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Synthesis and Characterization of the Double Cluster Complexes of $[(\mu\text{-RS})(\mu\text{-MeAs})Fe_2(CO)_6]_2$—the Crystal and Molecular Structure of $[(\mu\text{-}^i\text{PrS})(\mu\text{-MeAs})Fe_2(CO)_6]_2$

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ABSTRACT

By the reaction of $[(\mu\text{-RS})\ (\mu\text{-CO})Fe_2\ (CO)_6]\ [Et_3NH]$ *with* $MeAsI_2$, *six new double cluster complexes,* $[(\mu\text{-RS})\ (\mu\text{-MeAs})Fe_2(CO)_6]_2$ *(R = Et, Pr,* ^{i}*Pr, Bu,* C_5H_{11}*, Ph), were synthesized. The structure of the* ^{i}*Pr analog was determined by single crystal X-ray diffraction techniques. Crystal data were as follows: monoclinic,* $P2_1/n$, $Z = 4$, $a = 14.475(4)$, $b = 13.825(3)$, $c = 17.225(5)$ Å, $\beta = 112.44(2)°$, $V = 3186$ Å^3, $R = 0.037$, *and* $R_w = 0.052$.

INTRODUCTION

Because the chemistry of single cluster complexes has become well established, there is now an escalating interest in the synthesis and characterization of double cluster complexes [1–7]. In a previous article [8], we reported an unexpected reaction of the complex salt $[(\mu\text{-}^t\text{BuS})\ (\mu\text{-CO})Fe_2\ (CO)_6]$ $[Et_3NH]$ with $MeAsI_2$ to give a novel double cluster complex $[(\mu\text{-}^t\text{BuS})\ (\mu\text{-MeAs})Fe_2\ (CO)_6]_2$. In order to know whether this is a new, general reaction, we decided to investigate it using complex salts containing other thiolato ligands $[(\mu\text{-RS})\ (\mu\text{-CO})Fe_2\ (CO)_6]\ [Et_3NH]$ (R = Et, Pr, ^{i}Pr, Bu, C_5H_{11}, Ph) (**1**) instead of $[(\mu\text{-}^t\text{BuS})\ (\mu\text{-CO})Fe_2\ (CO)_6]\ [Et_3NH]$. Herein we report the results of this study.

RESULTS AND DISCUSSION

Preparative and Structural Assignment

The complex salts $[(\mu\text{-RS})\ (\mu\text{-CO})Fe_2\ (CO)_6]\ [Et_3NH]$ (**1**) may be prepared from $Fe_3(CO)_{12}$, RSH, and Et_3N in THF. They have been found to have a variety of interesting reactions [9–18]. The experiments reported in this article show that $MeAsI_2$ reacts with (**1**), where R = Et, Pr, ^{i}Pr, Bu, C_5H_{11}, Ph, to give the anticipated double cluster complexes of the type $[(\mu\text{-RS})\ (\mu\text{-MeAs})Fe_2\ (CO)_6]_2$ (**2**) (Scheme 1), thus, to some extent, demonstrating the generality of this new reaction for the formation of this type of double cluster complex. Complexes **2a–2f** are dark-red solids and soluble in common solvents such as petroleum ether, diethyl ether, and methylene chloride. The C/H analytical data, IR, and ^{1}H NMR data of **2a–2f** are in good agreement with the structure **2** shown in Scheme 1. Although we could not obtain the parent molecular ion peak from MS determinations, we obtained the fragment ion peaks corresponding to loss of a given number of CO ligands from the parent ion and the fragment peaks of the double cluster $Fe_4As_2S_2$ and the single cluster core Fe_2AsS, a fragment of As—As bond cleavage from the double cluster core.

Dedicated to Professor Yao-Zeng Huang on the occasion of his eightieth birthday.

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Complexes	2a	2b	2c	2d	2e	2f
R	Et	Pr	^{i}Pr	Bu	C_5H_{11}	Ph

SCHEME 1

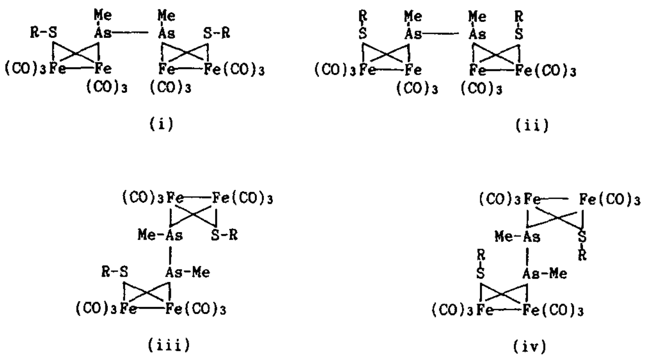

FIGURE 1 The possible isomers for complex **2**.

