Table 1. *Atomic coordinates and equivalent U factors* (Å^2)

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

	x	y	z	U_{eq}
Cs1	0	−0·0191 (1)	0·25	0·0660 (3)
Cs2	0·5	0·1219 (1)	0·25	0·0668 (3)
N1	0·0969 (13)	0·1733 (10)	0·0696 (13)	0·084 (3)
N2	0·2249 (14)	0·3226 (10)	0·2446 (15)	0·090 (3)
N3	0·1801 (12)	0·4131 (10)	0·4814 (13)	0·082 (3)
C1	0·1511 (12)	0·2464 (9)	0·1577 (12)	0·063 (3)
C2	0·1941 (12)	0·3676 (8)	0·3710 (14)	0·065 (3)

Table 2. *Bonds lengths* (Å) *and angles* (°) *in the dicyanamide anion*

N1—C1	1·18 (2)	N2—C2	1·31 (2)
C1—N2	1·26 (2)	C2—N3	1·13 (2)
N1—C1—N2	172 (2)	N3—C2—N2	173 (2)
C1—N2—C2	124 (2)		

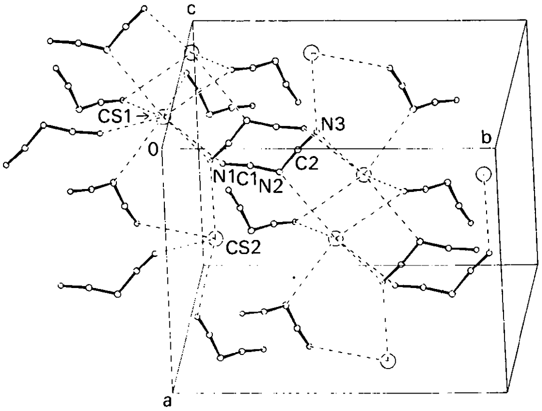

Fig. 1. View of the crystal structure. The dashed lines represent the Cs—N contacts shorter than 3·4 Å.

$wR = 0·0496$, the maximum shift/e.s.d. was 0·002, $\Delta\rho$ between −1·09 and 1·27 e Å^{-3} and the highest peaks were around the Cs$^+$ ions, $w = 1/\sigma^2(F)$, 56 parameters refined. Table 1 presents final atomic parameters, the geometry of the dicyanamide anion is shown in Table 2 and the *ORTEP*II (Johnson, 1976) view of the crystal packing is given in Fig. 1.*

Related literature. The theoretically predicted and the observed geometry of the dicyanamide anion was discussed by Jensen, Klewe & Tjelta (1977) and its coordination properties by Kapshuk & Skopenko (1986).

The author thanks Dr Zbigniew Ciunik for a gift of sodium dicyanamide.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54108 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1991). C**47**, 2199–2201

Structure of an Fe–S Cluster Complex

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(*Received* 14 *September* 1990; *accepted* 6 *March* 1991)

Abstract. Hexacarbonyl-1κ^3C,2κ^3C-μ-methoxymethanethiolato-μ-benzenethiolatodiiron(*Fe–Fe*), [$Fe_2(C_2H_5OS)(C_6H_5S)(CO)_6$], $M_r = 466·04$, monoclinic, $P2_1/n$, $a = 9·023$ (3), $b = 12·081$ (5), $c = 16·720$ (11) Å, $\beta = 97·04°$, $V = 1808$ (2) Å^3, $Z = 4$, $D_x = 1·71$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1·90$ mm^{-1}, $F(000) = 936$, room temperature, $R = 0·038$ for 1490 reflections with $I > 3\sigma(I)$. The two Fe

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Table 1. *Fractional atomic coordinates and thermal parameters*

$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ac(\cos\beta)B(1,3)]$.

