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REACTION OF IRON PENTACARBONYL WITH *N,N*-DIETHYL-*S*-ETHYLCARBAMATE; CRYSTAL STRUCTURE OF $[\{Fe_2(CO)_6\}_3(\mu_4\text{-}S)_2(\mu\text{-}CNEt_2)_2]$*3(Fe–Fe)*, A COMPOUND CONTAINING THREE BUTTERFLY UNITS

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Summary

Reaction of iron pentacarbonyl with *N,N*-diethyl-*S*-ethylcarbamate {S=C-(NEt$_2$)SEt} gave $[\{Fe_2(CO)_6\}\{Fe_3(CO)_9(\mu_4\text{-}S)(\mu\text{-}CNEt_2)(\mu\text{-}SEt)\}]$*(Fe–Fe)3-(Fe–Fe)* (I) and $[\{Fe_2(CO)_6\}_3(\mu_4\text{-}S)_2(\mu\text{-}CNEt_2)_2$*3(Fe–Fe)* (II). The structure of complex II has been determined by single crystal X-ray crystallography. It crystallizes from methylene chloride as $C_{26}H_{20}O_{18}N_2S_2Fe_6 \cdot \frac{1}{2}CH_2Cl_2$ in the monoclinic space group *C2/c* with *a* 46.754(2), *b* 9.268(1), *c* 19.628(2) Å, *β* 100.7(1)°, $Z = 8$, *V* 8358.6 Å^3, D_c 1.77 g cm^{-3}, D_m 1.77 g cm^{-3}. The structure was solved by direct methods and refined to $R = 0.0819$ ($R_w = 0.0469$) for 3017 reflections. It consists of three metal–metal bonded $Fe_2(CO)_6$ units linearly linked in butterfly configuration by two bridging sulphur atoms, and terminated on both sides by bridging diethyliminiocarbene (CNEt$_2$) ligands.

Introduction

Although a number of reactions between iron carbonyl complexes and hetero-atom-containing thioketone derivatives have been described [1], reactions between $[Fe(CO)_5]$ and dithiocarbamates, $S{=}C(NR^1_2)SR^2$, are unknown. Dean [2] has remarked on the remarkable mobility of the sulphur atom in $S{=}C(Cl)NMe_2$ during reactions with iron carbonyl complexes. We also established this mobility in conversions with chromium carbonyl compounds [3]. The thione $S{=}C(NMe_2)_2$ forms product A with $[Fe_2(CO)_9]$ [4].

When one of the NMe$_2$ groups is replaced by SEt similar products can be expected from the interaction of $S{=}C(NEt_2)SEt$ with $[Fe(CO)_5]$. Neither of the two possible structural isomers could be detected.

The work described here included the preparation and X-ray diffraction study of a compound containing three $Fe_2(CO)_6$ units linked and bridged by two sulphur

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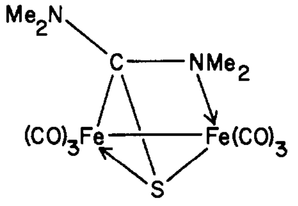


A

atoms and with two of the units also internally bridged by a formally cationic carbene ligand ($CNEt_2$). The results show that sulphur atoms as well as diethylaminocarbene and ethylsulphide groups are accessible from $S{=}C(NEt_2)SEt$ for bridging iron atoms in carbonyl complexes.