The IR spectra of **2a–2f** exhibited three to four very strong bands in a region between 1960 and 2060 cm^{-1}, indicating the presence of terminal CO ligands attached to the iron atoms.

In the ^{1}H NMR spectra, we observe all the signals of the organic groups attached to the arsenic and sulfur atoms in complexes **2a–2f**. In general, these complexes each may exist as a mixture of several stereoisomers, in terms of the reasoning applied to (μ-RS) (μ-R′S)$Fe_2(CO)_6$ [19]. It is fortunate that the ^{1}H NMR spectra of these six complexes show only one kind of peak corresponding to methyl on the arsenic atom. This means that the alkyl groups on two sulfur atoms in each complex are bound to sulfur atoms via the same type of bond, *a*-type or *e*-type [20]. Otherwise, the methyl groups on the two arsenic atoms would have different chemical shifts due to the influence of the different conformations of the alkyl groups on adjacent sulfur atoms. Therefore, the possible isomers for these complexes are (i)–(iv).

In (i) and (ii), the methyl groups are linked to arsenic by an axial type of bond (abbreviated as *a* hereafter); thus, the two arsenic atoms of the subcluster core are linked together by an equatorial type of bond (abbreviated as *e* hereafter). Nevertheless, in (i), the alkyl groups are attached to the two sulfur atoms by an *e*-bond, while in (ii) they are attached by an *a*-bond. Similarly to (i) and (ii), the alkyl groups in (iii) are attached to the sulfur atoms by an *e*-bond, whereas in (iv), they are attached by an *a*-bond. Again, from the same reasoning applied to (μ-RS) (μ-R′S)$Fe_2(CO)_6$ [9], conformers (ii) and (iv) might not actually be present due to steric effects [21].

Thus, the question left is: are both (i) and (iii) present together or is only one isomer (i) or (iii) present? In order to answer this question, we have determined the crystal and molecular structure of one representative, **2c**, of these six complexes by X-ray diffraction.

Crystal Structure

The structural determination indicated that the complex has the configuration of (iii) (R = ^{i}Pr). In view of having the same configuration for the ^{t}Bu analog, we may conclude that conformer (iii) is probably the most stable for this kind of double cluster complex.

The structure of **2c** is shown in Fig. 2. The final fractional coordinates with equivalent isotropic thermal parameters are available from us on request. The selected interatomic distances and angles are listed in Tables 1 and 2.

From Fig. 2, it can be seen that this molecular structure is totally similar to that of [(μ-^{t}BuS) (μ-MeAs)Fe_2 $(CO)_6]_2$ [8]. This molecule consists of two butterfly-shaped subclusters, (μ-^{i}Pr-S)(μ-MeAs)$Fe_2(CO)_6$, dimerized through an As—As bond. The angles of As(1) ... S(1)—C(51) = 162.6°, As(2) ... S(2)—C(61) = 163.5°, S(1) ... As(1)—C(1) = 168.8°, and S(2) ... As(2)—C(2) = 170.8° show that the two methyl groups and the two ^{i}Pr groups are connected with two arsenic atoms and two sulfur atoms all by an *e*-bond. The bond length of As(1)—As(2) is 2.4346(8) Å, which is almost the same as that for the ^{t}Bu analog [8] and very close to the normal As—As single bond length (*ca.* 2.4 Å) [22]. The average length of the two Fe—Fe bonds is 2.585 Å, and the average bond length of the four Fe—S

