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Synthesis and X-Ray Crystal Structures of Hypervalent Arsenic Compounds Bearing Arsenic-Iron Bonds

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ABSTRACT: *A hypervalent arsorane with an As–Fe bond [***6***: $Rf_2As^*FeCp(CO)_2$ (Rf: o-$C_6H_4C(CF_3)_2O$-)] was synthesized by the reaction of the arsoranide anion (***4-Et$_4$N***: $Rf_2As^{*-}Et_4N^+$) with $CpFe(CO)_2I$ (***5***) in the presence of $AgBF_4$. Diastereomeric arsoranes {***7a*** and ***7b***: $Rf_2As^*Fe^*Cp(CO)(PPh_3)$} were prepared by irradiation of* **6** *with a tungsten lamp in the presence of triphenylphosphine. X-ray crystallographic analysis of* **6** *and* **7a** *showed that the apical As–O bond lengths of these compounds are slightly longer than the equatorial As–C bonds. It is concluded that an electron-donating group at the equatorial position elongates the apical E–O bond by electron donation to the apical oxygens.* © 2000 John Wiley & Sons, Inc. Heteroatom Chem 11:42–47, 2000

INTRODUCTION

In 1991, Martin reported a stable phosphorus hypervalent compound with a phosphorus-iron bond, $Rf_2P^*FeCp(CO)_2$ (**1**) (Rf: *o*-$C_6H_4C(CF_3)_2O$-) [1]. Recently, we prepared several corresponding antimony compounds, $Rf_2Sb^*FeCp(CO)_2$ (**2**) and $Rf_2Sb^*Fe^*Cp(CO)(PPh_3)$ (**3a** and **3b**), and investigated the energy barrier of pseudorotation about the antimony atom [2]. In this article we report on the synthesis and X-ray crystal structures of corresponding arsenic compounds and discuss the periodical trend of apical and equatorial bond lengths among the group 15 element compounds.

RESULTS AND DISCUSSION

Tetraethylammonium bis[3,3-bis(trifluoromethyl)-benzenemethanolato(2-)*C*^{2},*O*] arsoranide anion (**4-Et$_4$N**) was prepared from lithium 1,1,1,3,3,3-hexafluoro-2-(2-lithiophenyl)-2-propoxide [3] as outlined in Scheme 1. The reaction of cyclopentadienylbis(carbonyl)iodo iron (**5**) [4] in the presence of silver tetrafluoroborate [5] with **4-Et$_4$N** gave **6** in good yield (72%).

Photoirradiation of **6** in the presence of an equimolar amount of PPh_3 gave a mixture of diastereomers, **7a** and **7b**, in 10% and 61% yields, respectively (Scheme 2). Separation of **7a** and **7b** was easily accomplished by TLC (SiO_2/AcOEt:*n*-hexane = 1:5). No bis(triphenylphosphine) adduct was obtained. Compounds **6**, **7a**, and **7b** were stable to atmospheric moisture, and assignment of each compound was performed by NMR (^1H and ^{19}F), elemental analysis, and X-ray analysis.