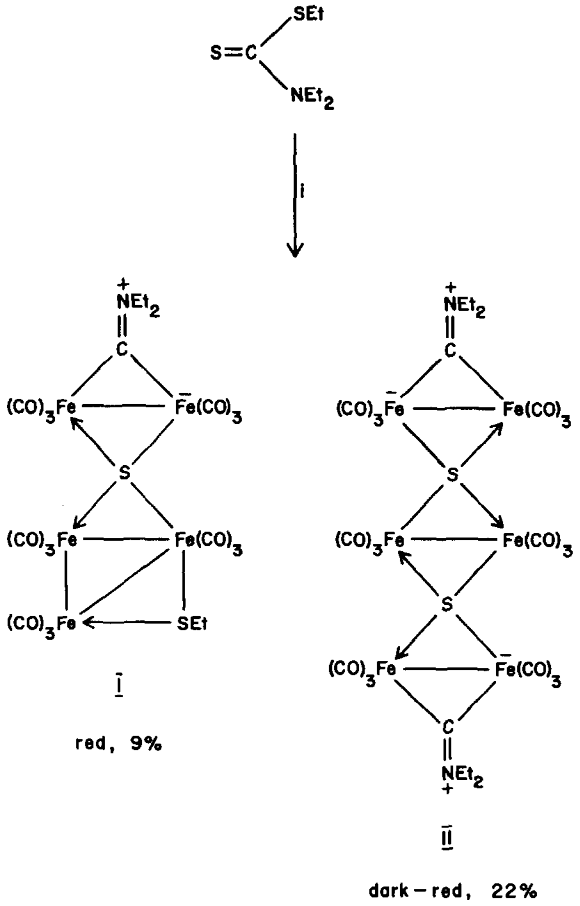
Results and discussion

Photochemical reaction of $S{=}C(NEt_2)SEt$ with three molar equivalents of $[Fe(CO)_5]$ in tetrahydrofuran (THF) gave two main products (I and II Scheme 1), which were separated by column chromatography and recrystallized from hexane/CH_2Cl_2. The structure of I was based on elemental analysis and spectroscopic data, whereas that of II was finally determined by a single crystal X-ray diffraction study.

The highest peak in the mass spectrum (70 eV) for I was found at m/z 737 and was assigned to the $[M-5CO]^+$ fragment ion. This decomposition was followed by the subsequent loss of ten more CO ligands. The signals corresponding to $Fe_3S_2{}^+$, Fe_3S^+ and Fe_2S^+ were clearly identifiable. We previously identified the $Fe_3S_2{}^+$ species in the mass spectrum of the trinuclear iron cluster $[Fe_3(CO)_9\{\mu\text{-}\overline{CS(CH_2)_2S}\}(\mu_3\text{-}S)]$ containing two bridging sulphur atoms [5]. Other spectroscopic data for I are given in the Experimental section.

Compound I has eight medium to strong infrared absorption bands in the terminal carbonyl region between 1978 and 2080 cm^{-1}. The proton chemical shifts indicate three non-equivalent ethyl groups in the molecule, from which the presence of a carbon–nitrogen double bond may be inferred. Non-rigidity of the molecule was established by ^{13}C NMR measurements at various temperatures and the results are listed in Table 1. The two resonances for SCH_2 at 303 K increase to four at 203 K indicating a slowing down of a fluxional process and also the presence of four diastereomers in solution. The carbonyl ligands also undergo site exchange; three carbonyl carbon resonances are exhibited at 303 K and the number increases to eight at 253 K and to ten at 203 K. At still lower temperatures crystallization occurs. There are two non-equivalent positions for the NEt_2 groups. The resonances at δ 309.2 (253 K) or 311.0 ppm (303 K) are assigned to the bridging carbene carbon atom. It is comparable with a value of 257.6 ppm at ambient temperature in the complex $[Fe_2(CO)_6(\mu\text{-}CNEt_2)_2]$ [6].

Compound II decomposed in the tube during ^{13}C NMR data collection and no molecular ion was obtained or carbonyl losses observed by mass spectroscopy. The infrared ^1H NMR data given in the Experimental section were not sufficient to establish the structure, and a crystal structure determination was undertaken. The



SCHEME 1. Reagent: (i) $[Fe(CO)_5]$, $h\nu$.

TABLE 1

$^{13}C\{^1H\}$ NMR DATA[a] FOR $[\{Fe_2(CO)_6\}\{Fe_3(CO)_9\}(\mu_4\text{-}S)(\mu\text{-}CNEt_2)(\mu\text{-}SEt)]$*(Fe–Fe)3(Fe–Fe)* (I)

Temperature (K)	CN	CO		NCH_2	SCH_2	NCH_2CH_3	SCH_2CH_3
303	311.0	209.4	209.6	58.2	31.0	17.6	13.6
		210.7		58.7	34.1		13.8
253	309.2	207.2	209.0	57.7	30.9	17.8	13.4
		210.1	210.5	58.4	33.8		13.6
		210.6	211.4				
		211.6	212.1				
203	308.6	208.7	209.6	57.1	30.9	17.6	13.2
		209.7	209.8	58.0	31.8		
		210.0	210.2		33.9		
		210.3	210.4		34.9		
		211.3	211.9				

[a] Measured in CD_2Cl_2 relative to internal $SiMe_4$ in ppm.