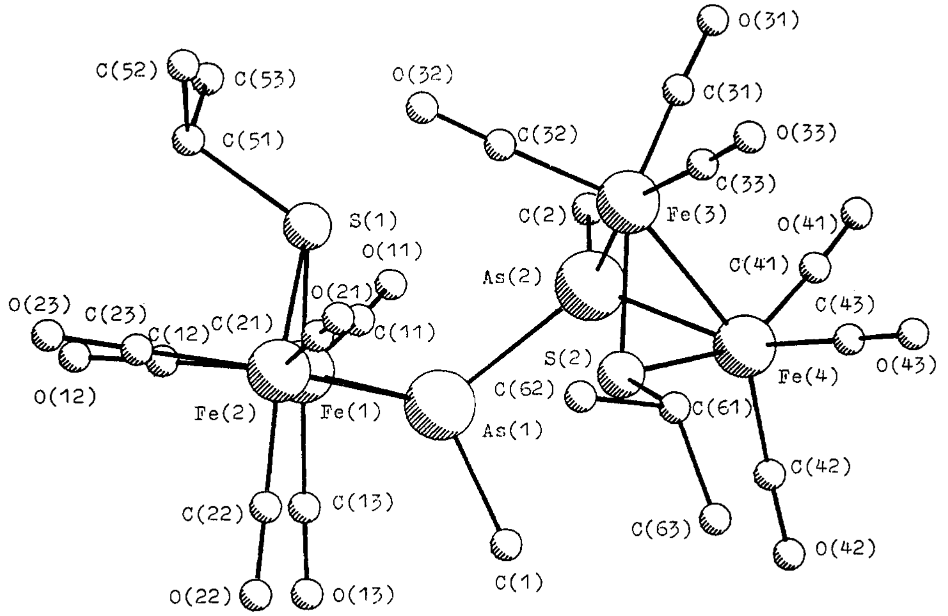

FIGURE 2 A perspective view of complex **2c**.

and four Fe—As bonds are 2.266 and 2.336 Å, respectively. These bond lengths are quite close to those observed in a similar monocluster complex $(\mu\text{-}^{i}\text{PrS})$ $(\mu\text{-Ph}_2\text{As})\text{Fe}_2(\text{CO})_6$ in which the bond lengths are 2.626 Å for Fe—Fe, 2.264 Å for average Fe—S, and 2.327 Å for average Fe—As bonds [18]. Obviously, the bond lengths for complex **2c** (R = ^{i}Pr) are actually the same as the corresponding bond lengths of the ^{t}Bu analog, 2.591 Å for Fe—Fe, 2.267 Å for Fe—S, and 2.339 Å for Fe—As [8].

In addition, as in the case of the ^{t}Bu analog (2.810 Å), the average distance between As and S in complex **2c** (2.849 Å) is shorter than the S . . . S distance in $(\mu\text{-CH}_3\text{S})$ $(\mu\text{-CH}_3\text{HgS})\text{Fe}_2(\text{CO})_6$ (2.886 Å) [21] and shorter than the As . . . S distance in the monocluster complex $(\mu\text{-}^{i}\text{PrS})$ $(\mu\text{-Ph}_2\text{As})\text{Fe}_2(\text{CO})_6$ (2.872 Å) [18].

EXPERIMENTAL

The reactions were carried out under an atmosphere of a highly purified tank nitrogen. Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl and purged with nitrogen prior to use. Triethylamine and mercaptans were deoxygenated by purging with nitrogen for 15 minutes. Dodecacarbonyl triiron [23] and methyldiiodoarsine [24] were prepared by literature procedures. Column chromatographic silica gel was about 300 mesh. GF 254 was used for TLC separation. Infrared spectra were obtained with a SP3-300 model infrared spectrophotometer. Proton NMR spectra were recorded on a JNMPMX 60 SI spectrometer. Elemental analysis was performed by use of a 240C Model analyzer. Melting points were determined on a PHMK-3003 melting point apparatus and are uncorrected. Mass spectra were obtained with an HP 5988A spectrometer operating at 70 *e*V.

Synthesis of $[(\mu\text{-}RS)\ (\mu\text{-}MeAs)Fe_2\ (CO)_6]_2$ **2a–2f**

A 250 ml two-necked flask equipped with a magnetic stir-bar and a rubber septum was flame-dried and then charged with 1.5 g (2.98 mmol) of $\text{Fe}_3(\text{CO})_{12}$ and 50 mL of THF. 3.38 mmol of mercaptans (RSH) and 0.5 mL (3.58 mmol) of Et_3N were added to the resulting green solution. After about 15 minutes, the solution became red-brown. Then 1.54 g (4.5 mmol) of MeAsI_2 was added. The solution turned red rapidly and was stirred overnight at room temperature. The solvent was removed and the residue was subjected to column chromatography (9:1 petroleum ether:CH_2Cl_2) to give a red solution, which was further separated by TLC (support: GF 254, eluent: 9:1 petroleum ether:CH_2Cl_2) to give the products.