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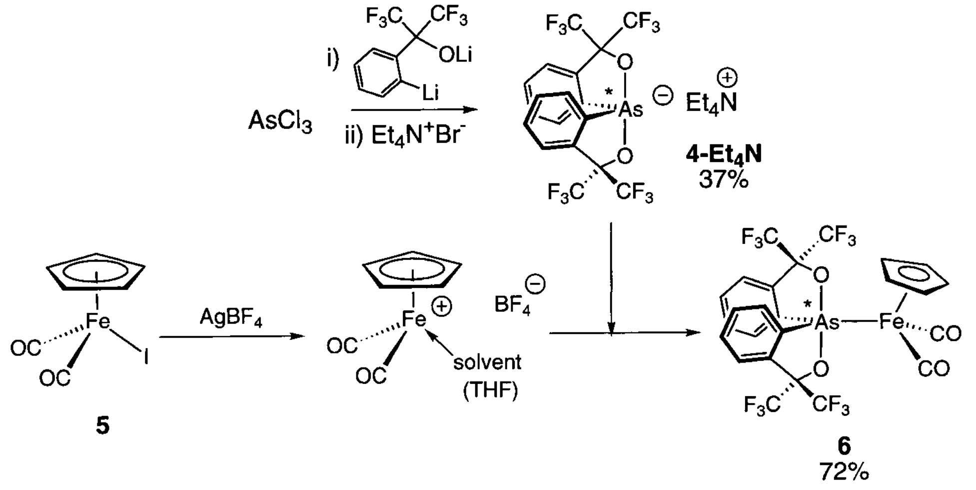
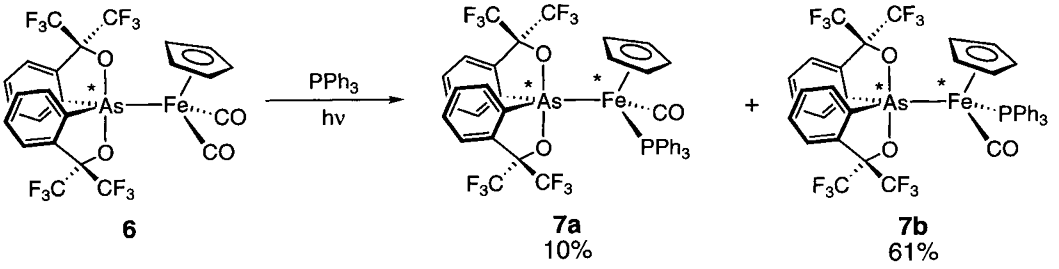


SCHEME 1



SCHEME 2

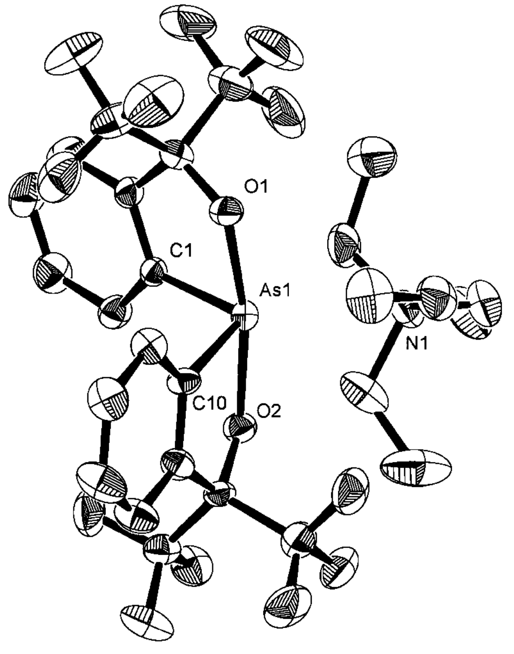


FIGURE 1 ORTEP drawing of **4-Et$_4$N**. The atoms are represented as 30% probability displacement ellipsoids. Hydrogen atoms are omitted for clarity.

X-Ray Crystal Structures of 4-Et$_4$N, 6, *and* 7a

Crystals of **4-Et$_4$N**, **6**, and **7a** suitable for X-ray analysis were obtained by recrystallization from acetone-*n*-heptane for **4-Et$_4$N**, and from benzene-*n*-hexane for **6** and **7a**. Figures 1–3 show the crystal structures of **4-Et$_4$N**, **6**, and **7a**. Selected bond lengths and bond angles for the structures of **6** (two independent molecules) and **7a** are listed in Table 1 in addition to the corresponding phosphorus and antimony compounds, $Rf_2P^*FeCp(CO)_2$ (**1**) [1a], $Rf_2Sb^*FeCp(CO)_2$ (**2**) (two independent molecules) [2], and $Rf_2Sb^*Fe^*Cp(CO)(PPh_3)$ (**3a**) [2] for comparison. The geometry about arsenic in **6** and **7a** is almost the same and can be considered as a distorted trigonal bipyramid (TBP) with the iron atom at the equatorial site of the TBP. Consistent with antimony compounds (**2** and **3a**) [2], the averaged apical As–O bond [1.991(4) Å] with the electron-donating equatorial substituent $Fe^*Cp(CO)(PPh_3)$ in **7a** is elongated in comparison with that in **6** [1.969(4) Å] with $FeCp(CO)_2$. The apical O–As–O bond angle [162.8(2)°] in **7a** is smaller than those of **6** [166.7(2)° and 166.8(2)°], and the As–Fe bond length [2.395(1)

