In the nitrate ion the longest N—O distance involves the O atom which is not hydrogen bonded (Table 2). Angles around the N atom show significant distortion from D_{3h} symmetry, due to hydrogen bonding.

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Acta Cryst. (1987). C**43**, 1533–1535

Structure of $[Fe_2(CO)_6\{\mu\text{-}C(SiMe_3)C(SiMe_3)CHCHCH(OEt)\}]$. An η^1, η^3-Allyl Bridged Diiron Complex

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Abstract. μ-1:2κ^2C^1-[5-Ethoxy-1,2-bis(trimethylsilyl)penta-2,4-dienyl]-1$\kappa^2C^{2,3}$;2$\kappa^2C^{4,5}$-bis(tricarbonyliron)(*Fe–Fe*), $[Fe_2(CO)_6(C_{13}H_{26}OSi_2)]$, $M_r = 534{\cdot}28$, triclinic, $P\bar{1}$, $a = 14{\cdot}594\,(4)$, $b = 10{\cdot}049\,(3)$, $c = 8{\cdot}776\,(3)$ Å, $\alpha = 79{\cdot}14\,(2)$, $\beta = 97{\cdot}61\,(2)$, $\gamma = 94{\cdot}84\,(2)^\circ$, $V = 1250\,(1)$ Å^3, $D_x = 1{\cdot}419$ g cm^{-3}, $Z = 2$, $F(000) = 552$, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo }K\alpha) = 12{\cdot}86$ cm^{-1}, $T = 288$ K. The structure was solved by direct methods and refined by full-matrix least squares to $R = 0{\cdot}057$ for 3720 reflections. It consists of $Fe_2(CO)_6$ units linked by an organic bridge which results formally from the coupling of ethoxymethylidene, ethanediylidene and bis(trimethylsilyl)ethanediylidene radicals. The Fe—Fe length [2·625 (1) Å] is longer than those values usually observed in $Fe_2(CO)_6$ centres.

Introduction. The synthesis of the dinuclear anionic complexes $[Fe_2(CO)_6(\mu\text{-}CO)\{\mu\text{-}C(R)C(R)H\}^-]$ has opened up a new area in the extensive field of study of the reactivity of organic molecules at dinuclear centres. In this paper we describe the crystal and molecular structure of the compound resulting from the interaction of an $Fe_2(CO)_6(\mu\text{-}COEt)(\mu\text{-}CHCH_2)$ complex with bis(trimethylsilyl)acetylene.

Experimental. A poor-quality crystal ($0{\cdot}1 \times 0{\cdot}1 \times 0{\cdot}2$ mm), Philips PW 1100 diffractometer. Cell parameters from 25 reflections ($4 \leq \theta \leq 12^\circ$) refined by least squares; graphite-monochromatized Mo $K\alpha$ radiation, ω-scan technique, scan width 1°, scan speed 0·03° s^{-1}. Three reflections every 2 h, no significant intensity decay observed. 3842 independent reflections ($2 \leq$