	x	y	z	B_{eq}(Å^2)
Fe(1)	0·1079 (1)	0·0903 (1)	−0·2164 (7)	3·27 (2)
Fe(2)	−0·1049 (1)	−0·0319 (1)	−0·2708 (8)	3·66 (3)
S(1)	0·1201 (3)	−0·0959 (2)	−0·2194 (1)	3·56 (4)
S(2)	0·0423 (3)	0·0597 (2)	−0·3490 (1)	3·65 (5)
O(1)	0·0320 (9)	0·0989 (6)	−0·0526 (4)	6·9 (2)
O(2)	−0·0004 (8)	0·3152 (5)	−0·2497 (4)	5·7 (2)
O(3)	0·4282 (8)	0·1346 (6)	−0·1843 (5)	7·1 (2)
O(4)	−0·3303 (8)	0·1386 (6)	−0·3142 (5)	7·0 (2)
O(5)	−0·2460 (8)	−0·0822 (8)	−0·1275 (4)	7·5 (2)
O(6)	−0·2325 (9)	−0·2164 (6)	−0·3714 (5)	7·4 (2)
O(7)	0·250 (1)	−0·2081 (6)	−0·0918 (5)	8·1 (2)
C(1)	0·063 (1)	0·0958 (7)	−0·1161 (6)	4·5 (2)
C(2)	0·0423 (9)	0·2300 (8)	−0·2370 (5)	3·8 (2)
C(3)	0·304 (1)	0·1191 (7)	−0·1974 (6)	4·5 (2)
C(4)	−0·244 (1)	0·0722 (8)	−0·2992 (6)	5·0 (2)
C(5)	−0·190 (1)	−0·0650 (9)	−0·1834 (6)	5·1 (2)
C(6)	−0·183 (1)	−0·1468 (9)	−0·3323 (6)	4·7 (2)
C(7)	0·113 (1)	−0·1653 (8)	−0·1207 (6)	5·0 (2)
C(8)	0·351 (2)	−0·130 (1)	−0·0526 (9)	9·0 (4)
C(11)	0·1711 (9)	−0·0267 (7)	−0·3936 (5)	3·6 (2)
C(12)	0·3214 (9)	0·0020 (8)	−0·3843 (5)	4·2 (2)
C(13)	0·417 (1)	−0·0578 (8)	−0·4245 (6)	5·0 (2)
C(14)	0·369 (1)	−0·1462 (8)	−0·4732 (6)	4·9 (2)
C(15)	0·220 (1)	−0·1722 (9)	−0·4819 (6)	5·5 (3)
C(16)	0·117 (1)	−0·1140 (8)	−0·4427 (6)	4·8 (2)

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

Fe(1)—Fe(2)	2·504 (1)	O(2)—C(2)	1·110 (7)
Fe(1)—S(1)	2·254 (2)	O(3)—C(3)	1·135 (7)
Fe(1)—S(2)	2·256 (2)	O(4)—C(4)	1·127 (7)
Fe(1)—C(1)	1·773 (8)	O(5)—C(5)	1·135 (8)
Fe(1)—C(2)	1·808 (7)	O(6)—C(6)	1·123 (8)
Fe(1)—C(3)	1·790 (8)	O(7)—C(7)	1·377 (9)
Fe(2)—S(1)	2·245 (2)	O(7)—C(8)	1·418 (11)
Fe(2)—S(2)	2·261 (2)	C(11)—C(12)	1·390 (8)
Fe(2)—C(4)	1·797 (8)	C(11)—C(16)	1·388 (8)
Fe(2)—C(5)	1·776 (8)	C(12)—C(13)	1·365 (9)
Fe(2)—C(6)	1·820 (8)	C(13)—C(14)	1·382 (10)
S(1)—C(7)	1·860 (7)	C(14)—C(15)	1·371 (10)
S(2)—C(11)	1·790 (6)	C(15)—C(16)	1·392 (9)
O(1)—C(1)	1·130 (7)		