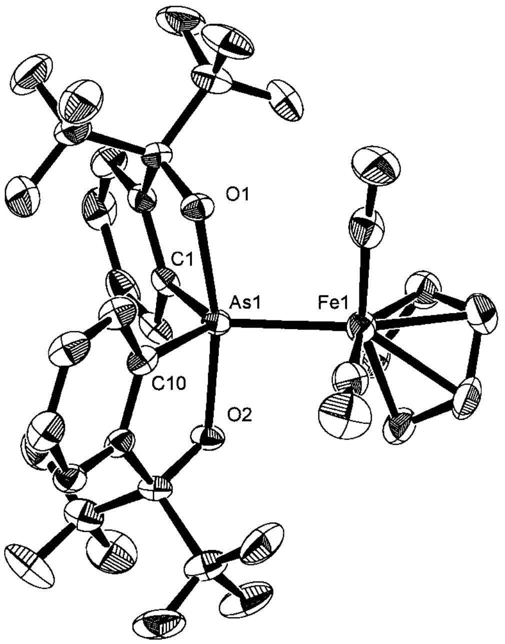


FIGURE 2 ORTEP drawing of **6** (one of the independent molecules). The atoms are represented as 30% probability displacement ellipsoids. Hydrogen atoms are omitted for clarity.

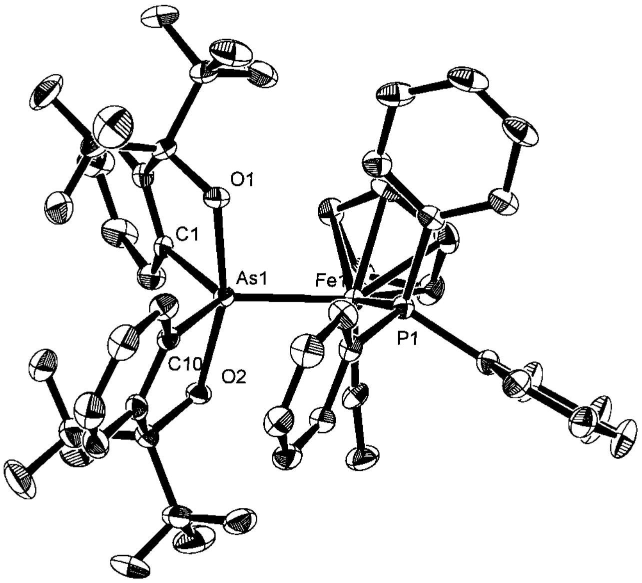


FIGURE 3 ORTEP drawing of **7a**. The atoms are represented as 30% probability displacement ellipsoids. Hydrogen atoms and the solvent (benzene) are omitted for clarity.

Å] in **7a** is longer than those of **6** [2.362(1), 2.353(1) Å].

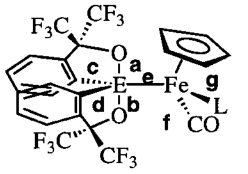
It is interesting to note that the X-ray crystallographic analysis of **6** and **7a** shows that the averaged apical As–O bond length [1.969(4) Å in **6**, 1.991(4) Å in **7a**] of these compounds is slightly longer than the averaged equatorial As–C bond lengths [1.938(7) Å in **6**, 1.949(6) Å in **7a**] since one should expect the As–O bond to be shorter than the As–C(Ph) bond. The latter is the case even in an arsorane, that is, 2-phenyl-2,2′-spirobi(4,4-dimethyl-5-oxo-1,3,2-dioxa-λ^5-arsorane, (Me$_2$C$_2$O$_3$)$_2$AsPh. The As–O bond lengths of the arsorane [apical As–O 1.864(2) Å, equatorial As–O 1.743(1) Å] are shorter than the As–C(Ph) bond length [1.905(3) Å] [6]. This is also true for other examples in Table 1: the averaged apical E–O bond (E = P or Sb) lengths {[1.824(3) Å in **1** [1a], 2.087(4) Å in **2** [2], and 2.114(2) Å in **3a** [2]} of the corresponding phosphorus and antimony analog are slightly shorter than the averaged equatorial E-C bond lengths [1.841(6) Å in **1** [1a], 2.110(3) Å in **2** [2], and 2.116(4) Å in **3a** [2]]. The reason for the irregular trend for the hypervalent arsenic compounds is not clear, but it may be related to the predicted instability of the hypervalent arsenic compounds due to the d-block contraction caused by an increase in the effective nuclear charge for the 4s electrons due to the filling of the first d shell [7].

