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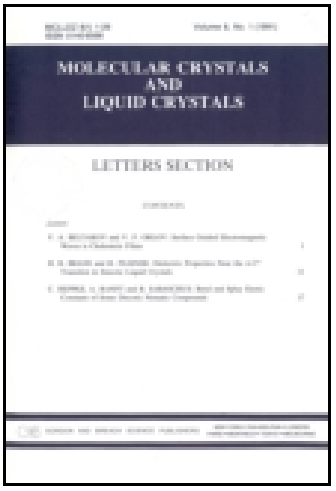

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Reactions of Cluster Complex Triiron Enneacarbonyl Disulfide with Phosphine Ligands

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Reactions of Cluster Complex Triiron Enneacarbonyl Disulfide with Phosphine Ligands

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*The cluster derivatives of the type $[Fe_3(\mu_3\text{-}S)_2(CO)_8L]$ or $[Fe_3(\mu_3\text{-}S)_2(CO)_7L]$ (L = phosphine ligands) have been prepared by carbonyl substitution reactions of $[Fe_3(\mu_3\text{-}S)_2(CO)_9]$ (**A**) with monophosphine PPh_3 or diphosphine $Ph_2PCH_2PPh_2$. Reaction of complex **A** with PPh_3 in the presence of $Me_3NO{\cdot}2H_2O$ in MeCN afforded $[Fe_3(\mu_3\text{-}S)_2(CO)_8PPh_3]$ (**1**) in 65% yield, whereas reaction of **A** with PPh_3 at reflux in toluene gave **1** and $[Fe_3(\mu_3\text{-}S)_2(CO)_7(PPh_3)_2]$ (**2**) in 36% and 42% yields, respectively. However, complex $[Fe_3(\mu_3\text{-}S)_2(CO)_7(Ph_2PCH_2PPh_2)]$ (**3**) could be prepared by two methods: (i) reaction of **A** with $Ph_2PCH_2PPh_2$ in the presence of $Me_3NO{\cdot}2H_2O$ in MeCN in 54% yield; and (ii) reaction of **A** with $Ph_2PCH_2PPh_2$ at reflux in toluene in 37% yield. The cluster complexes **1**–**3** were structurally characterized by elemental analysis, IR and NMR spectroscopic analysis. In addition, the molecular structures of **1** and **3** were determined by X-ray crystallography.*

[Supplemental materials are available for this article. Go to the publisher's online edition of Molecular Crystals and Liquid Crystals to view the free supplemental file.]

Keywords Carbonyl substitution; cluster complex; crystal structure; synthesis

1. Introduction

The triangular cluster complex $[Fe_3(\mu_3\text{-}S)_2(CO)_9]$ have been previously prepared by several methods: i) reaction of $Fe_3(CO)_{12}$ with ethylene sulfide in hexane under refluxing [1]; ii) the room temperature reaction of $Fe_2(CO)_9$ with $(\mu\text{-}S)_2Fe_2(CO)_6$ in THF [2]; iii) reaction between $HFe(CO)_4^-$ and sulfite ion [3]; iv) reaction between $Fe_3(CO)_{12}$ and cyclohexane sulfide or 3-chloropropylene sulfide in hexane under refluxing [4]; v) reaction of $Fe(CO)_5$ with bis(fluorocarbonyl)disulfane [5]. Derivatives of the type $[Fe_3(\mu_3\text{-}S)_2(CO)_8L]$ (L = $P(OC_6H_5)_3$, $P(n\text{-}C_4H_9)_3$ [6], CH_3CN [7], $P(SC_6H_5)Cl_2$ [8], $P(SC_6H_5)_3$ [9], $\eta^1\text{-}Ph_2PC_5H_4FeC_5H_4PPh_2$ [10]), $[Fe_3(\mu_3\text{-}S)_2(CO)_7L_2]$ (L = $P(OC_6H_5)_3$ $P(n\text{-}C_4H_9)_3$ [6], $N(C_2H_5)_3$ [7], $P(O\text{-}secC_4H_9)_3$, $P(S\text{-}nC_3H_7)_3$ [11]), or $[Fe_3(\mu_3\text{-}S)_2(CO)_7L]$ ($L = \mu_2\text{-}Ph_2PC_5H_4FeC_5H_4PPh_2$ [10]) were prepared by carbonyl substitution. In order to study ligand substitution effects, we selected the triangular cluster $[Fe_3(\mu_3\text{-}S)_2(CO)_9]$ and phosphine ligands. Fortunately, three cluster complexes $[Fe_3(\mu_3\text{-}S)_2(CO)_8PPh_3]$, $[Fe_3(\mu_3\text{-}S)_2(CO)_7(PPh_3)_2]$, and $[Fe_3(\mu_3\text{-}S)_2(CO)_7(Ph_2PCH_2PPh_2)]$ were prepared by carbonyl substitution and structurally characterized by spectroscopy and X-ray crystallography.