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Table 1. *Final atomic coordinates* (× 10^4, *for* Fe × 10^5) *and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{8}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Fe(1)	67114 (5)	32740 (7)	49096 (9)	3·16 (4)
Fe(2)	82117 (4)	44672 (7)	37871 (9)	3·37 (4)
Si(1)	7898 (1)	1443 (2)	2537 (2)	4·21 (7)
C(31)	7185 (6)	−198 (7)	2620 (11)	6·35 (39)
C(32)	8987 (4)	960 (7)	3844 (10)	5·70 (34)
C(33)	8194 (6)	2061 (8)	473 (10)	6·22 (40)
C(1)	7345 (3)	2912 (5)	3080 (7)	2·99 (19)
C(2)	6438 (3)	3329 (4)	2542 (6)	2·72 (19)
C(3)	6312 (3)	4652 (5)	2888 (7)	3·22 (21)
Si(2)	5371 (1)	2400 (2)	1625 (2)	3·44 (6)
C(41)	4358 (4)	3458 (7)	1375 (10)	5·77 (32)
C(42)	5013 (5)	744 (6)	2887 (9)	5·28 (32)
C(43)	5589 (5)	2185 (9)	−392 (8)	5·62 (37)
C(4)	7062 (3)	5698 (5)	2710 (7)	3·28 (21)
C(5)	7620 (4)	5825 (5)	1459 (8)	3·85 (23)
C(6)	8131 (3)	7010 (4)	1095 (6)	4·48 (19)
C(7)	8636 (7)	7139 (8)	−247 (12)	7·36 (49)
C(8)	8968 (10)	8435 (11)	−769 (19)	13·04 (96)
C(11)	5531 (4)	2985 (6)	5394 (7)	4·03 (26)
C(11)	4802 (3)	2881 (6)	5680 (6)	5·81 (25)
C(12)	7045 (4)	4386 (7)	6234 (8)	4·63 (31)
C(12)	7237 (4)	5074 (7)	7156 (7)	6·99 (32)
C(13)	7068 (4)	1685 (6)	6077 (7)	4·18 (26)
C(13)	7272 (4)	670 (5)	6836 (7)	6·73 (28)
C(21)	9125 (4)	4415 (6)	2625 (8)	4·45 (27)
C(21)	9756 (3)	4392 (6)	1964 (7)	6·61 (29)
C(22)	8648 (4)	5966 (6)	4600 (8)	4·58 (28)
C(22)	8889 (4)	6911 (6)	5092 (8)	7·84 (34)
C(23)	8775 (4)	3438 (7)	5489 (9)	4·92 (30)
C(23)	9161 (4)	2845 (6)	6595 (7)	6·43 (28)

$\theta \leq 30°$), with *hkl*: −20 to 20; −14 to 14; and 0 to 10, 3720 with $I \geq 2{\cdot}5\sigma(I)$. Lp corrections, absorption ignored. The Fe and Si atoms were located with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), remaining non-hydrogen atoms with *DIRDIF* (Beurskens *et al.*, 1981). Full-matrix least-squares refinement (*SHELX*76, Sheldrick, 1976). $\sum w\big||F_o| - |F_c|\big|^2$ minimized, $w = \sigma^{-2}(F_o)$. f, f' and f'' from *International Tables for X-ray Crystallography* (1974). 23 H were computed and refined with an overall isotropic temperature factor; the remaining atoms were refined anisotropically. Final R factor 0·057 ($wR = 0{\cdot}062$) for all observed reflections. Max. shift/e.s.d. = 0·26 in x of O(12). Max. and min. $\Delta\rho$ 0·3 and −0·4 e Å^{-3}, respectively. IBM 3083 computer. Final atomic coordinates are listed in Table 1.*

Discussion. The molecular structure of the title compound with the atom numbering is shown in Fig. 1. Selected bond lengths and angles are listed in Table 2. The structure consists of $Fe_2(CO)_6$ units linked by an organic bridge, which results formally from the coupling of ethoxymethylidene, acetylene and bis(trimethylsilyl)acetylene radicals.

The organic group is η^2-bonded to Fe(2) by C(4)—C(5), σ-bonded to Fe(2) by C(1) and η^2-bonded to Fe(1) by the C(1), C(2), C(3) allylic fragment. This bonding system is similar to those observed in

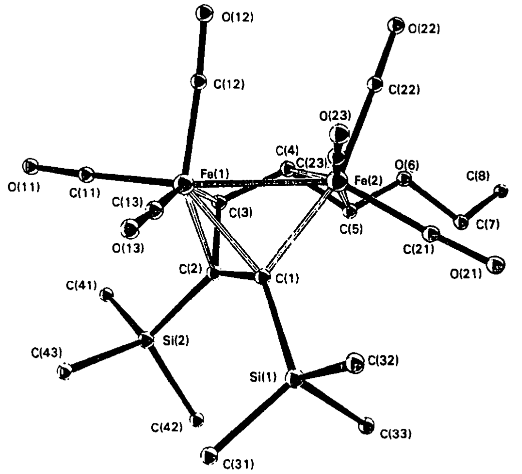

Fig. 1. View of the title complex with atomic numbering.