In order to elucidate the irregular trend, selected bond lengths and bond angles for **4-Et$_4$N** are also listed in Table 2 in addition to those of the corresponding phosphorus, antimony, and bismuth compounds (Nakamoto and Akiba, unpublished results, [8], [9]) for comparison. Although the trend is not obvious, the averaged As–O bond length in **4-Et$_4$N** [2.038(4) Å] is longer by 3.9% than the averaged As–C bond length [1.961(6) Å]. The ratio of the elongation (3.9%) is larger in **4-Et$_4$N** than that in **8-K(18-crown-6)** (2.4%) and **10-Et$_4$N** (2.1%). The E–O bond lengths of all the anions in Table 2 are longer than those of corresponding compounds in Table 1. This shows that the electron is donated to the apical oxygens and the E–O bond is elongated. This is also the case for the pairs of **6** and **7a** and **2** and **3a** in Table 1 to show that PPh$_3$ is more electron-donating than CO.

EXPERIMENTAL

Melting points were taken on a Yanagimoto micro melting point apparatus and are uncorrected. ^{1}H NMR (400 MHz) and ^{19}F NMR (376 MHz) were recorded on a JEOL EX-400 spectrometer. Chemical shifts are reported (δ scale) from internal tetramethylsilane for ^{1}H or from fluorotrichloromethane for ^{19}F. IR spectra were recorded on a Shimadzu 460 spectrometer. Elemental analysis was performed on a Perkin-Elmer 2400CHN elemental analyzer. Column chromatography was carried out on Merck silica gel 9385. Thin-layer chromatography was per-

TABLE 1 A Comparison of Bond Lengths (Å) and Bond Angles (°) in **6** and **7a** with those in Corresponding Phosphorus (1) and Antimony Compounds (**2** and **3a**)



E= P, L=CO, 1
E=As, L=CO, 6
E=Sb, L=CO, 2
E=As, L=PPh$_3$, 7a
E=Sb, L=PPh$_3$, 3a

	1[1a]	**6**		**2**[2]		**7a**	**3a**[2]
				Bond Lengths (Å)			
a	1.819(3)	1.974(4)	1.966(4)	2.091(3)	2.093(3)	1.999(4)	2.122(2)
b	1.829(3)	1.966(4)	1.971(4)	2.075(3)	2.087(3)	1.984(4)	2.105(2)
c	1.839(6)	1.943(6)	1.940(7)	2.116(3)	2.110(3)	1.945(6)	2.118(3)
d	1.843(5)	1.930(7)	1.939(7)	2.104(3)	2.110(3)	1.952(6)	2.114(4)
e	2.300(2)	2.362(1)	2.353(1)	2.4801(5)	2.4790(5)	2.395(1)	2.5106(6)
f	–	1.788(9)	1.77(1)	1.776(6)	1.768(6)	1.751(7)	1.749(4)
g	–	1.753(9)	1.78(1)	1.772(4)	1.751(4)	2.240(2)	2.230(1)
				Bond Angles (°)			
ab	170.3(2)	166.8(2)	166.7(2)	161.3(1)	163.69(9)	162.8(2)	157.4(1)
cd	119.6(2)	117.0(3)	117.3(3)	115.9(1)	111.1(1)	115.1(3)	112.5(2)
ac	85.4(2)	82.6(2)	83.1(2)	79.3(1)	78.9(1)	81.2(2)	77.9(1)
bd	–	83.3(2)	83.3(2)	79.7(1)	79.4(1)	82.6(2)	78.7(1)
ad	–	89.2(2)	90.5(2)	90.3(1)	90.4(1)	88.4(2)	88.5(1)
bc	–	91.1(2)	89.4(2)	90.8(1)	92.8(1)	89.3(2)	89.8(1)
ce	–	122.7(2)	125.2(2)	118.95(9)	124.16(8)	130.7(2)	133.2(1)
de	121.9(2)	120.3(2)	117.5(2)	125.10(9)	124.56(9)	114.2(2)	114.2(1)
ae	–	98.5(1)	98.1(2)	97.44(7)	96.42(7)	100.4(1)	103.75(8)
be	–	94.6(1)	95.2(1)	101.24(7)	99.85(7)	96.6(1)	98.52(8)

formed with Merck silica gel GF-254 plates. All reactions were carried out under N_2 or Ar. The preparation of cyclopentadienylbis(carbonyl)iodo iron (**5**) followed published procedures [4]. Tetrahydrofuran (THF) and diethyl ether were distilled from sodium-benzophenone.