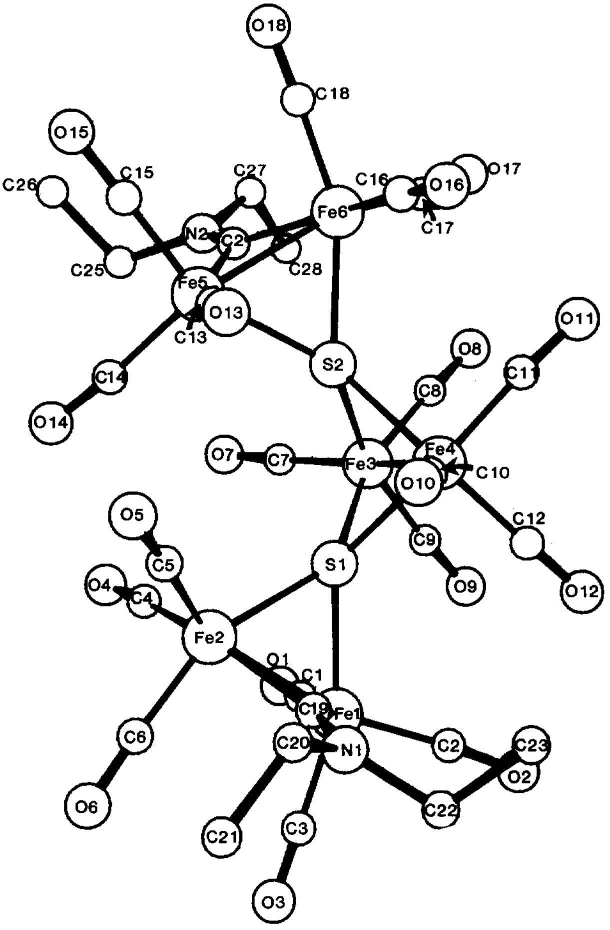


Fig. 1. View of the molecular structure of compound II with the atomic numbering scheme. H atoms are omitted for clarity.

structure found is shown in Fig. 1 and 2. It consists of three metal–metal bonded $Fe_2(CO)_6$ units, two sulphur atoms and two diethylimoniocarbene ligands. The two sulphur atoms S(1) and S(2) are at the connecting wing-tip positions of the butterfly units $Fe_2(CO)_6(CNEt_2)S(1)$, $Fe_2(CO)_6S(1)S(2)$ and $Fe_2(CO)_6(CNEt_2)S(2)$. The molecule contains a non-crystallographic C_2-axis passing through the midpoint of the bond between Fe(3) and Fe(4) and a point midway between S(1) and S(2). One half-mole equivalent of methylene chloride solvent co-crystallized, as indicated independently by a density measurement and an elemental analysis for Cl. Since the coordinated carbon–nitrogen distances of 1.30(2) and 1.29(2) Å correspond to double bonds and imply positive charges on the nitrogen atoms, two negative charges have to be distributed over the central $Fe_6(CO)_{18}S_2$ segment of the molecule. Similar bonding situations have been observed in simpler compounds: a part of the compound. $[\{Fe_2(CO)_6\}_2(\mu_2\text{-}S)(\mu\text{-}CNMe_2)(\mu\text{-}SCNMe_2)]$*2(Fe-Fe)* (**A**) [7] is similar to II, and the bridging carbene ligand is also present in the previously mentioned complex $[Fe_2(CO)_6(\mu\text{-}CNEt_2)_2]$ (**B**) [6].