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

Fe(2)—Fe(1)	2·625 (1)	C(23)—Fe(2)	1·792 (7)
C(1)—Fe(1)	2·059 (5)	C(1)—Si(1)	1·904 (5)
C(2)—Fe(1)	2·054 (5)	C(2)—Si(2)	1·911 (5)
C(3)—Fe(1)	2·083 (5)	C(1)—C(2)	1·415 (6)
C(11)—Fe(1)	1·815 (6)	C(2)—C(3)	1·450 (6)
C(12)—Fe(1)	1·756 (6)	C(3)—C(4)	1·455 (7)
C(13)—Fe(1)	1·803 (6)	C(4)—C(5)	1·433 (8)
C(1)—Fe(2)	2·062 (4)	C(5)—O(6)	1·358 (6)
C(4)—Fe(2)	2·174 (5)	O(6)—C(7)	1·451 (9)
C(5)—Fe(2)	2·337 (6)	C(7)—C(8)	1·367 (13)
C(21)—Fe(2)	1·792 (7)	av.(H_3C—Si)	1·88 (1)
C(22)—Fe(2)	1·818 (6)		

C(1)—Fe(1)—Fe(2)	50·5 (1)	C(1)—Fe(2)—Fe(1)	50·4 (2)
C(2)—Fe(1)—Fe(2)	75·6 (1)	C(4)—Fe(2)—Fe(1)	72·6 (1)
C(2)—Fe(1)—C(1)	40·2 (2)	C(4)—Fe(2)—C(1)	83·3 (2)
C(3)—Fe(1)—Fe(2)	71·7 (1)	C(5)—Fe(2)—Fe(1)	102·8 (1)
C(3)—Fe(1)—C(1)	69·9 (2)	C(5)—Fe(2)—C(1)	86·6 (2)
C(3)—Fe(1)—C(2)	41·0 (2)	C(5)—Fe(2)—C(4)	36·8 (2)
C(11)—Fe(1)—Fe(2)	161·2 (2)	C(21)—Fe(2)—Fe(1)	147·9 (2)
C(11)—Fe(1)—C(1)	133·3 (2)	C(21)—Fe(2)—C(1)	98·6 (2)
C(11)—Fe(1)—C(2)	98·5 (2)	C(21)—Fe(2)—C(4)	117·0 (2)
C(11)—Fe(1)—C(3)	92·0 (2)	C(21)—Fe(2)—C(5)	80·3 (2)
C(12)—Fe(1)—Fe(2)	80·2 (2)	C(22)—Fe(2)—Fe(1)	113·6 (2)
C(12)—Fe(1)—C(1)	130·6 (2)	C(22)—Fe(2)—C(1)	162·3 (2)
C(12)—Fe(1)—C(2)	139·5 (2)	C(22)—Fe(2)—C(4)	84·1 (2)
C(12)—Fe(1)—C(3)	100·5 (2)	C(22)—Fe(2)—C(5)	90·8 (2)
C(12)—Fe(1)—C(11)	94·1 (3)	C(22)—Fe(2)—C(21)	98·1 (3)
C(13)—Fe(1)—Fe(2)	105·2 (2)	C(23)—Fe(2)—Fe(1)	82·7 (2)
C(13)—Fe(1)—C(1)	90·7 (2)	C(23)—Fe(2)—C(1)	95·5 (2)
C(13)—Fe(1)—C(2)	116·4 (2)	C(23)—Fe(2)—C(4)	148·8 (2)
C(13)—Fe(1)—C(3)	157·4 (2)	C(23)—Fe(2)—C(5)	174·1 (2)
C(13)—Fe(1)—C(11)	93·4 (3)	C(23)—Fe(2)—C(21)	94·0 (3)
C(13)—Fe(1)—C(12)	101·0 (3)	C(23)—Fe(2)—C(22)	88·9 (3)