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2. Experimental

2.1. Materials and Methods

All reactions were performed using standard Schlenk and vacuum-line techniques under N_2 atmosphere. Toluene was distilled over sodium and acetonitrile was distilled over CaH_2 under N_2. $Me_3NO{\cdot}2H_2O$, PPh_3, $Ph_2PCH_2PPh_2$ and other materials were available commercially and used as received. $[Fe_3(\mu_3\text{-}S)_2(CO)_9]$ [2] was prepared according to literature procedures. IR spectra were recorded on a Nicolet MAGNA 560 FTIR spectrometer. ^1H (^{31}P, ^{13}C) NMR spectra were obtained on a Bruker Avance 500 MHz spectrometer. Elemental analyses were performed by a Perkin-Elmer 240C analyzer.

2.2. Synthesis of Complex $[Fe_3(\mu_3\text{-}S)_2(CO)_8PPh_3]$ (1)

To a solution of **A** (0.097 g, 0.2 mmol) in MeCN (10 mL) was added a solution of $Me_3NO{\cdot}2H_2O$ (0.022 g, 0.2 mmol) in MeCN (5 mL). The mixture was stirred at room temperature for 15 min and then was added PPh_3 (0.052 g, 0.2 mmol). The new mixture was stirred for 1 hr. The solvent was reduced *in vacuo* and the residue was subjected to TLC separation using petroleum ether as eluent. Collecting the main brown band afforded 0.093 g (65%) of **1** as a black solid. Anal. Calcd for $C_{26}H_{15}Fe_3O_8PS_2$: C, 43.49; H, 2.11. Found: C, 43.74; H, 2.25%. IR (KBr disk): $\nu_{C\equiv O}$ 2026 (s), 2000 (s), 1973 (s), and 1931 (s) cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): 7.38 (s, 15H, Ph*H*) ppm. ^{31}P NMR (200 MHz, $CDCl_3$, 85% H_3PO_4): –5.34 (s) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): 213.45 (C≡O), 137.15, 133.87, 133.72, 128.77, 128.54, and 128.51 (Ph*C*) ppm.

2.3. Synthesis of Complexes $[Fe_3(\mu_3\text{-}S)_2(CO)_8PPh_3]$ (1) and $[Fe_3(\mu_3\text{-}S)_2(CO)_7(PPh_3)_2]$ (2)

A mixture of **A** (0.097 g, 0.2 mmol), PPh_3 (0.157 g, 0.6 mmol), and toluene (15 mL) was refluxed for 2 hr. The solvent was reduced *in vacuo* and the residue was subjected to TLC separation using petroleum ether as eluent. Collecting the first brown band afforded 0.052 g (36%) of **1** as a black solid. Collecting the second brown band afforded 0.080 g (42%) of **2** as a black solid. **2**: Anal. Calcd for $C_{43}H_{30}Fe_3O_7P_2S_2$: C, 54.23; H, 3.18. Found: C, 54.48; H, 3.34%. IR (KBr disk): $\nu_{C\equiv O}$ 2039 (vs), 1995 (vs), and 1935 (vs) cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): 7.51, 7.41 (2s, 30H, Ph*H*) ppm. ^{31}P NMR (200 MHz, $CDCl_3$, 85% H_3PO_4): 60.25 (s) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): 212.78 (C≡O), 134.54, 134.22, 133.66, 133.58, 130.12, 128.34, and 128.26 (Ph*C*) ppm.