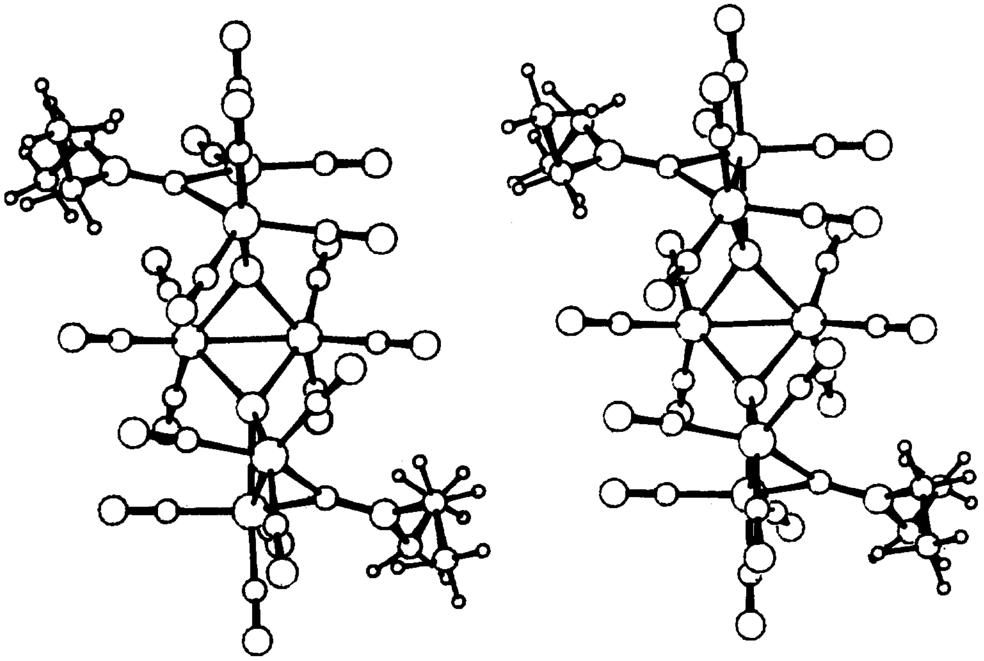


Fig. 2. A stereoview of II.

Selected bond lengths and angles for II are given in Tables 2 and 3. The Fe–Fe distances are nearly equal, with the central metal–metal bond (2.544(3) Å) slightly longer than those which are also carbene-bridged (both 2.479(3) Å). Dean and VanDerveer [7] reported a 2.512(2) Å bond distance between two iron atoms which are bridged by sulphur and the carbene group $CNMe_2$. The Fe–S distances in II range from 2.251(4) to 2.275(4) Å (av. 2.261 Å) which is shorter than the average value of 2.299 Å in **A** [4]. Both Fe_2CNEt units, ignoring the hydrogen atoms, are planar. Deviations from the best planes are listed in Table 4. Our values for the C(coordinated)–N bond lengths (1.30(2) and 1.29(2) Å) fall between the values reported for the same bonds in **A** (1.325(9) Å) and **B** (av. 1.282 Å). The Fe–C(carbene) distances average 1.90 Å, which is similar to the average value in **B**. Metal–carbonyl distances vary between 1.73(1) and 1.82(2) Å.

Experimental

Reactions were conducted under dry nitrogen using Schlenk techniques [8]. Solvents were dried and saturated with nitrogen before use. For column chromatography Merck Kieselgel 60 (particle size 0.063–0.200 nm) was used as stationary phase. A Philips 93136E mercury lamp with water-cooled jacked was employed for photochemical reactions. Melting points (Kofler hot-stage apparatus) are uncorrected. Infrared spectra were obtained with a Perkin–Elmer 297 spectrometer and ^1H NMR spectra with a Bruker WP-80 or Varian T60. A Bruker 500 was used for obtaining ^{13}C NMR data and a Hitachi–Perkin–Elmer RMU-6H for recording mass spectra. Commercial $[Fe(CO)_5]$ and $S{=}C(NEt_2)SEt$ (Aldrich) were used without further purification.

TABLE 2

BOND LENGTHS (Å)

Fe(1)–Fe(2)	2.479(3)	Fe(1)–S(1)	2.256(4)
Fe(1)–C(1)	1.81(2)	Fe(1)–C(2)	1.78(2)
Fe(1)–C(3)	1.76(2)	Fe(1)–N(1)	2.99(2)
Fe(1)–C(19)	1.88(2)	Fe(2)–S(1)	2.275(4)
Fe(2)–C(4)	1.82(2)	Fe(2)–C(5)	1.78(2)
Fe(2)–C(6)	1.76(2)	Fe(2)–N(1)	3.00(2)
Fe(2)–C(19)	1.92(2)	Fe(3)–Fe(4)	2.544(3)
Fe(3)–S(1)	2.255(4)	Fe(3)–S(2)	2.251(4)
Fe(3)–C(7)	1.76(2)	Fe(3)–C(8)	1.78(2)
Fe(3)–C(9)	1.75(2)	Fe(4)–S(1)	2.255(4)
Fe(4)–S(2)	2.258(4)	Fe(4)–C(10)	1.77(2)
Fe(4)–C(11)	1.78(2)	Fe(4)–C(12)	1.77(2)
Fe(5)–Fe(6)	2.479(3)	Fe(5)–S(2)	2.275(4)
Fe(5)–C(13)	1.81(2)	Fe(5)–C(14)	1.81(2)
Fe(5)–C(15)	1.73(2)	Fe(5)–N(2)	3.01(2)
Fe(5)–C(24)	1.91(2)	Fe(6)–S(2)	2.264(3)
Fe(6)–C(16)	1.82(2)	Fe(6)–C(17)	1.80(2)
Fe(6)–C(18)	1.76(2)	Fe(6)–N(2)	3.01(2)
Fe(6)–C(24)	1.90(2)	S(2)–C(24)	2.635(13)
O(1)–C(1)	1.16(2)	O(2)–C(2)	1.16(2)
O(3)–C(3)	1.12(2)	O(4)–C(4)	1.13(2)
O(5)–C(5)	1.14(2)	O(6)–C(6)	1.15(2)
O(7)–C(7)	1.17(2)	O(8)–C(8)	1.14(2)
O(9)–C(9)	1.16(2)	O(10)–C(10)	1.15(2)
O(11)–C(11)	1.14(2)	O(12)–C(12)	1.14(2)
O(13)–C(13)	1.13(2)	O(14)–C(14)	1.12(2)
O(15)–C(15)	1.18(2)	O(16)–C(16)	1.14(2)
O(17)–C(17)	1.12(2)	O(18)–C(18)	1.15(2)
C(8)–C(9)	2.42(2)	N(1)–C(19)	1.30(2)
N(1)–C(20)	1.47(2)	N(1)–C(22)	1.51(2)
C(20)–C(21)	1.51(2)	C(22)–C(23)	1.51(2)
N(2)–C(24)	1.29(2)	N(2)–C(25)	1.47(2)
N(2)–C(27)	1.51(2)	C(25)–C(26)	1.53(2)
C(27)–C(28)	1.49(2)	Cl–C(29)	1.68(2)