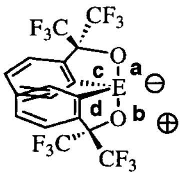
Preparation of **4-Et$_4$N**$[Rf_2As^{*-}Et_4N^+$ $(Rf\text{: }o\text{-}C_6H_4C(CF_3)_2O)]$

Lithium 1,1,1,3,3,3-hexafluoro-2-(2-lithiophenyl)-2-propoxide [3] was prepared from bis(trifluoromethyl)benzyl alcohol (5.50 mL, 35.6 mmol), *n*-BuLi (72.2 mmol in 46 mL of hexane), and *N,N,N′,N′*-tetramethylethylenediamine (TMEDA, 1.10 mL, 7.29 mmol) in 10 mL of THF and was dissolved in 80 mL of THF followed by addition to a cold (−78°C) stirred solution of $AsCl_3$ (1.0 mL, 2.15 g, 11.9 mmol) in 50 mL of THF. The mixture was allowed to warm to room temperature and was stirred for a further 12 hours. After the reaction was treated with water, the crude product was extracted with ethyl acetate (50 mL × 3) and washed with aqueous NaCl solution (100 mL). The crude **4-Na** (2.96 g, 5.08 mmol) in 15 mL of ethyl acetate was treated with tetraethylammonium bromide (3.29 g, 15.5 mmol), and the crude **4-Et$_4$N** was recrystallized from acetone-*n*-heptane to give colorless crystals (0.98 g, yield 37%); m.p. 183–184 °C (dec). ^{1}H NMR (acetone-d$_6$) 1.34 (tt, 12 H, J = 7, 2 Hz), 3.44 (q, 8 H, J = 7 Hz), 7.15–7.27 (m, 4 H), 7.14 (bd, 2 H, J = 7 Hz), 8.40 (bd, 2 H, J = 7 Hz). ^{19}F NMR (acetone-d$_6$) −73.2 (q, 6 F, J = 9 Hz), −75.8 (q, 6 F, J = 9 Hz). Anal. Calcd for $C_{26}H_{28}F_{12}NO_2As$: C, 45.30; H, 2.03. Found: C, 45.14; H, 2.14.

{Bis[3,3-bis(trifluoromethyl)benzene-methanolato(2-)C^2,O]arsoranide}dicarbonyl (cyclopentadienyl)iron(II) (**6**)

A mixture of dicarbonyl(cyclopentadienyl)iodo iron **5** (0.329 g, 1.08 mmol) and silver tetrafluoroborate (0.280 g, 1.44 mmol) in 4.5 mL of dry THF was stirred for 3 hours at room temperature. To the suspension, tetraethylammonium arsoranide (**4-Et$_4$N**) (0.508 g, 0.74 mmol) in 15 mL of dry THF was added. The mixture was heated under reflux for 18 hours and was filtered through Celite. After the solvent was

TABLE 2 A Comparison of Bond Lengths (Å) and Bond Angles (°) in **4-Et$_4$N** with those in Corresponding Phosphorus [**8-K (18-crown-6)**], Antimony (**9-Et$_4$N**), and Bismuth Compounds (**10-Et$_4$N**)



E= P, 8-K(18-crown-6)
E=As, 4-Et$_4$N
E=Sb, 9-Et$_4$N
E=Bi, 10-Et$_4$N

	8-K(18-crown-6) *[7]*	**4-Et$_4$N**	**9-Et$_4$N***[8]*	**10-Et$_4$N***[9]*
		Bond Lengths (Å)		
a	1.901(2)	2.011(4)	2.136(9)	2.306(5)
b	1.881(2)	2.064(4)	2.147(9)	2.273(5)
c	1.847(3)	1.947(6)	2.15(1)	2.237(8)
d	1.844(3)	1.975(6)	2.16(1)	2.249(7)
		Bond Angles (°)		
ab	171.2(1)	169.0(2)	158.6(4)	159.7(2)
cd	109.1(1)	100.9(3)	103.4(5)	94.1(3)
ac	84.3(1)	82.0(2)	77.3(4)	74.8(2)
bd	85.0(1)	80.5(2)	77.1(4)	73.8(2)
ad	89.8(1)	91.8(2)	90.0(4)	91.0(2)
bc	90.5(1)	91.8(2)	89.0(4)	92.6(2)