Fe(2)—Fe(1)—S(1)	56·0 (1)	S(2)—Fe(2)—C(6)	105·2 (2)
Fe(2)—Fe(1)—S(2)	56·4 (1)	C(4)—Fe(2)—C(5)	91·1 (3)
Fe(2)—Fe(1)—C(1)	96·5 (2)	C(4)—Fe(2)—C(6)	100·0 (3)
Fe(2)—Fe(1)—C(2)	105·2 (2)	C(5)—Fe(2)—C(6)	96·7 (3)
Fe(2)—Fe(1)—C(3)	151·1 (2)	Fe(1)—S(1)—Fe(2)	67·7 (1)
S(1)—Fe(1)—S(2)	79·8 (1)	Fe(1)—S(1)—C(7)	115·0 (3)
S(1)—Fe(1)—C(1)	94·3 (2)	Fe(2)—S(1)—C(7)	111·5 (3)
S(1)—Fe(1)—C(2)	160·7 (2)	Fe(1)—S(2)—Fe(2)	67·4 (1)
S(1)—Fe(1)—C(3)	98·5 (2)	Fe(1)—S(2)—C(11)	113·5 (2)
S(1)—Fe(1)—C(1)	150·8 (2)	Fe(2)—S(2)—C(11)	114·3 (2)
S(2)—Fe(1)—C(2)	85·7 (2)	C(7)—O(7)—C(8)	114·5 (8)
S(2)—Fe(1)—C(3)	110·0 (2)	Fe(1)—C(1)—O(1)	178·8 (7)
C(1)—Fe(1)—C(2)	92·2 (3)	Fe(1)—C(2)—O(2)	178·7 (6)
C(1)—Fe(1)—C(3)	99·0 (3)	Fe(1)—C(3)—O(3)	178·1 (6)
C(2)—Fe(1)—C(3)	98·4 (3)	Fe(2)—C(4)—O(4)	177·5 (7)
Fe(1)—Fe(2)—S(1)	56·3 (1)	Fe(2)—C(5)—O(5)	177·4 (7)
Fe(1)—Fe(2)—S(2)	56·2 (1)	Fe(2)—C(6)—O(6)	178·7 (7)
Fe(1)—Fe(2)—C(4)	99·4 (2)	S(1)—C(7)—O(7)	110·6 (6)
Fe(1)—Fe(2)—C(5)	102·8 (2)	S(2)—C(11)—C(12)	118·8 (5)
Fe(1)—Fe(2)—C(6)	152·1 (2)	S(2)—C(11)—C(16)	119·3 (5)
S(1)—Fe(2)—S(2)	79·8 (6)	C(12)—C(11)—C(16)	121·6 (6)
S(1)—Fe(2)—C(4)	155·7 (2)	C(11)—C(12)—C(13)	118·7 (7)
S(1)—Fe(2)—C(5)	93·8 (2)	C(12)—C(13)—C(14)	121·6 (8)
S(1)—Fe(2)—C(6)	103·1 (2)	C(13)—C(14)—C(15)	118·6 (8)
S(2)—Fe(2)—C(4)	86·8 (2)	C(14)—C(15)—C(16)	122·4 (8)
S(2)—Fe(2)—C(5)	158·1 (2)	C(11)—C(16)—C(15)	117·1 (7)

and S atoms in a molecule form a 'butterfly-like' four-membered ring with an Fe—Fe bond distance of 2·504 (1) Å. The average Fe—S and Fe—C bond distances are 2·254 (2) and 1·794 (8) Å, respectively, and the average Fe—S—Fe, S—Fe—S and Fe—C—O bond angles are 67·6 (1), 79·8 (4) and 178·2 (6)°, respectively. The C_6H_5 group adopts an axial position and the CH_2OCH_3 group an equatorial position.

Experimental. The title compound was prepared according to Song, Hu, Zhang & Feng (1988). Dark red prismatic crystals were obtained by evaporation from a petroleum ether/dichloromethane solution. A crystal of dimensions 0·5 × 0·5 × 0·6 mm was mounted on a glass fibre. Accurate cell parameters were obtained from centred setting angles of 25 reflections in the range $12 < \theta < 15°$. An Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71073$ Å) was used. Diffraction intensities in the range $2 < \theta < 25°$ were measured using the ω–2θ scan mode, index range h − 10 → 10, k 0 → 14, l 0 → 20. Deviation of three standard reflections, measured after each group of 200 reflections, was less than 1·4%. 3460 unique reflections were collected of which 1490 were considered observed [$I > 3\sigma(I)$]. The intensities were corrected for Lorentz and polarization effects, no absorption correction was made. The structure was solved by Patterson methods and from subsequent electron density difference maps; all H atoms, except three which were placed in calculated positions, were located from these maps. The structure was refined by full-matrix least-squares calculations on F, using unit weights, anisotropic temperature factors for non-H atoms and isotropic temperature factors for

