μ*-oxo-bis[5,15-dimethyloctaethylporphinato)iron(III)] [Fe—O = 1·752 (1) Å; Lay, Buchler, Kenny & Scheidt (1986)]. All of these complexes except the two perchlorato derivatives are high-spin species.

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* Lists of structure factors and calculated H-atom positions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43965 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (*E*)-[(2-Isopropylphenyl)(phenyl)methylene](mesityl)phosphine

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Abstract. $C_{25}H_{27}P$, $M_r = 358{\cdot}46$, triclinic, $P\bar{1}$, $a = 7{\cdot}991\,(1)$, $b = 9{\cdot}155\,(1)$, $c = 14{\cdot}937\,(4)$ Å, $\alpha = 102{\cdot}98\,(1)$, $\beta = 90{\cdot}20\,(2)$, $\gamma = 100{\cdot}63\,(1)°$, $V = 1045{\cdot}4\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}139$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 1{\cdot}3$ cm^{-1}, $F(000) = 384$, $T = 295$ K, $R = 0{\cdot}0487$ for 4106 observed reflections with $I > 2\sigma(I)$. The central phosphaalkene part of the molecule is planar within 0·027 (5) Å. The angles between this plane and the attached phenyl rings are 47·91 (8), 66·64 (8) and 69·49 (9)° respectively. The P=C distance is 1·682 (2) Å.

Experimental. An *E*-isomer crystal was obtained by crystallization from a 2:1 *E*/*Z*-isomer mixture in pentane. Colourless rhombus-shaped crystal 0·88 × 0·63 × 0·33 mm mounted under nitrogen in a Lindemann-glass capillary. Unit-cell parameters and their e.s.d.'s derived from a least-squares treatment of 17 reflections ($15{\cdot}4 < \theta < 18{\cdot}6°$). 5033 intensity data were collected within one half of the reflection sphere ($0 \le h \le 10$; $-12 \le k \le 11$; $-19 \le l \le 19$; $1{\cdot}40 < \theta < 28{\cdot}0°$) on an Enraf–Nonius CAD-4*F* diffractometer, Zr-filtered Mo $K\alpha$ radiation, $\omega/2\theta$ scan mode with $\Delta\omega = (0{\cdot}75 + 0{\cdot}35\tan\theta)°$. Three reference reflections ($\bar{2}\bar{2}\bar{7}$, $4\bar{1}\bar{2}$, $2\bar{3}\bar{3}$) measured every hour of X-ray exposure time showed a consistent linear increase of 5% in intensity during the 114 h of X-ray exposure time. Intensity data were corrected for this effect and for Lp. No absorption correction. Standard deviations of the intensities as obtained by counting statistics were increased according to an analysis of the excess variance of the reference reflections: $\sigma^2(I) = \sigma^2_{cs}(I) + (0{\cdot}0071I)^2$ resulting in 4106 observed reflections with $I > 2\sigma(I)$ (McCandlish, Stout & Andrews, 1975). Structure solved by direct methods (*SHELXS*86; Sheldrick, 1986), the solution with the best figure of merit revealed all non-H atoms. All non-H atoms were refined with anisotropic thermal parameters by full-matrix least-squares techniques, minimizing $\sum w\Delta F^2$, using the *SHELX*76 package (Sheldrick, 1976). H atoms were introduced on calculated positions (C—H = 0·98 Å)

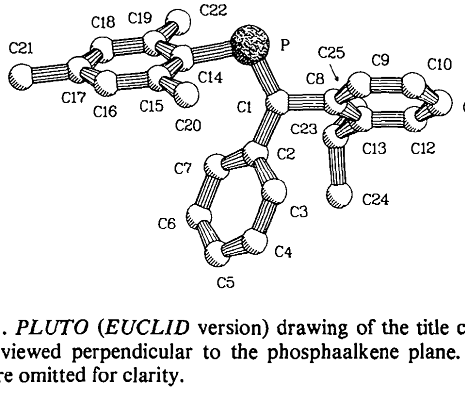

Fig. 1. *PLUTO* (*EUCLID* version) drawing of the title compound as viewed perpendicular to the phosphaalkene plane. H atoms were omitted for clarity.

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