evaporated in vacuo, the crude products were subjected to column chromatography (ether:*n*-hexane = 1:3) to give **6** (0.390 g, 72%) as orange needles (recrystallized from benzene-*n*-hexane); m.p. 209.5–210.5°C (dec). IR (KBr) 2015, 2050 cm^{-1}. ^{1}H NMR (CDCl$_3$) 5.03 (s, 5 H), 7.54 (t, 2 H, J = 7 Hz), 7.61 (t, 2 H, J = 7 Hz), 7.67 (d, 2 H, J = 7 Hz), 8.40 (d, 2 H, J = 7 Hz). ^{19}F NMR (CDCl$_3$) −74.7 (q, 6 F, J = 9 Hz), −75.2 (q, 6 F, J = 9 Hz). Anal. Calcd for $C_{25}H_{13}F_{12}O_4FeAs$: C, 40.79; H, 1.78. Found: C, 41.04; H, 1.63.

*{Bis[3,3-bis(trifluoromethyl)benzenemethanolato(2-)C^2,O]arsoranide}carbonyl (triphenylphosphine)(cyclopentadienyl)iron(II) (***7a** *and* **7b***)*

A solution of **6** (162 mg, 0.22 mmol) and triphenylphosphine (58 mg, 0.22 mmol) in 5 mL of 1,2-dichloroethane was irradiated with a tungsten lamp for 6 hours at room temperature. After the solvent was evaporated, the crude products were subjected to column chromatography (ethyl acetate:*n*-hexane = 1:5) to give the less polar diastereomer **7a** (Rf = 0.4) and the more polar diastereomer **7b** (Rf = 0.2). **7a** (20 mg, 10%) was obtained as orange needles (recrystallized from benzene-*n*-hexane); m.p. ca. 200°C (dec). ^{1}H NMR (acetone-d$_6$) 4.95 (d, 5 H, J = 1.5 Hz), 7.3–8.5 (m, 23 H). ^{19}F NMR (CDCl$_3$) −70.8 (bq, 6 F, J = 9 Hz), −72.0 (bq, 6 F, J = 9 Hz). ^{31}P NMR (CDCl$_3$) 63.9 (s, 1 P). Anal. Calcd for $C_{42}H_{28}F_{12}O_3PFeAs$: C, 51.98; H, 2.91. Found: C, 52.54; H, 3.11. **7b** (129 mg, 61%) was obtained as orange needles (recrystallized from ethyl acetate-*n*-hexane); m.p. ca. 200°C (dec). IR (KBr) 1972 cm^{-1}. ^{1}H NMR (CDCl$_3$) 4.46 (d, 5 H, J = 1.5 Hz), 7.2–8.4 (m, 23 H). ^{19}F NMR (CDCl$_3$) −70.9 (bq, 6 F, J = 9 Hz), −71.0 (bq, 6 F, J = 9 Hz). ^{31}P NMR (CDCl$_3$) 67.9 (s, 1 P). Anal. Calcd for $C_{42}H_{28}F_{12}O_3PFeAs \; 0.5C_6H_6$: C, 53.54; H, 3.10. Found: C, 53.14; H, 3.01.