2a (from EtSH): dark-red solid, yield 0.3 g (16%), mp 190–192°C. Anal calcd for $\text{C}_{18}\text{H}_{16}\text{As}_2\text{Fe}_4\text{O}_{12}\text{S}_2$: C, 25.09; H, 1.87%. Found: C, 24.86; H, 1.57%. IR (KBr disc): terminal C≡O, 1975(vs), 2024(vs), 2049(vs) cm^{-1}. ^{1}H NMR (CDCl_3): δ 1.43 (6H, t, J = 7.0 Hz, 2[$\text{C}H_3\text{CH}_2\text{S}$]), 2.17 (6H, s, 2 [$\text{CH}_3\text{As}$]), 2.60 (4H, q, J = 7.0 Hz 2[$\text{CH}_3\text{C}H_2\text{S}$]). Mass spectrum (EI), m/z (relative intensity): 805 (M^+—2CO, 2%), 777 (M^+—3CO, 0.7), 749 (M^+—4CO, 11.6), 721 (M^+—5CO, 1.6), 693 (M^+—6CO, 4.3), 665 (M^+—7CO, 6.6),

Table 1 Selected Bond Distances (Å) for **2c**

As(1)	As(2)	2.4346(8)	Fe(2)	C(22)	1.787(6)
As(1)	Fe(1)	2.3321(9)	Fe(2)	C(23)	1.797(6)
As(1)	Fe(2)	2.339(1)	Fe(3)	Fe(4)	2.578(1)
As(1)	C(1)	1.980(6)	Fe(3)	S(2)	2.271(2)
As(2)	Fe(3)	2.3322(9)	Fe(3)	C(31)	1.795(7)
As(2)	Fe(4)	2.3406(9)	Fe(3)	C(32)	1.826(7)
As(2)	C(2)	1.973(5)	Fe(3)	C(33)	1.796(7)
Fe(1)	Fe(2)	2.592(1)	Fe(4)	S(2)	2.266(2)
Fe(1)	S(1)	2.263(2)	Fe(4)	C(41)	1.791(7)
Fe(1)	C(11)	1.787(7)	Fe(4)	C(42)	1.813(7)
Fe(1)	C(12)	1.802(7)	Fe(4)	C(43)	1.792(6)
Fe(1)	C(13)	1.797(8)	S(1)	C(51)	1.864(6)
Fe(2)	S(1)	2.265(2)	S(2)	C(61)	1.861(5)
Fe(2)	C(21)	1.815(7)			

Table 2 Selected Bond Angles (°) for **2c**

As(2)	As(1)	Fe(1)	120.09(3)	As(1)	Fe(2)	Fe(1)	56.18(3)
As(2)	As(1)	Fe(2)	122.05(3)	As(1)	Fe(2)	S(1)	76.26(4)
As(2)	As(1)	C(1)	103.2(2)	Fe(1)	Fe(2)	S(1)	55.04(4)
Fe(1)	As(1)	Fe(2)	67.42(3)	As(2)	Fe(3)	Fe(4)	56.67(3)
Fe(1)	As(1)	C(1)	120.9(2)	As(2)	Fe(3)	S(2)	76.64(4)
Fe(2)	As(1)	C(1)	121.3(2)	Fe(4)	Fe(3)	S(2)	55.28(4)
As(1)	As(2)	Fe(3)	119.70(3)	As(2)	Fe(4)	Fe(3)	56.36(3)
As(1)	As(2)	Fe(4)	118.20(3)	As(2)	Fe(4)	S(2)	76.58(4)
As(1)	As(2)	C(2)	104.2(2)	Fe(3)	Fe(4)	S(2)	55.47(4)
Fe(3)	As(2)	Fe(4)	66.96(4)	Fe(1)	S(1)	Fe(2)	69.84(5)
Fe(3)	As(2)	C(2)	122.1(2)	Fe(1)	S(1)	C(51)	112.7(2)
Fe(4)	As(2)	C(2)	123.3(2)	Fe(2)	S(1)	C(51)	115.7(2)
As(1)	Fe(1)	Fe(2)	56.40(3)	Fe(3)	S(2)	Fe(4)	69.25(5)
As(1)	Fe(1)	S(1)	76.43(4)	Fe(3)	S(2)	C(61)	115.6(2)
Fe(2)	Fe(1)	S(1)	55.13(4)	Fe(4)	S(2)	C(61)	114.3(3)

637 (M^+—8CO, 0.7), 609 (M^+—9CO, 10.1), 553 (M^+—11CO, 0.7), 525 (M^+—12CO, 0.9), 437 ($Fe_4As_2S_2$, 100).