2.4. Synthesis of Complex $[Fe_3(\mu_3\text{-}S)_2(CO)_7(Ph_2PCH_2PPh_2)]$ (3)

Method A: To a solution of **A** (0.097 g, 0.2 mmol) in MeCN (10 mL) was added a solution of $Me_3NO{\cdot}2H_2O$ (0.022 g, 0.2 mmol) in MeCN (5 mL). The mixture was stirred at room temperature for 15 min and then was added $Ph_2PCH_2PPh_2$ (0.077 g, 0.2 mmol). The new mixture was stirred for 1 hr. The solvent was reduced *in vacuo* and the residue was subjected to TLC separation using CH_2Cl_2/petroleum ether ($v/v = 1{:}3$) as eluent. Collecting the main brown band afforded 0.087 g (54%) of **3** as a black solid. Anal. Calcd for $C_{32}H_{22}Fe_3O_7P_2S_2$: C, 47.33; H, 2.73. Found: C, 47.12; H, 2.63%. IR (KBr disk): $\nu_{C\equiv O}$ 2043 (vs), 2007 (vs), 1979 (vs), and 1937 (vs) cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$): 7.43, 7.26 (2s, 20H, Ph*H*), 3.02 (s, 2H, CH_2) ppm. ^{31}P NMR (200 MHz, $CDCl_3$, 85% H_3PO_4): 75.39 (s) ppm. ^{13}C

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Table 1. Crystal data and structure refinements details for **1** and **3**

Compound	**1**	**3**
Empirical formula	$C_{26}H_{15}Fe_3O_8PS_2$	$C_{32}H_{22}Fe_3O_7P_2S_2$
Formula weight	718.02	812.11
Temperature (K)	113(2)	113(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Triclinic	Triclinic
Space group	P-1	P-1
a (Å)	8.8265(16)	11.264(2)
b (Å)	9.8019(16)	11.503(2)
c (Å)	16.699(3)	12.939(3)
α (°)	106.143(6)	77.924(6)
β (°)	96.773(6)	88.356(9)
γ (°)	94.084(5)	84.700(8)
V (Å^3)	1369.9(4)	1632.3(6)
Z	2	2
D_{calc} (g·cm^{-3})	1.741	1.652
μ (mm^{-1})	1.827	1.588
F(000)	720	820
Crystal size (mm^3)	0.20 × 0.18 × 0.12	0.20 × 0.18 × 0.10
θ_{min}, θ_{max} (°)	2.18, 27.87	1.82, 27.85
Reflections collected/unique	14187/6446	16653/7619
R_{int}	0.0634	0.0386
hkl range	$-11 \le h \le 11$	$-14 \le h \le 13$
	$-12 \le k \le 11$	$-15 \le k \le 15$
	$-21 \le l \le 21$	$-16 \le l \le 16$
Completeness to θ_{max} (%)	98.7	98.2
Data/restraints/parameters	6446/638/523	7619/0/415
Goodness of fit on F^2	1.027	1.080
$R1/wR2$ $(I>2\sigma(I))$	0.0440/0.1352	0.0276/0.0721
$R1/wR2$ (all data)	0.0553/0.1397	0.0399/0.0761
Largest diff peak and hole/e Å^{-3}	1.389/−0.759	0.442/−0.495

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NMR (125 MHz, $CDCl_3$): 212.50, 205.73 (C≡O), 136.14, 135.95, 135.76, 131.97, 131.93, 131.89, 130.44, 128.63 (Ph*C*), and 30.28 (CH_2) ppm.

Method B: A mixture of **A** (0.097 g, 0.2 mmol), $Ph_2PCH_2PPh_2$ (0.077 g, 0.2 mmol) and toluene (10 mL) was refluxed for 1 hr. The solvent was reduced *in vacuo* and the residue was subjected to TLC separation using CH_2Cl_2/petroleum ether (v/v = 1:3) as eluent. Collecting the main brown band afforded 0.060 g (37%) of **3** as a black solid.

