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Synthesis and characterization of thio-bis(t-butylphosphido)diiron hexacarbonyls, $Fe_2(CO)_6(Bu^tP)_2S_x$ ($x = 2, 3$). Molecular structure of $Fe_2(CO)_6(Bu^tPS_2PBu^t)$

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Abstract

Two isomers of dithio-bis(t-butylphosphido)diiron hexacarbonyl, $Fe_2(CO)_6$-$(Bu^tP)_2S_2$, and trithio-bis(t-butylphosphido)diiron hexacarbonyl, $Fe_2(CO)_6$-$(Bu^tP)_2S_3$were isolated from the reaction of the lithio compound $Fe_2(CO)_6(Bu^tPLi)_2$ with sulfur monochloride, S_2Cl_2 at $-10\,°C$. The new complexes were characterized by IR, ^1H NMR and mass spectra and elemental analysis. The crystal and molecular structure of $Fe_2(CO)_6(Bu^tPS_2PBu^t)$ (I) was determined by single crystal X-ray diffraction. Compound I crystallises in the orthorhombic system, space group $Pna2_1$ with a 2433.1(3), b 906.75(8), c 973.43(7) pm; $Z = 4$; d_{calc} 1.61 g cm^{-3}; $R_1 = 0.044$; $R_2 = 0.0515$. The molecular symmetry of compound I is approximately C_{2v} and the axis of the two sulfur atoms lies in the same plane which includes the two phosphorus atoms and the two P–C bonds with the Bu^t groups. Reaction of the lithio compound $Fe_2(CO)_6(Bu^tPLi)_2$ with CH_2I_2 yielded $Fe_2(CO)_6(Bu^tP)_2CH_2$, along with an iron cluster of unknown structure.

Introduction

Organometallic anions are often reactive nucleophiles and a large number of new products have been isolated from the reaction of organometallic anions with different electrophiles [1,2]. Reaction of sulfur monochloride S_2Cl_2 and the dichlo-

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ride SCl_2 with organometallic anions has been used to introduce bridged sulfur atoms between two metal atoms. Organometallic sulfur compounds having the formula $L_nMS_xML_n$, (M = Fe, (x = 1, 2, 3, 4) [3], M = Cr, (x = 1); M = Mo, W, x = 2 [4]) which can be regarded as organometallic sulfanes were obtained from such reactions. SCl_2 was also used to introduce (μ_3-S) in the iron cluster (μ_3-S)$Fe_3(CO)_9$(μ_3-RP) when the anion $\{Fe_3(CO)_9(RP)\}^{2-}$ was treated with SCl_2 at 20 °C [5]. These reactions demonstrate that S_2Cl_2 and SCl_2 are versatile reagents for the preparation of sulfur-bridged organometallic compounds. We report here the preparation of $Fe_2(CO)_6(Bu^tP)_2S_x$ (x = 2, 3) from the reaction of the phosphorus-centered anion, $\{Fe_2(CO)_6(Bu^tP)_2\}^{2-}$ with S_2Cl_2. The reaction of the anion with CH_2I_2 is also described.

Experimental

The doubly t-BuPH-bridged dinuclear iron carbonyl, $Fe_2(CO)_6$-$(BuPH)_2$ was prepared as described previously [6]. It was deprotonated by MeLi in diethyl ether at −10 °C [7]. The solution of the dilithio species, $Fe_2(CO)_6(Bu^t\text{-}PLi)_2$ thus obtained was used directly for the reactions described below.

Sulfur monochloride was used as purchased. All reactions and manipulations of chemicals were performed under dry nitrogen. Infrared spectra were recorded with a Perkin–Elmer 782 spectrometer and ^1H NMR spectra with a Varian T-60 instrument. Spectral data are presented in Table 1.