2b (from PrSH): dark-red solid, yield 0.2 g (15%), mp 133–135°C. Anal calcd for $C_{20}H_{20}As_2Fe_4O_{12}S_2$: C, 27.00; H, 2.27%. Found: C, 26.70; H, 2.00%. IR (KBr disc): terminal $C{\equiv}O$, 1975(vs), 2010(vs), 2049(vs) cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.13 (6H, t, J = 6.8 Hz, $2[CH_3CH_2CH_2S]$), 1.47–2.03 (4H, m, $2[CH_3CH_2CH_2S]$), 2.17 (6H, s, $2[CH_3As]$), 2.53 (4H, t, J = 6.8 Hz, $2[CH_3CH_2CH_2S]$). Mass spectrum (EI), m/z (relative intensity): 833 (M^+—2CO, 1%), 805 (M^+—3CO, 4), 777 (M^+—4CO, 2), 749 (M^+—5CO, 2), 721 (M^+—6CO, 1), 693 (M^+—7CO, 15), 665 (M^+—8CO, 4), 637 (M^+—9CO, 8), 609 (M^+—10CO, 7), 581 (M^+—11CO, 1) 553 (M^+—12CO, 13), 437 ($Fe_4As_2S_2$, 100).

2c (from ^{i}PrSH): dark-red solid, yield 0.3 g (23%), mp 188–190°C. Anal calcd for $C_{20}H_{20}As_2Fe_4O_{12}S_2$: C, 27.00; H, 2.27%. Found: C, 27.02; H, 2.19%. IR (KBr disc): terminal $C{\equiv}O$, 1967(vs), 2024(vs), 2049(vs) cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.45 (12H, d, J = 6.6 Hz, $2[(CH_3)_2CHS]$), 2.17 (6H, s, $2[CH_3As]$), 2.33–3.00 (2H, m, $2[(CH_3)_2CHS]$). Mass spectrum (EI), m/z (relative intensity): 805 (M^+—3CO, 4%), 777 (M^+—4CO, 1), 749 (M^+—5CO, 1), 721 (M^+—6CO, 1), 693 (M^+—7CO, 18), 665 (M^+—8CO, 5), 637 (M^+—9CO, 9), 609 (M^+—10CO, 7), 581 (M^+—11CO, 1), 553 (M^+—12CO, 14), 437 ($Fe_4As_2S_2$, 100), 219 (Fe_2AsS, 1).

2d (from BuSH): dark-red solid, yield 0.3 g (22%), mp 139–141°C. Anal calcd for $C_{22}H_{24}As_2Fe_4O_{12}S_2$: C, 28.79; H, 2.64%. Found: C, 28.79; H, 2.48%. IR (KBr disc): terminal $C{\equiv}O$, 1967(vs), 2016(vs), 2032(vs), 2049(vs) cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.97 (6H, t, J = 6.9 Hz, $2[CH_3CH_2CH_2CH_2S]$), 1.20–2.00 (8H, m, $2[CH_3CH_2CH_2CH_2S]$), 2.17 (6H, s, $2[CH_3As]$), 2.53 (4H, t, J = 7.0 Hz, $2[CH_3CH_2CH_2CH_2S]$). Mass spectrum (EI), m/z (relative intensity): 833 (M^+—3CO, 3%), 805 (M^+—4CO, 2), 777 (M^+—5CO, 2), 749 (M^+—6CO, 1), 721 (M^+—7CO, 13), 693 (M^+—8CO, 3), 665 (M^+—9CO, 10), 637 (M^+—10CO, 7), 609 (M^+—11CO, 2), 581 (M^+—12CO, 12), 437 ($Fe_4As_2S_2$, 100), 219 Fe_2AsS, 1).