Preparation of the complexes I and II

A mixture of $[Fe(CO)_5]$ (5.9 g, 4.0 ml, 30 mmol) and 1.88 g (10 mmol) $S{=}C(NEt_2)SEt$ was irradiated for 12 h at room temperature in 100 ml THF. The two main products observed on SiO_2 thin layer chromatographic test plates were separated by column chromatography (column: 2.8×35 cm; cooled to $-10\,°C$) using hexane/CH_2Cl_2 (1/6 to 1/1) as eluant mixture. The first (orange) fraction collected was stripped of solvent under reduced pressure and recrystallized from ether/pentane ($-30\,°C$) to afford 0.45 g (9% based on ligand) of red needles of compound I. M.p. 129–131°C. MS (m/z, 70 eV): 737 $(M-5CO)^+$; consecutive loss of another 10 CO ligands; 232 ($Fe_3S_2^+$); 200 (Fe_3S^+) and 144 (Fe_2S^+). IR (hexane, CO-region): 2080m, 2038s, 2029s, 1955(sh), 1988s, 1973m and 1968m cm^{-1}. ^1H NMR spectrum ($CDCl_3$, δ-values in ppm relative to int. $SiMe_4$): 4.2–3.7 (m, 4H, NCH_2); 2.49 (q, 2H, SCH_2); 1.39 (t, 9H, CH_3). Analysis. Found: C, 30.38; H, 1.74; N, 1.76; S. 7.48. $Fe_5C_{22}H_{15}NO_{15}S_2$ calcd.: C, 30.14; H, 1.72; N, 1.60; S, 7.31%.

TABLE 3

BOND ANGLES (DEGREES)

Angles around Fe(1)

	Fe(2)	S(1)	C(1)	C(2)	C(3)
S(1)	57.2(1)				
C(1)	101.1(5)	91.5(4)			
C(2)	152.9(5)	107.6(5)	101.5(7)		
C(3)	100.5(5)	157.7(5)	93.1(7)	92.8(6)	
C(19)	49.8(4)	77.7(4)	150.4(5)	87.7(7)	

Angles around Fe(2)

	Fe(1)	S(1)	C(4)	C(5)	C(6)
S(1)	56.5(1)				
C(4)	101.8(5)	91.5(5)			
C(5)	150.1(5)	106.2(5)	102.8(7)		
C(6)	98.9(5)	155.3(5)	92.2(7)	96.8(7)	
C(19)	48.6(4)	76.5(4)	149.9(6)	107.0(6)	88.3(6)

Angles around Fe(3)

	Fe(4)	S(1)	S(2)	C(7)	C(8)
S(1)	55.6(1)				
S(2)	55.8(1)	82.5(1)			
C(7)	149.7(5)	105.5(5)	101.9(5)		
C(8)	102.6(5)	156.1(5)	92.9(5)	98.3(7)	
C(9)	102.0(6)	88.8(5)	157.1(6)	100.9(8)	86.5(7)

Angles around Fe(4)

	Fe(3)	S(1)	S(2)	C(10)	C(11)
S(1)	55.7(1)				
S(2)	55.5(1)	82.4(1)			
C(10)	148.7(5)	99.7(5)	106.8(5)		
C(11)	104.3(5)	159.8(5)	88.1(4)	100.1(7)	
C(12)	99.5(6)	90.5(5)	153.4(6)	99.6(8)	90.1(7)