Crystallographic Studies of **4-Et$_4$N, 6**, *and* **7a**

Crystal data and numerical details of the structure determinations are given in Table 3. Crystals suitable for X-ray structure determination were mounted on a Mac Science MXC3 diffractometer and irradiated with graphite-monochromated Mo Kα radiation (λ = 0.71073 Å) for **4-Et$_4$N** and **6** and Cu Kα radiation (λ = 1.5418 Å) for **7a** for data collection. Lattice parameters were determined by least-squares fitting of 31 reflections for **4-Et$_4$N** with $31° < 2\theta < 35°$, 31 reflections for **6** with $26° < 2\theta < 30°$, and of 31 reflections for **7a** with $45° < 2\theta < 51°$. Data were collected with the $2\theta/\omega$ scan mode. The structures were solved using the SIR-92 program in teXsan (Rigaku) package [10] and refined by full-matrix least-squares. No absorption correction was made for **4-Et$_4$N** and **6**, and the empirical absorption correction (DIF-ABS) was made for **7a**. Refinement on F was carried out by full-matrix least-squares. Hydrogen atoms were included in the refinement on calculated positions (C–H = 1.0 Å) riding on their carrier atoms with isotropic thermal parameters, except for solvent benzene (population 0.5) in **7a**, which could be found on a difference Fourier map, and these coordinates were included in the refinement. All computations were carried out on an SGI O$_2$ using the teXsan program [10].

SUPPLEMENTARY MATERIAL AVAILABLE

A complete description of the X-ray crystallographic structure determinations of **4-Et$_4$N**, 6, and **7a** have been deposited at the Cambridge Crystallographic Data Centre (CCDC).

ACKNOWLEDGMENTS

We thank to Central Glass Co. for supplying us with 1,1-bis(trifluoromethyl)benzyl alcohol.

TABLE 3 Crystallographic Data for $Rf_2As^-Et_4N^+$ (**4-Et$_r$N**), $Rf_2AsFeCp(CO)_2$ (**6**), and $Rf_2AsFeCp(CO)(PPh_3)$ (**7a**)

Molecule	$Rf_2As^-Et_4N^+$ (**4-Et$_4$N**)	$Rf_2AsFeCp(CO)_2$ (**6**)	$Rf_2AsFeCp(CO)(PPh_3)$ (**7a**)
Formula	$C_{26}H_{18}NO_2F_{12}As$	$C_{25}H_{13}O_4F_{12}FeAs$	$C_{42}H_{28}O_3F_{12}PFeAs + 0.5C_6H_6$
Mol Wt	679.3	736.1	1009.5
Cryst Syst	orthorhombic	monoclinic	triclinic
Space Group	*Pbca*	$P2_1/a$	$P\bar{1}$
Cryst Dimens (mm)	1.00 × 0.75 × 0.35	0.65 × 0.45 × 0.30	0.45 × 0.30 × 0.10
Color	colorless	orange	orange
Habit	plate	plate	plate
a, Å	19.727(8)	19.041(4)	12.896(3)
b, Å	18.338(8)	16.854(4)	13.664(4)
c, Å	16.190(8)	17.968(4)	13.925(3)
α, deg	90	90	82.72(2)
β, deg	90	114.98(2)	67.74(2)
γ, deg	90	90	68.58(2)
V, $Å^3$	5856(7)	5226(2)	2113(1)
Z	8	8	2
D_{calc} (g cm^{-3})	1.54	1.87	1.53
abs coeff, cm^{-1}	11.93	18.59	47.30
F(000)	2704	2896	1014
Radiation; λ, Å	Mo Kα, 0.71073	Mo Kα, 0.71073	Cu Kα, 1.5418
Temp, °C	23 ± 1	23 ± 1	23 ± 1
2θ max (deg)	55	45	120
Scan Rate (deg/min)	5.0	3.0	5.0
Linear Decay (%)	–	3	–
Data Collected	+h, +k, +1	±h, +k, +1	±h, ±k, −1
Total Data Collected, Unique, Observed	7474, 7399, 3038 ($I > 3\sigma(I)$)	7466, 7128, 4607 ($I > 3\sigma(I)$)	6677, 6305, 5303 ($I > 3\sigma(I)$)
No. of Params Refined	379	775	553
R, R_w,	0.049, 0.052[a]	0.038, 0.040[a]	0.058, 0.069[a]
Goodness Of Fit (obs)	0.21	0.16	0.56
Max Shift in Final Cycle	0.0055	0.0012	0.097
Final Diff Map, Max (e/$Å^3$)	0.57	0.58	1.05

[a] Function minimized was sum $[w(|Fo|^2\text{-}[Fc|^2)^2]$ in which $w = 1.0/\text{sigma}|Fo|^2$.
$R = \text{sum}[||Fo|\text{-}|Fc||)/\text{sum}|Fo|$. $Rw = [\text{sumw}(|Fo|\text{-}|Fc|)^2/\text{sum}|Fo|^2]^{1/2}$.

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