2.5. X-Ray Structure Determination

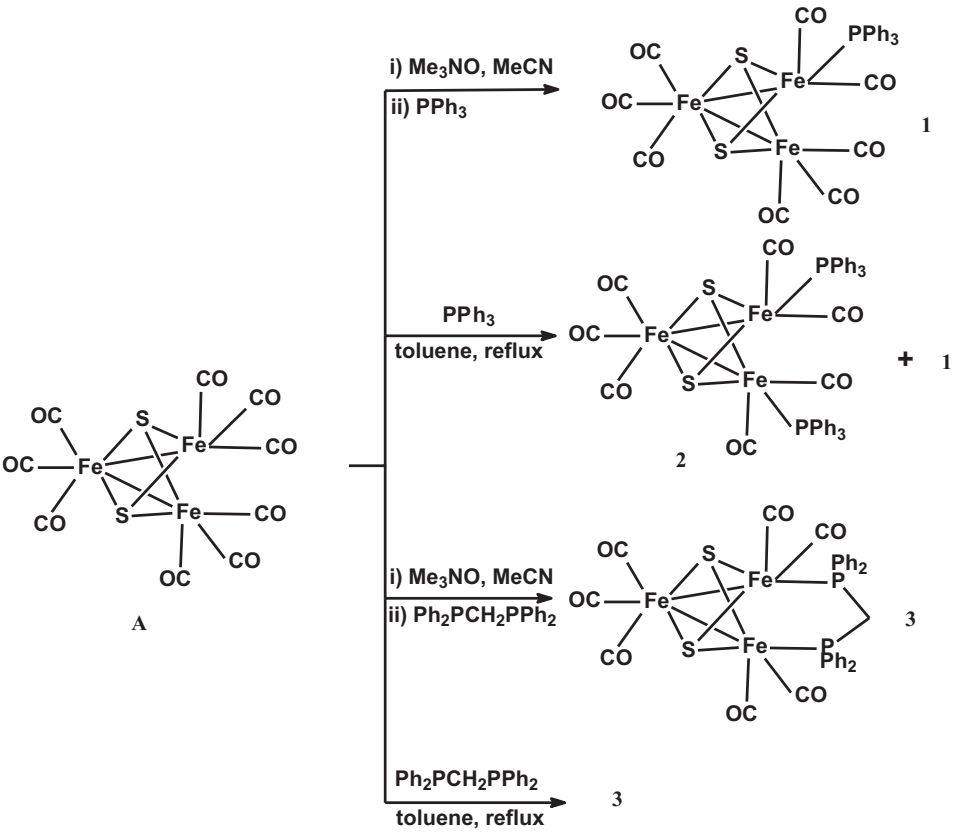
Single crystals of **1** and **3** suitable for X-ray diffraction analysis were grown by slow evaporation of CH_2Cl_2/hexane solutions of **1** and **3** at 4 °C. A single crystal of **1** or **3** was mounted on a Rigaku MM-007 CCD diffractometer. Data were collected at 113 K by using a graphite monochromator with Mo Kα radiation ($\lambda = 0.71073$ Å) in the ω-φ scanning mode.

Data collection, reduction and absorption correction were performed by *CRYSTALCLEAR* program [12]. The structure was solved by direct methods using the SHELXS-97 program [13] and refined by full-matrix least-squares techniques SHELXL-97 [14] on F^2. Hydrogen atoms were located using the geometric method. Details of crystal data, data collections, and structure refinement are summarized in Table 1.

3. Results and Discussion

3.1. Synthesis and Characterization

Synthetic methods for **1**-**3** are depicted in Scheme 1. We prepared complex **1** by carbonyl substitution reaction of parent complex **A** with PPh_3 in the presence of the CO-removing reagent $Me_3NO{\cdot}2H_2O$ in MeCN in 65% yield. Reaction of the same reactants at reflux in toluene afforded monosubstituted derivative **1** and disubstituted derivative **2** in 36% and 42% yields, respectively. Interesting, complex **3** with bridging diphosphine ligand could be prepared either by the reaction of **A** with $Ph_2PCH_2PPh_2$ in the presence of $Me_3NO{\cdot}2H_2O$ in 54% yield (method A) or by the reaction of **A** and $Ph_2PCH_2PPh_2$ at reflux in 37% yield (method B).



Scheme 1. Preparation of complexes **1**–**3**.