2e (from $C_5H_{11}SH$): dark-red solid, yield 0.2 g (14%), mp 120–2°C. Anal calcd for $C_{24}H_{28}As_2Fe_4O_{12}S_2$: C, 30.48; H, 2.98%. Found: C, 30.37; H, 2.98%. IR (KBr disc): terminal $C{\equiv}O$, 1967(vs), 2008(vs), 2032(vs), 2049(vs) cm^{-1}. ^1H NMR ($CDC13$): δ 0.98 (6H, t, J = 6.9 Hz, $2[\mathit{CH_3}\mathrm{CH_2CH_2CH_2CH_2S}]$), 1.17–2.07 (12H, m, $2[\mathrm{CH_3}\mathit{CH_2CH_2CH_2}\mathrm{CH_2S}]$), 2.17 (6H, s, $2[CH_3As]$), 2.57 (4H, t, J = 6.9 Hz, $2[\mathrm{CH_3CH_2CH_2CH_2}\mathit{CH_2}\mathrm{S}]$). Mass spectrum (EI), m/z (relative intensity): 861 (M^+—3CO, 3%), 833 (M^+—4CO, 1), 805 (M^+—5CO, 2), 777 (M^+—6CO, 1), 749 (M^+—7CO, 12), 721 (M^+—8CO, 2), 693 (M^+—9CO, 4), 665 (M^+—10CO, 7), 637 (M^+—11CO, 1), 609 (M^+—12CO, 9), 437 ($Fe_4As_2S_2$, 100), 219 (Fe_2AsS, 1).

2f (from PhSH): dark-red solid, yield 0.1 g (7%), mp 174°C (dec.). Anal calcd for $C_{26}H_{16}As_2Fe_4O_{12}S_2$: C, 32.60; H, 1.68%. Found C, 32.97; H, 1.69%. IR (KBr disc): terminal $C{\equiv}O$, 1959(vs), 2024(vs), 2057(vs) cm^{-1}. ^1H NMR ($CDCl_3$): δ 2.26 (6H, s, $2[CH_3As]$), 7.25 (10H, s, $2[C_6H_5S]$). Mass spectrum (EI), m/z (relative intensity): 706 (M^+—9CO, 7.2%), 622 (M^+—12CO, 6.8), 437 ($As_2Fe_4S_2$, 100), 219 ($AsFe_2S$, 7.1).

Determination of X-Ray Crystal Structure

A solution of **2c** dissolved in 7:3 petroleum ether:CH_2Cl_2 was slowly evaporated at room temperature to give a dark red-single crystal suitable for X-ray diffraction determination. The crystal with approximate dimensions $0.2 \times 0.2 \times 0.3$ mm^3 was mounted on glass fiber in an arbitrary orientation. Preliminary examination and data collection were performed with Mo-K_α radiation ($\lambda = 0.71073$ Å) on an Enraf-Nonius CAD4 diffractometer equipped with a graphite monochromator. A total of 4811 independent reflections was collected in the range of $2° \leq \theta \leq 23°$ by ω-2θ scan technique at room temperature (23 ± 1°C), of which 2905 reflections with $I \geq 3\sigma(I)$ were considered to be observed and used for subsequent refinement. The corrections for Lp factors and empirical absorption were applied to intensity data.

The crystal is monoclinic, space group $P2_1/n$ with $a = 14.475(4)$, $b = 13.825(3)$, $c = 17.225(5)$ Å, $\beta = 112.44(2)°$, $V = 3186$ Å^3, $Mr = 889.74$, $Z = 4$, $Dx = 1.86$ g/cm^3, $\mu = 40.2$ cm^{-1}, $F(000) = 1752$.

The structure was solved by a direct phase determination method (MULTAN 82). From E-map, four Fe atoms and two As atoms were located. The other nonhydrogen atoms were found by successive difference Fourier syntheses. The hydrogen atoms were not included in the refinement and calculations of structure factors. The final refinement by full-matrix least-squares method with anisotropic thermal parameters for nonhydrogen atoms converged to unweighted and weighted agreement factors (R and R_w) of 0.037 and 0.052. The highest peak on the final difference Fourier map had a height of 0.63 e/Å^3. All calculations were performed on a PDP 11/44 computer using the SDP-PLUS program system.

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