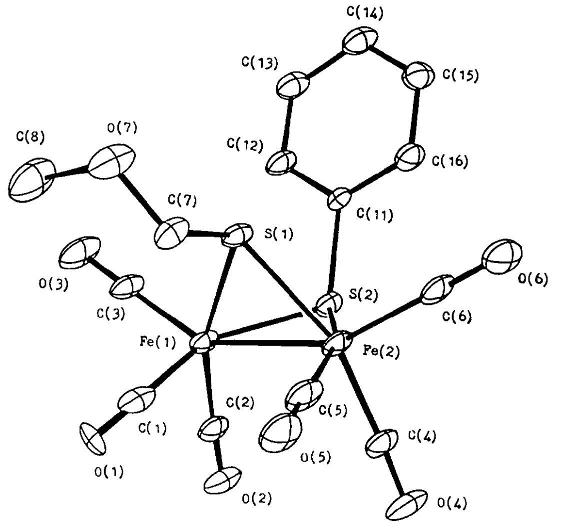

Fig. 1. Perspective drawing of the molecular structure of (μ-C_6H_5S)(μ-CH_2OCH_3S)$Fe_2(CO)_6$.

H atoms. Structure refinement converged to $R = 0{\cdot}038$, $wR = 0{\cdot}043$ and $S = 2{\cdot}21$ for 267 parameters. Maximum shift/e.s.d. in final cycle was 1·64 and no residual electron density > $|0{\cdot}36|$ e Å^{-3} was found. Calculations were carried out on a PDP11/34 computer with the *SDP* system (B. A. Frenz & Associates, Inc., 1982). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B).

The fractional coordinates and thermal parameters for non-H atoms are given in Table 1* and selected bond lengths and angles in Table 2. A perspective drawing of the molecule is shown in Fig. 1.

Related literature. The cluster molecule has a 'butterfly-like' four-membered-ring skeleton composed of two Fe and two S atoms. The Fe(1)—Fe(2) bond of 2·254 (2) Å arises from the d^2sp^3 orbitals of Fe forming a bent metal–metal bond (Dahl, Martell & Wampler, 1961). Each Fe is surrounded by three carbonyls and two S atoms forming a regular tetragonal pyramid. The average Fe—C and C—O bond lengths compare well with distances found in other compounds, $Fe_2(CO)_6(COH)_2(CH_3C_2CH_3)$ (Hock & Mills, 1958), $C_8H_8Fe(CO)_3$ (Dickens & Lipscomb, 1961), $Fe_2(CO)_6(C_6H_5C_2H)_3$ (King, 1962) and $[C_2N_5SFe(CO)_3]_2$ (Dahl & Wei, 1963).

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* Lists of structure-factor amplitudes, anisotropic displacement parameters for the non-H atoms, H-atom parameters, C—H bond lengths and calculations of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54070 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1991). C**47**, 2201–2203

Structure of (Cyano)(2,3,7,8,12,13,17,18-octaethylporphinato)(pyridine)iron(III) Chloroform Solvate

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(*Received* 23 *August* 1990; *accepted* 11 *March* 1991)

Abstract. $[Fe(C_{36}H_{44}N_4)(CN)(C_5H_5N)].CHCl_3$, $M_r = 813{\cdot}12$, monoclinic, $P2_1/n$, $a = 10{\cdot}345$ (2), $b = 14{\cdot}748$ (2), $c = 27{\cdot}302$ (3) Å, $\beta = 92{\cdot}12$ (3)°, $V = 4162{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}30$, $D_m = 1{\cdot}29$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 5{\cdot}92$ cm^{-1}, $F(000) = 1708$, $T = 293$ K, $R = 0{\cdot}046$ for 5193 unique observed $[F > 3\sigma(F)]$ reflections. The iron(III) atom is six-coordinate with an average Fe—N(p) distance of 1·980 (4) Å and axial Fe—C and Fe—N distances of 1·934 (4) and 2·087 (3) Å, respectively.

Experimental. The title compound was synthesized as part of a resonance Raman study of iron–ligand vibrations (Uno, Hatano, Nishimura & Arata, 1988). D_m measured by flotation. A purple, air-stable crystal of [Fe(oep)(CN)(py)] recrystallized from $CHCl_3$ with approximate dimensions of 0·31 × 0·27 × 0·27 mm was mounted on the end of a glass fiber. All measurements were performed with graphite-monochromated Mo $K\alpha$ radiation on an Enraf–Nonius CAD-4 diffractometer. Intensity data were measured by θ–2θ scans at a scan rate of 3°θ min^{-1}. Data were collected to a maximum 2θ of 54·9°, and standard θ–2θ CAD-4 moving-crystal moving-counter background measurements were used. 25 reflections used for measuring lattice parameters

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