Angles around Fe(5)

	Fe(6)	S(2)	C(13)	C(14)	C(15)
S(2)	56.7(1)				
C(13)	100.9(5)	92.2(5)			
C(14)	152.3(5)	106.9(5)	102.0(6)		
C(15)	98.3(5)	155.0(5)	91.4(7)	96.5(7)	
C(24)	49.1(4)	77.4(4)	149.1(6)	108.9(6)	86.9(6)

Angles around Fe(6)

	Fe(5)	S(2)	C(16)	C(17)	C(18)
S(2)	57.1(1)				
C(16)	101.1(5)	92.1(5)			
C(17)	152.2(5)	105.7(5)	101.2(8)		
C(18)	100.6(5)	157.8(5)	92.6(7)	94.7(7)	
C(24)	49.7(4)	78.0(4)	149.9(7)	108.8(7)	87.1(6)

Angles around S(1)

	Fe(1)	Fe(2)	Fe(3)
Fe(2)	66.3(1)		
Fe(3)	128.0(2)	141.5(2)	
Fe(4)	132.3(2)	132.8(2)	68.7(1)

continued

TABLE 3 (continued)

Angles around S(2)

	Fe(3)	Fe(4)	Fe(5)
Fe(4)	68.7(1)		
Fe(5)	133.6(2)	141.1(2)	
Fe(6)	130.1(2)	129.5(2)	66.2(1)

Other angles

Fe(1)–C(19)–Fe(2)	81.6(6)	Fe(5)–C(24)–Fe(5)	81.2(6)
Fe(1)–C(19)–N(1)	140.2(11)	Fe(5)–C(24)–N(2)	138.7(10)
Fe(2)–C(19)–N(1)	137.6(11)	Fe(6)–C(24)–N(2)	140.1(10)
C(20)–N(1)–C(19)	125.1(12)	C(25)–N(2)–C(24)	124.5(12)
C(20)–N(1)–C(22)	113.4(12)	C(25)–N(2)–C(27)	114.8(12)
C(22)–N(1)–C(19)	121.4(13)	C(27)–N(2)–C(24)	120.7(12)
N(1)–C(22)–C(23)	111.2(12)	N(2)–C(25)–C(26)	111.9(12)
N(1)–C(20)–C(21)	110.8(14)	N(2)–C(27)–C(28)	113.3(12)

From the second (red) fraction compound II was obtained as dark-red needles (1.18 g, 22%) after recrystallization. M.p. 165–165 °C. IR (hexane, CO-region): 2059s, 2049s, 2037s, 2030(sh), 1995(sh), 1983s and 1966s cm^{-1}. ^{1}H NMR spectrum ($CDCl_3$, δ-values in ppm relative to int. $SiMe_4$): 3.99 (q, 2H, NCH_2); 1.44 (t, 3H, CH_3). Analysis. Found: C, 30.43; H, 2.06; Cl, 3.04; N, 2.36; S, 5.74. $Fe_6C_{28.5}H_{21}ClO_{18}N_2S_2$ calcd.: C, 30.72; N, 1.90; Cl, 3.18; N, 2.51; S, 5.75%.

TABLE 4

INTRAMOLECULAR PLANES FOR COMPOUND II

Atom	deviation (Å)	Atom	deviation (Å)
(a) The plane Fe(3)–Fe(4)–C(7)–C(10)			
Equation: $-0.922x+0.024y-0.038z+18.14=0$			
^{a} Fe(3)	0.04	S(1)	1.44
^{a} Fe(4)	−0.05	S(1)	−1.53
^{a} C(7)	−0.02	N(1)	3.65
^{a} C(10)	0.02	N(2)	−3.67
Fe(1)	3.70	C(19)	3.31
Fe(6)	−3.78	C(24)	−3.42
Fe(2)	2.34	C(11)	−1.33
Fe(5)	−2.59	C(12)	1.18
(b) The plane Fe(1)–Fe(2)–N(1)–C(19)–C(20)–C(22)			
Equation: $0.815x-0.532y-0.229z+13.49=0$			
^{a} Fe(1)	0.05	^{a} C(20)	0.09
^{a} Fe(2)	−0.06	^{a} C(22)	−0.03
^{a} N(1)	0	C(21)	1.49
^{a} C(19)	−0.06	C(23)	−1.42
(c) The plane Fe(5)–Fe(6)–N(2)–C(24)–C(25)–C(27)			
Equation: $-0.888x+0.450y-0.093z+16.93=0$			
^{a} Fe(5)	0.02	^{a} C(25)	−0.02
^{a} Fe(6)	−0.02	^{a} C(27)	0
^{a} N(2)	0	C(26)	−1.44
^{a} C(24)	0.03	C(28)	1.33

^{a} Atoms used in calculation of planes.