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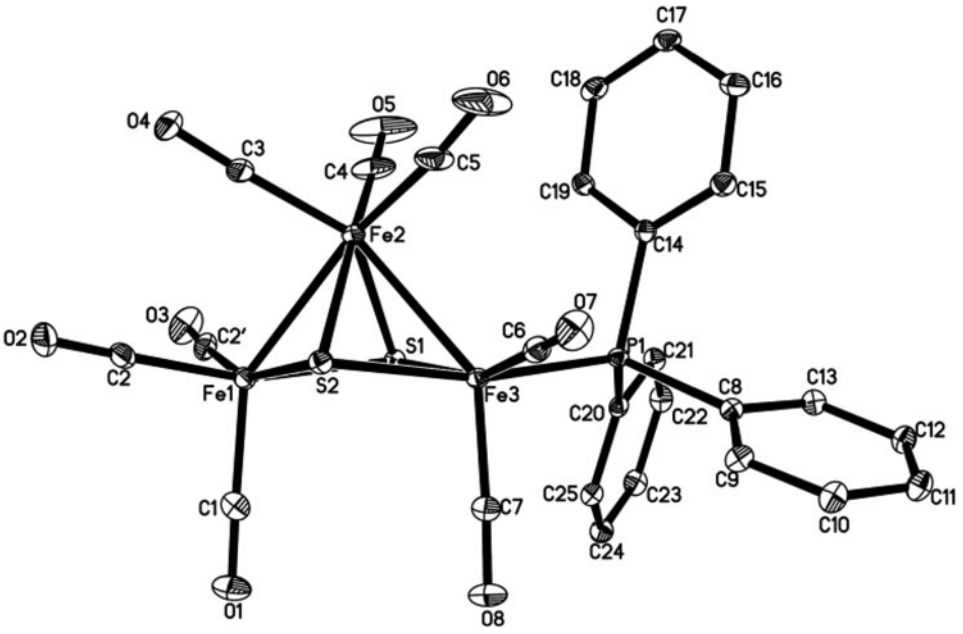

Figure 1. ORTEP view of **1** with 30% probability level ellipsoids.

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Table 2. Selected bond lengths (Å) and angles (°) for **1** and **3**

1			
Fe(1)-S(1)	2.2409(10)	Fe(2)-S(2)	2.2602(10)
Fe(1)-S(2)	2.2485(11)	Fe(2)-Fe(3)	2.6448(8)
Fe(1)-Fe(2)	2.5524(8)	Fe(3)-S(1)	2.2163(10)
Fe(2)-S(1)	2.2572(10)	Fe(3)-S(2)	2.2435(10)
S(1)-Fe(1)-Fe(2)	55.73(3)	S(1)-Fe(3)-S(2)	80.56(4)
S(2)-Fe(1)-Fe(2)	55.74(3)	S(1)-Fe(3)-P(1)	92.48(4)
S(1)-Fe(2)-S(2)	79.34(4)	S(2)-Fe(3)-P(1)	166.18(4)
S(1)-Fe(2)-Fe(1)	55.13(3)	S(1)-Fe(3)-Fe(2)	54.47(3)
Fe(1)-Fe(2)-Fe(3)	81.40(2)	P(1)-Fe(3)-Fe(2)	111.96(3)
3			
Fe(1)-S(1)	2.2356(7)	Fe(2)-S(2)	2.2578(8)
Fe(1)-S(2)	2.2430(8)	Fe(2)-Fe(3)	2.5930(7)
Fe(1)-Fe(2)	2.6141(7)	Fe(3)-S(1)	2.2276(8)
Fe(2)-S(1)	2.2538(8)	Fe(3)-S(2)	2.2316(7)
S(1)-Fe(1)-S(2)	80.81(3)	S(2)-Fe(2)-Fe(3)	54.248(19)
S(1)-Fe(1)-Fe(2)	54.71(2)	S(1)-Fe(2)-Fe(1)	54.066(19)
S(2)-Fe(1)-Fe(2)	54.757(19)	Fe(3)-Fe(2)-Fe(1)	80.267(18)
S(1)-Fe(2)-S(2)	80.10(3)	P(2)-Fe(3)-S(2)	94.83(3)