Reaction of S_2Cl_2 with $\{Fe_2(CO)_6(Bu^tP)_2\}^{2-}$

A solution of S_2Cl_2 (0.5 g, 3.5 mmol) in 30 ml diethyl ether was added slowly (20 min) to a rapidly stirred solution of $\{Fe_2(CO)_6(Bu^tP)_2\}^{2-}$ (1.60 g, 3.5 mmol) in 150

Table 1

Properties of compounds I–V

Compound	Color	M.p. (°C)	IR ν(CO) (cm^{-1}) [a]	^{1}H NMR [c] $\delta(CH_3)$ [J]
$Fe_2(CO)_6(Bu^tPS_2PBu^t)$ (I)	orange	99	2068m, 2038s 1986s	1.75 [17.4]
$Fe_2(CO)_6\{S\text{-}P(Bu^t)SP(Bu^t)\}$ (II)	brown-red	84–85	2069s, 2030s 1998s, 1987s 1972s, 1965sh	1.23 [20], 1.6 [19]
$Fe_2(CO)_6\{S\text{-}P(Bu^t)S_2P(Bu^t)\}$ (III)	brown-red	129–130 (dec.)	2082s, 2048vs [b] 2022w, 1997m 1987vs	1.47 [21] 1.65 [18]
IV	yellow-orange	134–135 (dec.)	2059m, 2018s 1983s, 1965m 1948w	1.00 [16], 1.45 [18]
V	black	240–5 (dec.)	2957m, 2032vs 2025sh, 1984vs 1978sh, 1965w 1945m, 1930w	See Fig. 3

[a] IR spectra were measured in n-pentane. [b] In petroleum ether (B.p. 40–60 °C). [c] In $CDCl_3$; δ in ppm, J in Hz.

ml diethyl ether at −10 °C. A color change from red to dark brown and formation of a precipitate were observed. The mixture was stirred for an additional 1 h and then warmed to room temperature. Solvent was removed in vacuo and the residue was extracted with 20 ml benzene/petroleum ether (1/20, v/v). The extract was filtered and chromatographed on silica. Elution with petroleum ether (b.p. 40–60 °C)

Table 2

Atomic coordinates for $Fe_2(CO)_6(Bu^tPS_2PBu^t)$ [a]

Atom	x	y	z
Fe(1)	0.1567(1)	0.1111(1)	0.0000(0)
Fe(2)	0.1543(1)	0.3454(1)	−0.1642(2)
S(1)	0.0167(1)	0.2175(4)	−0.0705(4)
S(2)	0.0407(1)	0.0997(3)	−0.2381(4)
P(1)	0.0951(1)	0.2907(2)	−0.0004(3)
P(2)	0.1272(1)	0.1178(3)	−0.2150(3)
O(11)	0.2158(4)	0.200(1)	0.2483(9)
O(12)	0.0885(4)	−0.1197(9)	0.119(1)
O(13)	0.2593(4)	−0.050(1)	−0.0558(9)
O(21)	0.2700(4)	0.3114(9)	−0.2381(9)
O(22)	0.1027(4)	0.473(1)	−0.4051(9)
O(23)	0.1803(4)	0.6315(7)	−0.0432(8)
C(11)	0.1929(5)	0.172(1)	0.152(1)
C(12)	0.1149(5)	−0.032(1)	0.072(1)
C(13)	0.2200(5)	0.013(1)	−0.041(1)
C(21)	0.2247(4)	0.321(1)	−0.214(1)
C(22)	0.1230(4)	0.424(1)	−0.310(1)
C(23)	0.1700(5)	0.518(1)	−0.085(1)
C(1)	0.0714(4)	0.408(1)	0.147(1)
C(5)	0.1498(6)	0.001(1)	−0.366(1)
C(2)	0.1223(6)	0.466(1)	0.229(1)
H(21)	0.1444(6)	0.383(1)	0.286(1)
H(22)	0.1110(6)	0.555(1)	0.297(1)
H(23)	0.1482(6)	0.508(1)	0.148(1)
C(3)	0.0388(5)	0.538(1)	0.089(1)
H(31)	0.0008(5)	0.508(1)	0.039(1)
H(32)	0.0637(5)	0.603(1)	0.020(1)
H(33)	0.0297(5)	0.602(1)	0.180(1)
C(4)	0.0349(6)	0.306(1)	0.240(1)
H(41)	0.0519(6)	0.200(1)	0.267(1)
H(42)	−0.0066(6)	0.292(1)	0.203(1)
H(43)	0.0343(6)	0.376(1)	0.331(1)
C(6)	0.2101(6)	0.021(2)	−0.393(1)
H(61)	0.2102(6)	0.136(2)	−0.422(1)
H(62)	0.2282(6)	−0.044(2)	−0.475(1)
H(63)	0.2337(6)	0.006(2)	−0.300(1)
C(7)	0.1348(7)	−0.161(1)	−0.335(1)
H(71)	0.1544(7)	−0.214(1)	−0.248(1)
H(72)	0.1415(7)	−0.226(1)	−0.426(1)
H(73)	0.0912(7)	−0.151(1)	−0.315(1)
C(8)	0.1133(6)	0.049(2)	−0.493(1)
H(81)	0.1185(6)	0.163(2)	−0.519(1)
H(82)	0.0701(6)	0.025(2)	−0.479(1)
H(83)	0.1292(6)	−0.020(2)	−0.575(1)