Crystal structure determination of $[\{Fe_2(CO)_6\}_3(\mu_4\text{-}S)_2(\mu\text{-}CNEt_2)_2]\cdot\frac{1}{2}CH_2Cl_2$

Details are given in Table 5. A disordered methylene chloride molecule of crystallization is present in the crystal and situated across the centre of symmetry at ($\frac{1}{4}$, $\frac{1}{4}$, 0), such that the two centrosymmetrically related chlorine atoms bond to two centre-related half-weight methylene groups. It was refined as a rigid group of isotropic atoms with ideal geometry, tied to the centre of symmetry. All other non-hydrogen atoms were refined with anisotropic temperature factors. The ethyl group hydrogens were refined in calculated positions with a common isotropic temperature factor, and riding on the carbons to which they are atttached. The methyl groups were entered as rigid groups free to rotate. The final coordinates and equivalent temperature factors are listed in Table 6. The numbering system is shown

TABLE 5

DETAILS OF THE STRUCTURE DETERMINATION

Crystal data	
Formula	$C_{26}H_{20}O_{18}N_2S_2Fe_6\cdot\frac{1}{2}CH_2Cl_2$
Crystal habit	Dark red needles
Crystal size (mm)	0.20×0.15×0.12
Symmetry	Monoclinic, $C2/c$
Unit cell determination: least-squares fit to	25 reflections, θ(Mo) < 12°
Unit cell dimensions (Å)	a 46.754(9), b 9.268(2), c 19.628(4) β 100.7(2)°
Packing: V (Å^3), Z	8358.6, 8
D (g cm^{-3}), M, F(000)	1.77, 1114.17, 4436
Experimental data	
Diffractometer	Philips PW1100
Radiation	Mo-K_α
Monochromator	Graphite
Collection mode	ω scans, $\theta \leq 22°$ 0.94° θ scan width 0.038° s^{-1} scan speed
Reflections measured	4953
Observable reflections	3017 with $\lvert F\rvert > 2\sigma(F)$
Stability	3 reflections every 60 min, no variation
μ (cm^{-1}).	21.6
Absorption corrections	Empirical [9]
Solution and refinement	
Solution	SHELX76 [10],
Refinement	Least-squares, 2 blocks 398 parameters/block,
Number of variables	437
Reflections used	3017 observed with $F > 2\sigma$
Weighting scheme	$w = (\sigma(F))^{-2}$
Final Δ peaks	0.7 eÅ^{-3} near the methylene chloride
Final R, R_w	0.082, 0.047
Scattering factors	International Tables for X-Ray Crystallography [11]

TABLE 6

FRACTIONAL COORDINATES ($\times10^4$, $\times10^5$ FOR Fe) AND U_{eq} [a] ($\times10^4$, $\times10^3$ FOR Fe) FOR NON-H ATOMS