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and additional bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43938 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

μ-1:2κ^2C^1-[5-ethoxy-1,2-bis(trifluoromethyl)penta-2,4-dienyl]-1$\kappa^2C^{2,3}$;2$\kappa^2C^{4,5}$-bis(tricarbonyliron)(*Fe–Fe*) (Ros, Commenges, Mathieu, Solans & Font-Altaba, 1985). The substitution of trifluoromethyl by the trimethylsilyl group produces slight differences between the two molecular structures. Thus, the Fe—Fe bond length [2·625 (1) Å] is intermeduate between those observed in the trifluoromethyl complex [2·617 (3) Å] and those obtained for the product resulting from the reaction of 2,7-dimethoxyepine with $Fe(CO)_5$ [2·642 (1) Å] (Aumann, Averbeck & Krüger, 1975), but this distance is quite long for an iron–iron single-bond distance (Krüger, Barnett & Brauer, 1978). The Fe(2) atom is asymmetrically bonded to the olefin part of the organic ligand with Fe(2)—C(5) [2·337 (6) Å] 0·163 Å longer than the Fe(2)—C(4) bond [2·174 (5) Å] [2·36 (1) and 2·21 (1) Å, respectively, in the trifluoromethyl complex].

C(1) is equidistant from Fe(1) and Fe(2) [average value 2·061 (1) Å] and Fe(1) is equidistant from C(1), C(2) and C(3) [average value 2·065 (13) Å]. The C(1)—O(6) chain gives evidence of extensive electronic delocalization with C—C lengths ranging from 1·415 (6) to 1·455 (7) Å and the C(5)—O(6) length shorter than the O(6)—C(7) length.

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Structure of Bis[*tert*-butyl (2-Lithiophenyl) Sulfide] [*N,N,N',N'*-Tetramethylethylenediamine]

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Abstract. *catena*-Poly{[lithium-bis-μ-(*tert*-butylthiophenyl-*S*,μ-C)-lithium]-μ-(*N,N,N',N'*-tetramethylethylenediamine-*N,N'*)}, $[\{Li(C_{10}H_{13}S)\}_2(C_6H_{16}N_2)]$, $M_r = 460{\cdot}64$, triclinic, $P\bar{1}$, $a = 7{\cdot}832\ (3)$, $b = 9{\cdot}419\ (1)$, $c = 10{\cdot}771\ (1)$ Å, $\alpha = 85{\cdot}93\ (1)$, $\beta = 72{\cdot}90\ (2)$, $\gamma = 69{\cdot}64\ (3)^\circ$, $V = 711{\cdot}6\ (3)$ Å^3, $Z = 1$, $D_x = 1{\cdot}075$ g cm^{-3} $\lambda(Cu\ K\alpha) = 1{\cdot}54184$ Å, $\mu = 17{\cdot}23$ cm^{-1}, $F(000) = 250$, $T = 295$ K, $R = 0{\cdot}055$ for 2025 observed reflections. The structure consists of centrosymmetric *tert*-butyl (2-lithiophenyl) sulfide dimers [around ($\frac{1}{2}$00)] which are linked together by bridging centrosymmetric *N,N,N',N'*-tetramethylethylenediamine (TMEDA) molecules [around (000)] to form one-dimensional infinite chains. The Li atom has a distorted tetrahedral coordination sphere consisting of two C atoms of symmetry-related phenyl rings, one N atom and one S atom. The phenyl ring is distorted in a way which is common for all aryl groups bonded to electropositive elements.

Introduction. Whereas the activating and *ortho*-directing effect of several hetero substituents (*e.g.* —O*R*,

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