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

Cu–N(1)	2·005 (2)	Cu–N(4)	2·016 (2)
Cu–N(2)	2·010 (2)	Cu–O(9)	2·238 (2)
Cu–N(3)	2·031 (2)		
N(1)–Cu–N(2)	91·0 (1)	N(2)–Cu–N(3)	166·4 (1)
N(1)–Cu–N(3)	92·9 (1)	N(1)–Cu–O(9)	97·2 (1)
N(2)–Cu–N(4)	91·7 (1)	N(2)–Cu–O(9)	101·7 (1)
N(3)–Cu–N(4)	81·3 (1)	N(3)–Cu–O(9)	90·7 (1)
N(1)–Cu–N(4)	164·8 (1)	N(4)–Cu–O(9)	97·0 (1)

X-ray Crystallography (1974) and programs used were those of Enraf–Nonius (1982) *SDP.** Table 1 gives the atom coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the complex cation with the numbering scheme.

Related literature. Square-pyramidal geometry for five coordinate Cu^{II} is well established (O'Brien, 1984; Hathaway, 1982, 1983). Structures of similar phenanthroline complexes with an apical water molecule have been determined, but these have mixed atom coordination in the basal plane. These include complexes with the following ligands: malonate (Kwik, Ang, Chan, Chebolu & Koch, 1986), oxalate (Fabretti, Franchini, Zannini & Divaira, 1985), phthalate (Krstanović, Karanović & Stojaković, 1985), and acetylacetone (Bailey, Fenton, Franklin & Hall, 1980).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44033 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
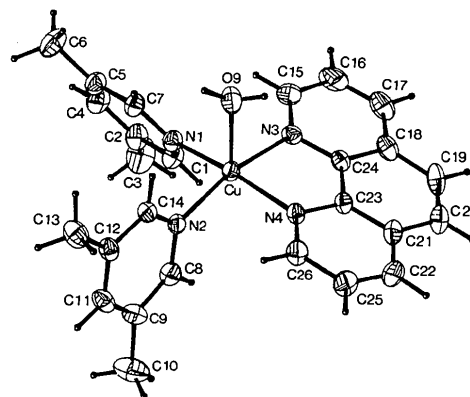

Fig. 1. *ORTEP*II diagram (Johnson, 1976) and atom-numbering scheme. Non-H ellipsoids at 30% probability level, H atoms given arbitrary radii.

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Structure of Dicarbonyl(η⁵-cyclopentadienyl)(*O,O'*-diethyl dithiophosphato)iron(II)

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Abstract. [Fe(C₅H₅)(C₄H₁₀O₂PS₂)(CO)₂], $M_r =$ 362·18, monoclinic, $P2_1/n$, $a = 7·440$ (1), $b = 14·545$ (1), $c = 14·454$ (1) Å, $\beta = 94·415$ (3)°, $V = 1559·5$ (1) Å³, $Z = 4$, $D_x = 1·543$ Mg m⁻³, λ(Mo $K\alpha$) $= 0·7107$ Å, $\mu = 1·329$ mm⁻¹, $F(000) = 744$, room temperature, final $R = 0·042$ ($wR = 0·041$) for 2517 [$I > 3\sigma(I)$] observed reflections. The compound belongs to a series of cyclopentadienyl–Fe^{II} derivatives which contains a very uncommon monodentate dithiophosphate ester ligand, linked to Fe through an S atom. The cyclopentadienyl ligand is situated on one side of the Fe atom with the carbonyl and dithiophosphate ligands on

Table 1. *Atomic parameters for* $[Fe(C_5H_5)(C_4H_{10}O_2PS_2)(CO)_2]$

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*a_ia_j\cos(a_i,a_j) \times 10^5.$$

	x	y	z	$U_{eq}(Å^2)$
Fe	0.28452 (6)	0.11220 (3)	0.83482 (3)	395 (1)
P	0.61401 (12)	0.26484 (6)	0.94168 (6)	436 (3)
S1	0.50026 (13)	0.14003 (6)	0.95458 (7)	532 (3)
S2	0.80636 (16)	0.28689 (8)	1.03681 (8)	689 (4)
O1	0.2141 (4)	0.2981 (2)	0.7687 (2)	748 (11)
O2	0.5683 (5)	0.0729 (3)	0.7133 (2)	934 (15)
O3	0.4634 (3)	0.34289 (17)	0.93588 (16)	505 (8)
O4	0.6699 (3)	0.26875 (17)	0.83785 (17)	492 (8)
C1	0.2497 (5)	0.2268 (3)	0.7962 (3)	496 (12)
C2	0.4569 (5)	0.0898 (3)	0.7604 (3)	574 (13)
C3	0.3551 (7)	0.3606 (3)	1.0135 (3)	747 (17)
C4	0.7527 (6)	0.3506 (3)	0.8025 (3)	591 (14)
C5	0.2371 (7)	0.4388 (4)	0.9909 (4)	838 (20)
C6	0.7553 (7)	0.3408 (4)	0.6995 (3)	751 (17)
C7	0.2096 (6)	−0.0248 (3)	0.8588 (3)	613 (4)
C8	0.1635 (6)	0.0289 (3)	0.9319 (3)	637 (15)
C9	0.0425 (6)	0.0966 (3)	0.8977 (4)	757 (19)
C10	0.0145 (6)	0.0846 (4)	0.8007 (4)	794 (19)
C11	0.1187 (7)	0.0084 (3)	0.7770 (3)	719 (16)