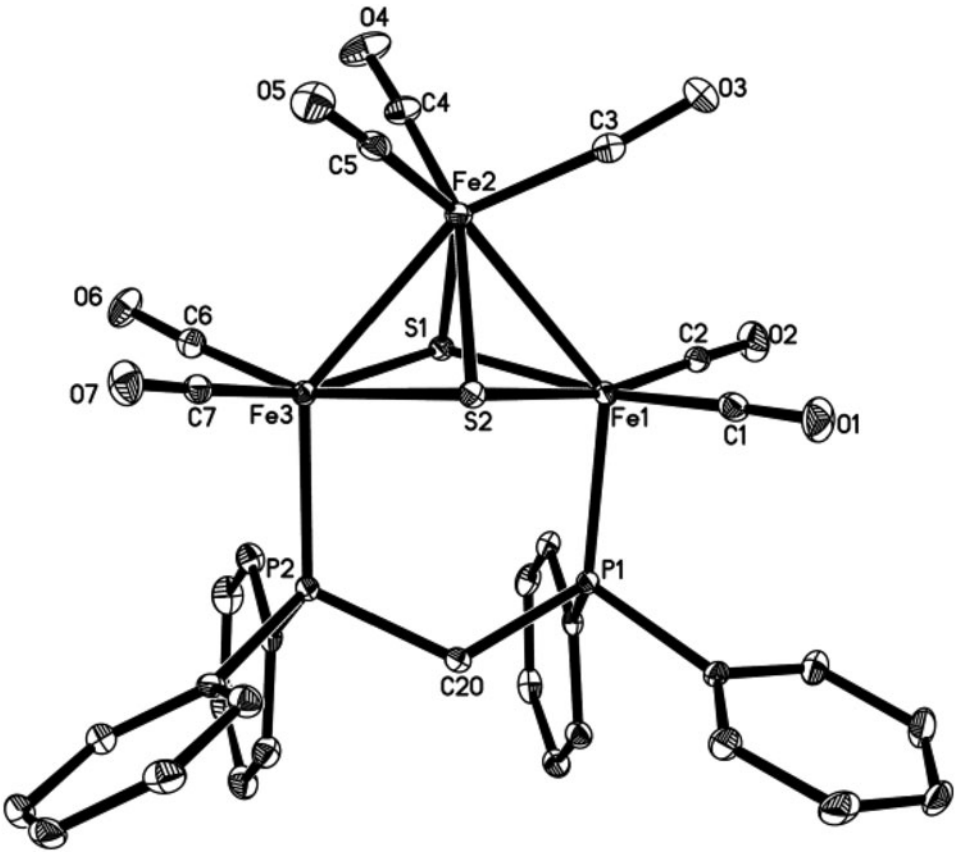

Figure 2. ORTEP view of **3** with 30% probability level ellipsoids.

Complexes **1**–**3** are air-stable black solids, which have been characterized by elemental analysis, IR and NMR spectroscopic techniques. IR spectra of **1**–**3** showed three to four absorption bands in the range of 2043−1931 cm^{-1} for their terminal carbonyls and the $\nu_{C\equiv O}$ values shifted towards lower frequencies relative to parent complex **A** (2065, 2042, 2026, 2010, and 1986 cm^{-1}) [2] because PPh_3 and $Ph_2PCH_2PPh_2$ in **1**–**3** are stronger electron-donating ligands than CO [15]. The ^{31}P NMR spectra of **1**–**3** displayed a singlet for phosphorus atoms of PPh_3 or $Ph_2PCH_2PPh_2$ coordinated to iron atoms of triiron subsite. The ^{13}C NMR spectra of **1**–**3** exhibited one or two singlets at δ 213–205 ppm for their terminal carbonyls.

3.2. X-Ray Crystal Structures

The molecular structures of **1** and **3** have been characterized by single crystal X-ray diffraction analysis. While the ORTEP views are shown in Figs. 1 and 2, selected bond lengths and angles are given in Table 2, respectively. Complex **1** crystallizes in the triclinic space group *P*-1. As shown in Fig. 1, the crystal structure consists of an open triangle of three iron atoms bicapped by two μ_3-S ligands with eight terminal carbonyls and one PPh_3. It should be noted that the phosphorus atom of the PPh_3 ligand attached to one of the non-bonding iron atoms resides in a basal position of the square-pyramidal geometry of

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the Fe3 atom, consistent with the structures of complexes $[Fe_3(\mu_3\text{-}S)_2(CO)_8(NHMe_2)]$ [1], $Fe_3(\mu_3\text{-}S)_2(CO)_8[P(SC_6H_5)Cl_2]$ [8,16], and $Fe_3(\mu_3\text{-}S)_2(CO)_8[P(SC_6H_5)_3]$ [9]. The Fe-Fe bond lengths are significantly different [Fe(1)-Fe(2) = 2.5524(8) Å and Fe(2)-Fe(3) = 2.6448(8) Å] but are close to those found in **A** [2.582(9) Å and 2.609(10) Å] [17] and $[Fe_3(\mu_3\text{-}S)_2(CO)_8(NHMe_2)]$ (2.507(2) Å and 2.645(2) Å) [1].