[a] Hydrogen atoms were assigned a common temperature factor of 0.111.

yielded a yellow band, from which $Fe_2(CO)_6(Bu^tPS_2PBu^t)$ (I) was isolated. It was recrystallized from n-pentane at −20 °C as orange crystals. 0.13 g, yield 25%, m.p. 99 °C. Anal. Found: C, 32.59; H, 3.53; S, 12.16. $C_{14}H_{18}Fe_2O_6P_2S_2$ calc: C, 32.33; H, 3.46; S, 12.31%. EI-MS *m/e* 520 (M^+). Further elution with a mixture of petroleum ether/benzene (15/1, v/v) developed two reddish-orange bands. The first band yielded brownish-red crystals of $Fe_2(CO)_6(Bu^tP)_2S_2$ (II), which was recrystallized from n-pentane. 0.102 g, yield 18% m.p. 84–85 °C. Anal. Found: C, 32.76; H, 3.54; S, 12.08. $C_{14}H_{18}Fe_2O_6P_2S_2$ calc: C, 32.33; H, 3.46; S, 12.31%. EI-MS *m/e* 520 (M^+). The second reddish-orange band was eluted with a mixture of petroleum ether/benzene (5/1, v/v). The product, $Fe_2(CO)_6(Bu^tP)_2S_3$ (III), was recrystallized from n-pentane as brown-red crystals m.p. 129–130 °C (decomposition), 0.093 g, yield 10%. Anal. Found: C, 31.00; H, 3.39; S, 17.07. $C_{14}H_{18}Fe_2O_6P_2S_3$ calc: C, 30.43; H, 3.26; S, 17.39%.

Reaction of CH_2I_2 with $\{Fe_2(CO)_6(Bu^tP)_2\}^{2-}$

A solution of CH_2I_2 (0.1 ml, 1.17 μmol) in 20 ml diethyl ether was added slowly to a rapidly stirred solution of $\{Fe_2(CO)_6(Bu^tP)_2\}^{2-}$ (0.3 g, 0.7 mmol) in 100 ml diethyl ether at −10 °C. The mixture was evaporated to dryness under vacuum and the residue extracted with petroleum ether. The extract was filtered and the volume of the filtrate was reduced and the solution then divided between two preparative

Table 3

Anisotropic thermal parameters for $Fe_2(CO)_6(Bu^tPS_2PBu^t)$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(1)	0.0425(6)	0.0381(5)	0.0388(6)	0.0003(5)	−0.000(6)	0.0049(6)
Fe(2)	0.0417(6)	0.0391(5)	0.0363(5)	−0.0039(5)	−0.0016(6)	0.0041(5)
S(1)	0.042(1)	0.079(2)	0.077(2)	−0.002(1)	0.004(1)	−0.007(2)
S(2)	0.051(1)	0.071(2)	0.074(2)	−0.009(1)	−0.014(1)	0.011(2)
P(1)	0.042(1)	0.042(1)	0.042(1)	0.0015(9)	0.004(1)	0.002(1)
P(2)	0.045(1)	0.043(1)	0.041(1)	−0.004(1)	−0.0020(9)	0.000(1)
O(11)	0.108(7)	0.105(7)	0.078(6)	0.014(6)	−0.052(6)	−0.018(6)
O(12)	0.099(7)	0.068(5)	0.155(9)	−0.029(5)	0.036(7)	0.024(6)
O(13)	0.071(6)	0.133(8)	0.084(6)	0.049(6)	0.013(5)	0.006(6)
O(21)	0.079(6)	0.086(6)	0.080(6)	−0.015(5)	0.014(5)	−0.010(5)
O(22)	0.112(7)	0.086(6)	0.070(6)	−0.007(5)	−0.036(5)	0.022(5)
O(23)	0.106(6)	0.044(4)	0.078(6)	−0.010(4)	−0.004(5)	−0.009(4)
C(11)	0.070(7)	0.048(5)	0.062(7)	0.011(5)	−0.011(6)	0.004(5)
C(12)	0.067(7)	0.059(6)	0.065(7)	0.000(6)	0.011(6)	−0.004(6)
C(13)	0.081(8)	0.074(7)	0.044(6)	0.003(6)	−0.002(5)	0.011(5)
C(21)	0.045(5)	0.046(5)	0.053(5)	−0.010(4)	0.001(4)	0.001(4)
C(22)	0.064(6)	0.047(5)	0.053(6)	0.001(5)	−0.005(5)	0.004(5)
C(23)	0.063(6)	0.049(5)	0.048(5)	−0.005(5)	−0.007(5)	0.008(5)
C(1)	0.060(6)	0.059(6)	0.051(5)	−0.015(5)	0.010(5)	0.000(5)
C(5)	0.10(1)	0.052(6)	0.043(5)	−0.001(6)	0.009(6)	−0.009(5)
C(2)	0.12(1)	0.084(8)	0.050(7)	0.020(8)	0.010(7)	−0.024(6)
C(3)	0.092(9)	0.071(8)	0.083(9)	0.027(7)	−0.011(8)	−0.022(7)
C(4)	0.084(9)	0.10(1)	0.069(8)	−0.016(8)	0.030(7)	−0.009(7)
C(6)	0.13(1)	0.11(1)	0.066(8)	−0.01(1)	0.045(9)	−0.031(8)
C(7)	0.13(1)	0.046(6)	0.093(4)	−0.008(7)	0.030(9)	−0.014(7)
C(8)	0.13(1)	0.11(1)	0.049(6)	0.020(9)	−0.013(9)	−0.023(8)

thin layer silica plates (20 × 20 cm, 0.1 mm). Elution with a mixture of petroleum ether/diethyl ether, (10/1, v/v) under argon produced two well-separted bands, the first yellow and the second brownish-red. Both compounds were extracted with diethyl ether and the extracts were evaporated and the residues recrystallized from n-pentane at −20 °C. The yellow band yielded yellow-orange crystals of IV, m.p. 134–135 °C (dec.). Anal. Found: C, 39.00; H, 4.48. $C_{15}H_{20}Fe_2O_6P_2$ calc: C, 38.28; H, 4.25%. Black crystals of V were similarly obtained from the brownish-red band. It decomposes in the range 240–245 °C. Anal. Found: C, 38.17; H, 4.37. $C_{29}H_{38}Fe_4O_{12}P_4$ calc: C, 37.58; H, 4.10%. EI-MS ions with m/e 828 $[M^+ - (CH_2 + 3CO)]$ and 828-n(CO) with $n = 1$–9 were observed.

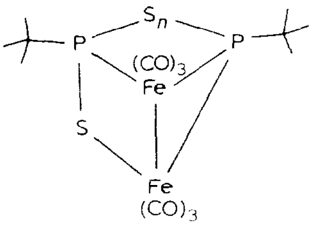
X-Ray study

Single-crystal specimens suitable for X-ray study were obtained by recrystallization of $Fe_2(CO)_6(Bu^tPS_2PBu^t)$ (I) from n-pentane at −20 °C. A single crystal of dimensions ca. 0.45 × 0.45 × 0.20 mm was selected for data collection. Compound I crystallises in the orthorhombic system, space group $Pna2_1$ with a 2433.1(3), b 906.75(8), c 973.43(7) pm, d_{calc} 1.61 g cm^{-3}, μ 8.15 cm^{-1}, $Z = 4$. Data were collected with an Enraf–Nonius CAD-4 diffractometer with Ag-K_α radiation. A total of 3519 reflections were collected, and 2352 reflections with $I > 3\sigma$ used to solve and refine the structure. The structure was solved (Patterson) and refined with the SHELX program. All nonhydrogen atoms were refined with anisotropic thermal parameters. The methyl hydrogen atoms were treated as rigid groups and were refined with a common isotropic thermal parameter. The refinement converged at $R_1 = 0.044$ and $R_2 = 0.0515$. A list of atomic coordinates is given in Table 2 and anisotropic thermal parameters in Table 3 [16].

Results and discussion

Thio-bis(dialkylphosphine) compounds, $R_2PS_xPR_2$, ($x = 2, 1$) are known and can be stabilized by complexation, as, for example, in $\{(\eta^5\text{-}C_5H_5)Mn(CO)_2Me_2PS\}_2$ and $\{(\eta^5\text{-}C_5H_5)Mn(CO)_2Me_2P\}_2S$, in which Mn–P bonds exhibit π-character, as is evident from the crystal structure [8]. These complexes were prepared by oxidation of $(\eta^5\text{-}C_5H_5)Mn(CO)_2Me_2PSH$ [9]. We describe here a further example of a coordinated dithiobis(t-butylphosphine) in which each phosphorus is a three-electron donor and doubly-bridged to two iron atoms. The complex was isolated from the reaction of the lithio compound $Fe_2(CO)_6(Bu^tPLi)_2$ with S_2Cl_2 at −10 °C. The dithio-bis(t-butylphosphine)diiron complex, $Fe_2(CO)_6(Bu^tPS_2PBu^t)$ (I) was separated from two other thiophosphineiron complexes (II and III) by column chromatography, and recrystallized from n-pentane. The infrared spectrum of compound I showed three strong ν(CO) bands, all attributed to terminal carbonyls. Almost all related phosphine-bridged dinuclear iron carbonyl complexes exhibit four to five carbonyl frequencies [10], and the appearence of only three ν(CO) bands for compound I could be due to identical frequencies for some stretching CO modes for the approximate C_{2v} symmetry of compound I.

The ^1H NMR spectrum of compound I showed a pseudotriplet at δ 1.75 ppm for the protons of the t-butyl groups with a coupling constant 17.4 Hz. This indicates that the two t-butyl groups are chemically equivalent. A similar result was observed for the complex $Fe_2(CO)_2(Bu^tP)_2$, which contains bridged ditertiary butyldiphosphene [7,10].



$n = 1$, II

$n = 2$, III

Fig. 1. Proposed structures for compounds II and III.

Two other compounds, II and III, were isolated from the $Fe_2(CO)_6(Bu^tPLi)_2/S_2Cl_2$ reaction. Elemental analysis and mass spectra indicate that they are $Fe_2(CO)_6(Bu^tP)_2S_2$ and $Fe_2(CO)_6(Bu^tP)_2S_3$, respectively. In contrast to compound I, these species display six or five ν(CO) bands and chemically nonequivalent Bu^t groups in their ^1H NMR spectra (Table 1). We therefore suggest μ_2-η^3-P_2S_2 and μ_2-η^3-P_2S_3 structures for these species (see Fig. 1).

It is not surprising that the products formed from the reaction of the lithio compound, $Fe_2(CO)_6(Bu^tPLi)_2$ with S_2Cl_2 contained numbers of sulfur atoms different from those in the sulfur reagent. This can be attributed to the well-known tendency of sulfur towards catenation [11].

Reaction of $Fe_2(CO)_6(Bu^tPLi)_2$ with CH_2I_2 yielded compound IV, as orange crystals. The infrared spectrum (Table 1) of IV in n-pentane showed five ν(CO) bands. This pattern is very similar to that observed for the analogous compound, $Fe_2(CO)_6(C_6H_5P(CH_2)_3PC_6H_5)$, which was prepared from the reaction of $Fe_2(CO)_6(C_6H_5PLi)_2$with $Br(CH_2)_3Br$ [12]. However, the ^1H NMR spectrum of IV showed two doublets, at δ 1.00 and 1.45 ppm, that must be attributed to two chemically nonequivalent t-butyl groups. The bridged methylene protons appeared as a doublet of doublets centered at 1.21 ppm. This means that the phosphorus

Fig. 2. IR spectrum (n-pentane) for compound V.

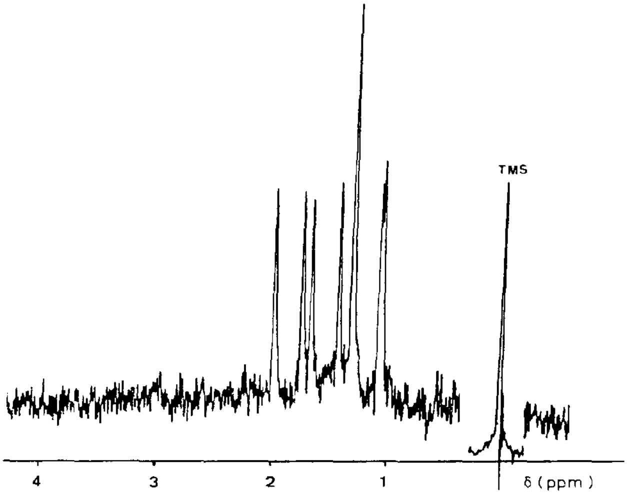


Fig. 3. ^{1}H NMR spectrum ($CDCl_3$) for compound V.

atoms are unsymmetrically coordinated to the iron atoms; possibly a P–C–Fe linkage, similar to that observed by Huttner [13], is involved.

Reaction of CH_2I_2 with $Fe_2(CO)_6(Bu^tPLi)_2$ also produced black crystals of compound V. Its infrared spectrum is shown in Fig. 2 (cf. Table 1). The ^{1}H NMR spectrum is shown in Fig. 3, and reveals the presence of four chemically nonequivalent t-butyl groups. Compound V probably involves a cluster of four iron atoms without bridging CO groups.

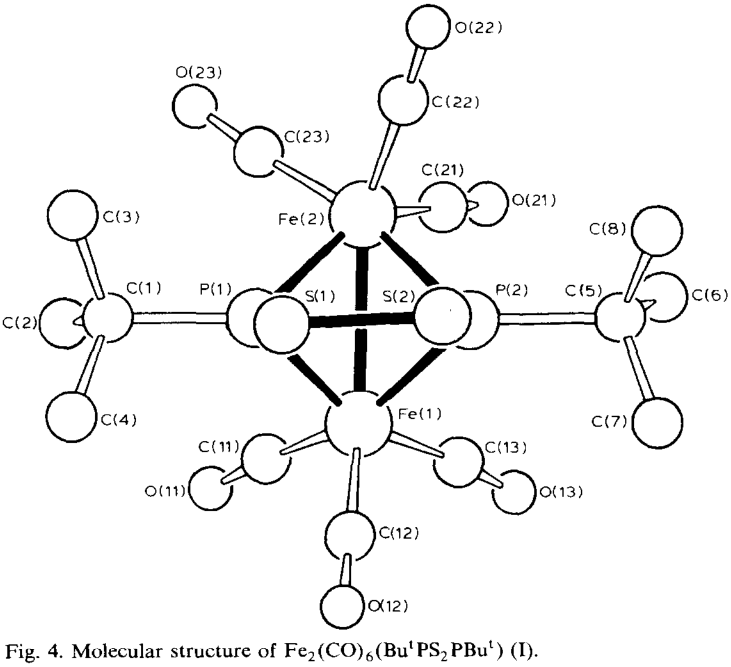


Fig. 4. Molecular structure of $Fe_2(CO)_6(Bu^tPS_2PBu^t)$ (I).

Molecular structure of $Fe_2(CO)_6(Bu^tPS_2PBu^t)$ (I)

The molecular structure of compound I is shown in Fig. 4 with a direction of view which illustrates the central folded Fe_2P_2 ring with basic geometry typical of $Fe_2(CO)_6(\mu\text{-}X_2)$ compounds (14,15). Bond distances and selected bond angles are shown in Table 4 and Table 5, respectively [16]. A side view of I, with selected bond angles is shown in Fig. 5. The dithio, S–S, group in the bridged $Bu^tPS_2PBu^t$, lies in the plane which includes the two phosphorous and their bonds to the two t-butyl carbons. The molecule has approximate C_{2v} symmetry. The structure of the Fe_2P_2 unit has general features similar to the other phosphorus-bridged compounds [15]. The Fe–Fe bond distance is 2.659, Fe–P distance 2.213 Å. The P · · · P distance of 2.712 Å is slightly shorter than that (2.86–2.725 Å) in other $Fe_2(CO)_6(\mu\text{-}PRR')_2$ complexes with nonbonded P · · · P interactions [15]. This smaller P · · · P distance is a result of the S_2 bridge, which also results in smaller P–Fe–P angles, 76.1 and 76.0°, compared with the range of 80.5 to 76.4 in $Fe_2(CO)_6(\mu\text{-}PRR')_2$ compound. The Fe–P–Fe angles of 73.7(1) and 74.0(1)° are in the same range (72.0–74.3°) of the bridged phosphorous complexes. The P–S bond distances of 2.133(3) and 2.124(4) Å are a typical of single P–S σ bonds as found in phosphine sulfide [17].

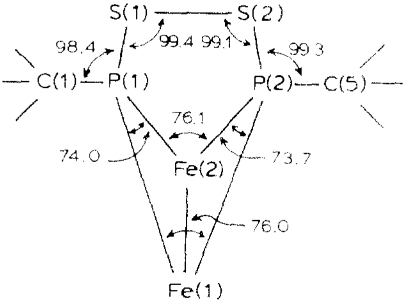


Fig. 5. Side view of $Fe_2(CO)_6(Bu^tPS_2PBu^t)$ (I) with selected bond angles, showing the approximate C_{2v} symmetry for the compound.

Table 4

Bond distances (Å) for $Fe_2(CO)_6(Bu^tPS_2PBu^t)$

Fe(1)–Fe(2)	2.659(2)	P(1)–C(1)	1.878(11)
Fe(1)–P(1)	2.213(2)	P(1)–S(1)	2.133(3)
Fe(1)–P(2)	2.214(3)	S(1)–S(2)	2.036(5)
Fe(1)–C(11)	1.811(12)	P(2)–C(5)	1.894(11)
Fe(1)–C(12)	1.793(11)	P(2)–S(2)	2.124(4)
Fe(1)–C(13)	1.821(12)		
Fe(2)–P(1)	2.204(3)		
Fe(2)–P(2)	2.222(3)		
Fe(2)–C(21)	1.794(9)		
Fe(2)–C(22)	1.761(11)		
Fe(2)–C(23)	1.788(10)		

The C–O distances range from 1.11(1) to 1.17(2) Å. The C–C distances in the t-Bu group range from 1.50(1) to 1.57(1) Å.

Table 5

Selected bond angles (°) for $Fe_2(CO)_6(Bu^tPS_2PBu^t)$

Fe(1)-P(2)-C(5)	128.7(4)	C(11)-Fe(1)-Fe(2)	105.1(3)
Fe(1)-P(1)-C(1)	128.5(3)	C(11)-Fe(1)-P(1)	96.1(3)
Fe(2)-P(2)-C(5)	127.4(4)	C(11)-Fe(1)-P(2)	157.5(3)
Fe(2)-P(1)-C(1)	128.8(3)	C(12)-Fe(1)-Fe(2)	143.3(4)
Fe(2)-P(2)-Fe(1)	73.7(1)	C(12)-Fe(1)-P(1)	98.6(4)
Fe(2)-P(1)-Fe(1)	74.0(1)	C(12)-Fe(1)-P(2)	102.0(4)
P(2)-Fe(1)-P(1)	76.0(1)	C(13)-Fe(1)-Fe(2)	106.0(4)
P(2)-Fe(2)-P(1)	76.1(1)	C(13)-Fe(1)-P(1)	158.5(4)
P(2)-Fe(1)-Fe(2)	53.3(1)	C(13)-Fe(1)-P(2)	94.6(3)
P(1)-Fe(2)-Fe(1)	53.1(1)	C(21)-Fe(2)-Fe(1)	92.5(3)
S(1)-P(1)-C(1)	98.4(3)	C(21)-Fe(2)-P(1)	142.3(3)
S(2)-P(2)-C(5)	99.3(4)	C(21)-Fe(2)-P(2)	96.3(3)
S(2)-P(2)-Fe(1)	114.8(1)	C(22)-Fe(2)-Fe(1)	144.7(3)
S(2)-P(2)-Fe(2)	112.9(1)	C(22)-Fe(2)-P(1)	113.0(4)
S(1)-P(1)-Fe(1)	112.2(1)	C(22)-Fe(2)-P(2)	93.8(3)
S(1)-P(1)-Fe(2)	115.0(2)	C(23)-Fe(2)-Fe(1)	115.7(3)
P(2)-S(2)-S(1)	99.1(2)	C(23)-Fe(2)-P(1)	91.3(3)
P(1)-S(1)-S(2)	99.4(2)	C(23)-Fe(2)-P(2)	166.7(3)

The Fe-C-O bond angles are in the range of 174.5 to 179.3

The S-S bond distance 2.036(5) Å is consistent with the distance of S-S single bond of the average of 2.06 Å in orthorhombic S_8 [18] and 2.04 Å in $(CH_3)_2S_3$ [19].

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