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

Fe—Cp*	1.720 (1)	Cp*—Fe—S1	119.1 (1)
Fe—S1	2.304 (1)	Cp*—Fe—C1	122.6 (1)
Fe—C1	1.770 (4)	Cp*—Fe—C2	124.9 (1)
Fe—C2	1.767 (4)	C1—Fe—C2	94.4 (2)
Fe—C7	2.105 (4)	S1—Fe—C2	89.7 (1)
Fe—C8	2.108 (4)	S1—Fe—C1	98.5 (1)
Fe—C9	2.090 (5)	O3—P—O4	99.1 (1)
Fe—C10	2.070 (4)	S2—P—O4	115.7 (1)
Fe—C11	2.083 (5)	S2—P—O3	113.5 (1)
P—S1	2.018 (1)	S1—P—O4	105.2 (1)
P—S2	1.933 (1)	S1—P—O3	110.3 (1)
P—O3	1.593 (3)	S1—P—S2	112.1 (1)
P—O4	1.588 (2)	Fe—S1—P	111.2 (1)
O1—C1	1.135 (5)	P—O3—C3	121.0 (2)
O2—C2	1.139 (5)	P—O4—C4	120.7 (2)
O3—C3	1.453 (5)	Fe—C1—O1	174.6 (3)
O4—C4	1.451 (5)	Fe—C2—O2	178.1 (4)
C3—C5	1.457 (7)	O3—C3—C5	109.0 (4)
C4—C6	1.498 (6)	O4—C4—C6	108.1 (3)
C7—C8	1.379 (6)	C8—C7—C11	108.6 (4)
C7—C11	1.402 (6)	C7—C8—C9	108.6 (4)
C8—C9	1.398 (6)	C8—C9—C10	107.8 (4)
C9—C10	1.412 (8)	C9—C10—C11	107.2 (5)
C10—C11	1.409 (7)	C7—C11—C10	107.7 (4)

* Cp is the centre of the cyclopentadienyl ring.

the opposite side. Fe—CO = 1.767 (4), 1.770 (4) Å; Fe—S = 2.304 (1) Å; distance from Fe to centre of ring = 1.720 (1) Å.

Experimental. The title compound was synthesized by Morán & Cuadrado (1985) and provided by Professor J. G. Rodríguez, Facultad de Ciencias, Departamento de Química Orgánica, Universidad Autónoma de Madrid. Red crystals $0.50 \times 0.16 \times 0.14$ mm mounted on a glass fiber. 4521 unique reflections ($R_{int} = 0.01$) measured with an Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; $\omega/2\theta$ scan mode. Unit-cell parameters determined from least-squares analysis of angle data for 48 reflections with $2 < \theta < 30°$. Data collected to $(\sin\theta)/\lambda$ of 0.70 Å^{-1}, $-10 < h < 10$, $0 < k < 20$, $0 < l < 20$. Two standard reflections ($10\bar{6}$, $20\bar{6}$) checked every 90 min, intensities showed no appreciable variation. Systematic absences consistent with $P2_1/n$. 2517 observed reflections [$I > 3\sigma(I)$]. In reducing the data Lorentz and polarization factors were applied, but no absorption correction was made.

Structure solved with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) which gave positions of Fe, S and P atoms and *DIRDIF* (Beurskens, Bosman, Doesburg, Gould, van den Hark, Prick, Noordik, Beurskens & Parthasarathi, 1981) which revealed all O and C atoms. All H atoms located from a difference synthesis and included fixed in the refinement with an overall isotropic temperature factor. Empirical weighting scheme (*PESOS*; Martínez-Ripoll & Cano, 1975) to give no trends in $\langle w\Delta F\rangle$ vs $\langle F_o\rangle$ and $\langle(\sin\theta)/\lambda\rangle$; final $R = 0.042$ ($wR = 0.041$) for all observed reflections, $S = 1.23$, $(\Delta/\sigma)_{max} = 0.005$ and $(\Delta/\sigma)_{av} = 0.001$. Min. and max. residual electron density -1.10 and 0.53 e Å^{-3}, both near Fe-atom
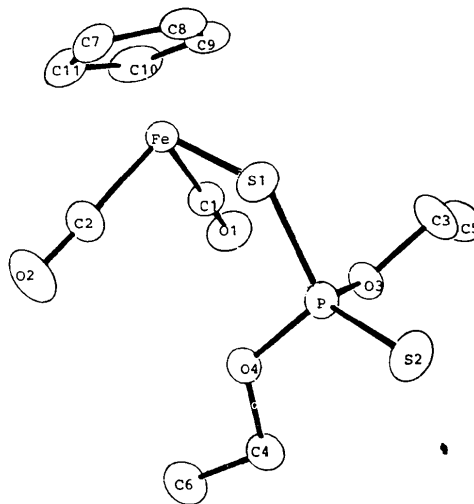

Fig. 1. General view of the molecule. H atoms are omitted for clarity.

position. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). Calculations were carried out using the *XRAY* system (Stewart, Kundell & Baldwin, 1970) and *PARST* (Nardelli, 1983) on a VAX 11/750 computer.

Table 1* lists fractional coordinates and equivalent

* 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 44021 (36 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
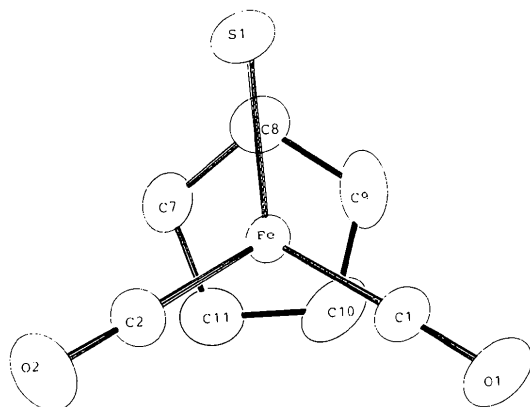
Fig. 2. Projection of the Fe environment onto the cyclopentadienyl plane.

isotropic thermal parameters, while bond distances and angles may be found in Table 2. Fig. 1 displays a view of the molecule with the atom numbering and Fig. 2 shows a projection of the Fe-atom environment onto the cyclopentadienyl plane.

Related literature. Structures of two other related cyclopentadienyl–Fe^{II} derivatives have been published (Sanz-Aparicio, Martínez-Carrera & García-Blanco, 1986a,b). For other dithiophosphate ester ligands in a monodentate configuration see Molloy, Hossain, van

der Helm, Zuckerman & Haiduc (1979) and Shetty & Fernando (1970).

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The Structure of 3,5-Dinitroisoxazole*

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Abstract. $C_3HN_3O_5$, $M_r = 159.06$, $P\bar{1}$, $a = 9.858$ (3), $b = 10.382$ (3), $c = 11.675$ (2) Å, $\alpha = 115.92$ (2), $\beta = 111.37$ (2), $\gamma = 60.91$ (3)°, $V = 921.39$ Å^3, $Z = 6$, $D_x = 1.720$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha_1) = 0.70927$ Å, $\mu = 0.16$ mm^{-1}, $F(000) = 480$, room temperature, final $R = 0.044$ for 1215 observed reflections with $I > 2\sigma(I)$ out of 2411 independent reflections. There are three molecules in the asymmetric unit. The packing is very inefficient as the planes of these three molecules are almost normal to each other. The geometries of the three molecules are very similar. Rings are planar within 0.005 Å. Possible C—H···O hydrogen bonds (3.35 Å) are present.

Experimental. Title compound prepared by method of Golod, Novatskii & Bagal (1973). Crystals for X-ray diffraction grown by sublimation. Selected crystal *ca* 0.43 × 0.19 × 0.11 mm. CAD-4 diffractometer, θ–2θ scan. Scan range $1 + 0.34\tan\theta°$, scan speed 1.6 to 5.5° min^{-1}. Background first and last 1/6 of scan. Graphite-monochromated Mo $K\alpha$ radiation. Unit cell, 25 reflections $7 < \theta < 20°$. No absorption corrections. $(\sin\theta)/\lambda$ max. $= 0.540$ Å^{-1}. Index range $-10 \le h \le 10$, $-11 \le k \le 11$, $-12 \le l \le 12$; 4812 reflections

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