Complex **3** crystallizes in triclinic space group *P*-1. As shown in Fig. 2, the crystal structure consists of an open triangle of three iron atoms bicapped by two μ_3-S ligands with seven terminal carbonyls and one $Ph_2PCH_2PPh_2$. It is interesting to find out that two phosphorus atoms of $Ph_2PCH_2PPh_2$ attached to the nonbonding Fe-Fe bond (Fe1 and Fe3) resides in an apical-apical position of the square-pyramidal geometry of the Fe1 and Fe3 atoms, close to the solid-state structure of $[Fe_3(\mu_3\text{-}S)_2(CO)_7(\mu_2\text{-}Ph_2PC_5H_4FeC_5H_4PPh_2)]$ [10] but different from those found in **1** and other monosubstituted derivatives [1,8,9]. The Fe-Fe bond lengths [Fe(1)-Fe(2) = 2.6141(7) Å and Fe(2)-Fe(3) = 2.5930(7) Å] are different from **1** but are close to **A** [2.582(9) Å and 2.609(10) Å] [17]. Those differences between **1** and **3** may be due to the different substitution modes of **1** and **3**.

4. Conclusions

In summary, three triiron derivatives **1**–**3** were prepared by carbonyl substitution reactions in two different methods. Those complexes were characterized by elemental analysis, IR and NMR spectroscopy. In addition, the molecular structure of **1** and **3** were confirmed by X-ray crystallography.

Acknowledgments

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Appendix A. Supplementary Material

CCDC 917736 (**1**) and 917737 (**3**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

References

[1] Adams, R. D., & Babin, J. E. (1986). *Inorg. Chem.*, *25*, 3418.
[2] Cowie, M., Dekock, R. L., Wagenmaker, T. R., Seyferth, D., Henderson, R. S., & Gallagher, M. K. (1989). *Organometallics*, *8*, 119.
[3] Hieber, W., & Gruber, J. (1958). *Z. Anorg. Allg. Chem.*, *296*, 91.
[4] King, R. B. (1963). *Inorg. Chem.*, *2*, 326.
[5] Chaudhuri, M. K., Haas, A., & Welcman, N. (1975). *J. Organomet. Chem.*, *85*, 85.
[6] Cetini, G., Stanghellini, P. L., Rossetti, R., & Gambino, O. (1968). *J. Organomet. Chem.*, *15*, 373.
[7] Chaudhuri, M. K., Haas, A., & Welcman, N. (1975). *J. Organomet. Chem.*, *91*, 81.
[8] Liu, S. T., Liu, Q. W., Wu, B. F., Suo, Q. L., & Hu X. (1990). *Acta Phys.-Chim. Sin.*, *6*, 730.
[9] Liu, Q. W., Wu, B. F., Hu, X., Liu, S. T., & Yan, X.Y. (1992). *Acta Chim. Sin.*, *50*, 778.

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[10] Fang, Z. G., Wen, Y. S., Wang, R. K. L., Ng, S. C., Liu, L. K., & Hor, T. S. A. (1994). *J. Clust. Sci.*, *5*, 327.
[11] Wang, G. Q., Hu, X., Liu, S. T., & Liu, Q.W. (1990). *Chem. J. Chinese U.*, *11*, 121.
[12] *CRYSTALCLEAR 1.3.6.* Rigaku and Rigaku/MSC: The Woodlands, TX, 2005.
[13] Sheldrick, G. M. *SHELXS97, A Program for Crystal Structure Solution*, University of Göttingen: Germany, 1997.
[14] Sheldrick, G. M. *SHELXL97, A Program for Crystal Structure Refinement*, University of Göttingen: Germany, 1997.
[15] Liu, X. F., Xiao, X. W., & Shen, L. J. (2011). *J. Coord. Chem.*, *64*, 1023.
[16] Suo, Q. L., Liu, S. T., Hu, X., & Liu, Q.W. (1990). *Chem. J. Chinese U.*, *11*, 454.
[17] Wei, C. H., & Dahl, L. F. (1965). *Inorg. Chem.*, *4*, 493.

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