Atom	x	y	z	U_{eq}
Fe(1)	29934(4)	29450(20)	39225(10)	456(13)
Fe(2)	33030(4)	7924(19)	38669(10)	458(12)
Fe(3)	37087(4)	38568(19)	53701(10)	448(12)
Fe(4)	38426(4)	43793(19)	41960(10)	469(12)
Fe(5)	43258(4)	7512(20)	51362(9)	430(12)
Fe(6)	45783(4)	30478(21)	55117(10)	499(13)
S(1)	3475(1)	2920(4)	4358(2)	42(2)
S(2)	4102(1)	2939(4)	5013(2)	41(2)
O(1)	2861(2)	2613(9)	5336(4)	76(7)
O(2)	2802(2)	5919(10)	3593(6)	91(8)
O(3)	2437(2)	1838(11)	3241(5)	88(8)
O(4)	3297(2)	−523(10)	5230(5)	84(8)
O(5)	3789(2)	−587(10)	3376(5)	73(7)
O(6)	2852(2)	−1152(10)	3182(5)	87(8)
O(7)	3629(2)	1777(12)	6443(5)	98(8)
O(8)	4047(2)	6010(10)	6259(5)	83(7)
O(9)	3229(2)	5844(10)	5352(5)	85(8)
O(10)	3939(2)	3292(11)	2864(5)	95(9)
O(11)	4300(2)	6556(9)	4479(5)	71(7)
O(12)	3433(2)	6715(10)	3781(6)	100(9)
O(13)	4380(2)	692(11)	3667(4)	74(7)
O(14)	3887(2)	−1473(9)	5199(5)	77(7)
O(15)	4819(2)	−1189(10)	5506(4)	67(7)
O(16)	4735(2)	4034(11)	4195(5)	85(8)
O(17)	4674(2)	5669(12)	6348(6)	105(10)
O(18)	5155(2)	1937(11)	6065(5)	76(7)
C(1)	2907(3)	2750(16)	4778(8)	52(11)
C(2)	2891(3)	4764(17)	3725(8)	61(11)
C(3)	2652(3)	2276(16)	3509(8)	58(11)
C(4)	3310(3)	−19(16)	4715(9)	46(10)
C(5)	3602(3)	−55(15)	3582(8)	50(10)
C(6)	3033(3)	−410(13)	3465(8)	51(10)
C(7)	3656(3)	2612(17)	6011(8)	57(11)
C(8)	3919(3)	5150(16)	5919(8)	53(10)
C(9)	3413(3)	5010(18)	5367(8)	67(11)
C(10)	3899(3)	3732(17)	3385(8)	56(11)
C(11)	4130(3)	5664(15)	4379(8)	43(10)
C(12)	3592(3)	5782(17)	3913(9)	67(12)
C(13)	4359(3)	705(16)	4232(8)	50(10)
C(14)	4046(3)	−583(15)	5164(7)	37(9)
C(15)	4619(3)	−405(16)	5359(7)	53(10)
C(16)	4681(3)	3648(17)	4708(9)	58(11)
C(17)	4620(3)	4690(19)	6011(9)	67(12)
C(18)	4930(4)	2412(15)	5845(7)	55(11)
N(1)	3134(3)	2383(13)	2519(7)	59(9)
C(19)	3149(3)	2196(15)	3180(8)	52(10)
C(20)	3261(3)	1418(17)	2063(8)	73(12)
C(21)	3037(4)	358(20)	1704(9)	111(18)
C(22)	2983(3)	3681(18)	2149(8)	62(12)
C(23)	3197(4)	4873(20)	2093(10)	103(16)
N(2)	4432(3)	1255(4)	6676(6)	58(9)
C(24)	4438(3)	1580(13)	6039(7)	40(9)

TABLE 6 (continued)

Atom	x	y	z	U_{eq}
C(25)	4309(3)	−87(19)	6899(7)	66(12)
C(26)	4545(4)	−1214(18)	7151(8)	86(14)
C(27)	4556(3)	2272(18)	7256(7)	64(12)
C(28)	4329(3)	3094(17)	7537(8)	81(12)
C(29) [b]	2333(6)	2314(18)	194(22)	231(24) [c]
Cl	2481(1)	3966(6)	216(3)	178(3) [c]

[a] U_{eq} is defined as the geometric mean of the diagonal elements of the diagonalised matrix of U_{ij}. [b] Disordered, occupancy = 0.5. [c] Isotropic U values.

in Fig. 1. Figure 2 presents a stereoview of the molecule. Lists of thermal factors, hydrogen coordinates and structure factors are available from the authors (GJK).

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References

1 H.G. Raubenheimer, G.J. Kruger and A. van A. Lombard, submitted for publication.
2 W.K. Dean, J. Organomet. Chem., 135 (1977) 195.
3 H.G. Raubenheimer, G.J. Kruger and A. Lombard, J. Organomet. Chem., 240 (1982) C11.
4 H. Alper and A.S.K. Chan, Inorg. Chem., 13 (1974) 225.
5 G.J. Kruger, L. Linford and H.G. Raubenheimer, J. Organomet. Chem., 262 (1984) 69.
6 G.C. Cash, R.C. Pettersen and R.B. King, J. Chem. Soc., Chem. Commun., (1977) 30.
7 W.K. Dean and D.G. VanDerveer, J. Organomet. Chem., 146 (1978) 143.
8 D.F. Shriver, The Manipulation of Air-Sensitive Compounds, McGraw Hill, New York, 1969.
9 N. Walker and D. Stuart, Acta Cryst. A, 39 (1983) 158.
10 G.M. Sheldrick in H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld and G.C. Bassi (Eds.), Computing in Crystallography, Delft, University Press, 1978.
11 International Tables for X-ray Crystallography, 1974. Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht).