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## Structure of a Tetranuclear Uranium(IV) Compound: Hemiaquatetrakis(trifluoroacetato)uranium(IV)–4,7,13,16,21,24-Hexaoxa- 1,10-diazabicyclo[8.8.8]hexacosane (4/2)

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**Abstract.**  $4[\text{U}(\text{CF}_3\text{COO})_4(\text{H}_2\text{O})_{0.5}]_2\text{C}_{18}\text{H}_{36}\text{N}_2\text{O}_6$ ,  $M_r = 3549$ , triclinic,  $P\bar{1}$ ,  $a = 13.003$  (5),  $b = 13.338$  (7),  $c = 16.738$  (4) Å,  $\alpha = 84.36$  (3),  $\beta = 68.71$  (2),  $\gamma = 87.19$  (4)°,  $V = 2691$  (4) Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 2.190$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.71069$  Å,  $\mu = 5.853$  mm<sup>-1</sup>,  $F(000) = 1680$ ,  $T = 295$  K,  $R = 0.038$  for 3534 unique observed reflections. Four U<sup>IV</sup> atoms are connected by bridging trifluoroacetate ligands and water molecules to form a centrosymmetric tetrameric neutral unit in which the four U atoms are 4.0769 (4) and 3.5969 (6) Å apart at the corners of a lozenge. The trifluoroacetato group behaves as either a unidentate or a bridging group. Two water molecules, located inside the uranium tetranuclear unit, are triply shared by U atoms. The two cryptand molecules, related by an inversion center, have an *endo-endo* type configuration: no significant contacts exist between the tetramer and cryptand units.

**Introduction.** The <sup>1</sup>H NMR spectra of complexes of U<sup>IV</sup> salts with crown ethers and cryptands in solvents with high electric constants have given evidence for direct coordination of the U atoms to the O atoms of the macrocyclic ligands; in the solid state, a structural determination has confirmed the U<sup>IV</sup> insertion as the UCl<sub>3</sub><sup>+</sup> cation in dicyclohexyl-18-crown-6 (de Villardi, Charpin, Costes, Folcher, Plurien, Rigny & de Rango, 1978). Attempts to isolate such complexes

with cryptands have led to crystals which could not be handled. However, when a solution of U<sup>IV</sup> trifluoroacetate and ⟨222⟩ cryptand (⟨222⟩ = 4,7,13,16,21,24-hexaoxa-1,10-diazabicyclo[8.8.8]hexacosane) in acetonitrile was evaporated in air particularly stable crystals were formed. We report here the structure of the compound so obtained.

**Experimental.** Green crystal approximately 0.5 × 0.45 × 0.3 mm; Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo Kα radiation;  $\omega$ -2 $\theta$  scan,  $\omega$ -scan width: (0.8 + 0.35tan $\theta$ )°; determination of the lattice parameters from 25 reflections (8 <  $\theta$  < 12°); three standard reflections ( $\bar{3}\bar{1}\bar{4}$ , 321,  $\bar{1}\bar{1}\bar{3}$ ) were monitored every hour, decay of 1.6% in 55 h, not corrected; 5479 reflections measured, 4715 unique ( $R_{\text{int}} = 0.023$ ), 3534 observed with  $I > 3\sigma(I)$  which were used in the refinement;  $2 \leq \theta \leq 20^\circ$ ,  $0 \leq h \leq 12$ ,  $-12 \leq k \leq 12$ ,  $-16 \leq l \leq 16$ ; Lp corrections; absorption correction using the program *DIFABS* (Walker & Stuart, 1983), max. and min. corrections 1.16 and 0.82; the structure was solved by the Patterson function, for U, and the remaining atoms were located by the difference Fourier technique; full-matrix least-squares refinement on  $F$ ; anisotropic thermal parameters for U and F, isotropic for O and the rest due to lack of data. The F atoms in one CF<sub>3</sub> group [belonging to the monodentate carboxylate C(71)—C(72)], which showed high thermal motion,

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters*

	x	y	z	<i>B</i> (Å <sup>2</sup> )
U(1)	-0.06168 (4)	-0.11493 (4)	0.05989 (4)	2.58 (1)*
U(2)	-0.03658 (5)	-0.11781 (4)	0.19217 (3)	2.68 (1)*
F(1)	0.3324 (9)	-0.1922 (9)	0.0367 (9)	9.6 (4)*
F(2)	0.3758 (8)	-0.044 (1)	0.0277 (9)	10.4 (4)*
F(3)	0.3913 (9)	-0.110 (1)	-0.0833 (8)	9.9 (4)*
F(11)	-0.392 (1)	-0.1254 (8)	0.3415 (9)	9.1 (4)*
F(12)	-0.4548 (9)	0.003 (1)	0.292 (1)	13.8 (5)*
F(13)	-0.377 (1)	0.016 (1)	0.373 (1)	13.8 (6)*
F(21)	-0.3737 (9)	-0.240 (1)	-0.003 (1)	13.0 (5)*
F(22)	-0.300 (1)	-0.374 (1)	0.004 (1)	14.1 (5)*
F(23)	-0.257 (1)	-0.287 (1)	-0.1098 (8)	12.5 (5)*
F(31)	-0.107 (1)	0.4644 (9)	-0.004 (1)	13.8 (6)*
F(32)	-0.202 (1)	0.433 (1)	0.1194 (9)	13.6 (5)*
F(33)	0.242 (1)	-0.379 (1)	-0.031 (1)	12.9 (5)*
F(41)	-0.301 (1)	0.005 (1)	-0.3100 (9)	13.5 (5)*
F(42)	-0.235 (1)	-0.129 (1)	-0.291 (1)	13.7 (4)*
F(43)	-0.356 (1)	-0.073 (2)	-0.190 (1)	15.6 (7)*
F(51)	-0.028 (1)	-0.180 (1)	0.3840 (7)	10.2 (4)*
F(52)	-0.171 (1)	-0.227 (1)	0.3752 (8)	12.6 (5)*
F(53)	-0.023 (1)	-0.3013 (9)	0.3192 (8)	11.9 (5)*
F(61)	-0.101 (1)	0.121 (1)	0.5219 (8)	15.8 (5)*
F(62)	-0.157 (2)	-0.013 (1)	0.489 (1)	15.4 (7)*
F(63)	-0.005 (1)	0.060 (2)	0.405 (1)	14.4 (6)*
F(71)	0.056 (3)	-0.464 (3)	-0.217 (3)	16 (1)
F(72)	-0.014 (2)	-0.480 (2)	-0.323 (2)	10.9 (9)
F(73)	-0.111 (3)	-0.464 (3)	-0.214 (2)	14 (1)
F(74)	-0.134 (3)	-0.401 (3)	-0.258 (2)	13 (1)
F(75)	-0.057 (4)	-0.460 (3)	-0.172 (3)	17 (1)
F(76)	0.047 (3)	-0.493 (3)	-0.310 (3)	16 (1)
O	-0.0213 (7)	0.0410 (7)	0.0763 (5)	2.4 (2)
O(1)	-0.2099 (8)	-0.0206 (7)	0.0294 (6)	3.3 (2)
O(2)	0.1375 (8)	-0.1265 (8)	0.0365 (6)	3.6 (2)
O(11)	-0.2279 (8)	-0.0917 (7)	0.1920 (6)	3.3 (2)
O(12)	-0.2266 (8)	0.0693 (8)	0.2202 (6)	3.5 (2)
O(21)	-0.2113 (8)	-0.2374 (8)	0.0727 (6)	4.0 (2)
O(22)	-0.1162 (8)	-0.1926 (8)	-0.0634 (6)	3.5 (2)
O(31)	0.0218 (8)	-0.2795 (8)	0.0178 (6)	3.8 (2)
O(32)	-0.1218 (8)	0.2463 (8)	0.1223 (6)	3.9 (2)
O(41)	-0.190 (1)	0.092 (1)	-0.2315 (8)	6.3 (3)
O(42)	0.1250 (9)	0.0613 (8)	0.2017 (7)	4.5 (3)
O(51)	-0.037 (1)	-0.1887 (9)	0.1871 (8)	5.6 (3)
O(52)	-0.0628 (9)	-0.0545 (9)	0.2607 (7)	5.1 (3)
O(61)	-0.255 (2)	0.178 (2)	0.458 (1)	12.0 (6)
O(62)	-0.140 (1)	0.132 (1)	0.3324 (8)	6.6 (3)
O(71)	0.006 (1)	0.265 (1)	0.2269 (8)	6.2 (3)
O(72)	0.119 (5)	-0.375 (4)	-0.266 (4)	12 (2)
O(72')	0.004 (3)	-0.303 (3)	-0.362 (2)	14 (1)
C(1)	0.214 (1)	-0.067 (1)	0.0019 (9)	2.8 (3)
C(2)	0.329 (1)	-0.102 (1)	-0.002 (1)	4.7 (4)
C(11)	-0.264 (1)	-0.017 (1)	0.2317 (9)	3.2 (3)
C(12)	-0.374 (2)	-0.035 (2)	0.307 (1)	6.2 (5)
C(21)	-0.196 (1)	-0.233 (1)	-0.0054 (9)	3.4 (3)
C(22)	-0.282 (2)	-0.285 (1)	-0.031 (1)	5.8 (5)
C(31)	0.095 (1)	-0.297 (1)	-0.0518 (9)	3.3 (3)
C(32)	0.158 (1)	-0.396 (1)	-0.048 (1)	4.4 (4)
C(41)	-0.186 (1)	0.000 (1)	-0.227 (1)	4.6 (4)
C(42)	-0.270 (2)	-0.053 (2)	-0.254 (1)	6.7 (5)
C(51)	-0.049 (1)	-0.146 (1)	0.250 (1)	3.9 (4)
C(52)	-0.067 (1)	-0.214 (1)	0.332 (1)	4.3 (4)
C(61)	-0.170 (2)	0.142 (2)	0.411 (1)	6.8 (5)
C(62)	-0.104 (2)	0.080 (2)	0.461 (2)	9.0 (7)
C(71)	0.007 (2)	-0.329 (2)	-0.281 (2)	10.9 (8)
C(72)	-0.027 (3)	-0.440 (3)	-0.254 (2)	14 (1)
O(101)	-0.691 (1)	-0.658 (1)	0.4482 (8)	6.3 (3)
O(102)	-0.7706 (9)	-0.4585 (9)	0.4151 (7)	5.4 (3)
O(201)	-0.396 (1)	-0.488 (1)	0.3756 (8)	6.8 (3)
O(202)	-0.465 (1)	-0.322 (1)	0.290 (1)	9.0 (4)
O(301)	-0.4739 (9)	-0.6420 (9)	0.2344 (7)	4.7 (3)
O(302)	-0.6212 (9)	-0.4934 (9)	0.1928 (7)	4.9 (3)
N(101)	-0.462 (1)	-0.682 (1)	0.399 (1)	5.6 (4)
N(102)	-0.678 (1)	-0.322 (1)	0.2808 (8)	4.2 (3)
C(101)	-0.545 (2)	-0.768 (2)	0.445 (1)	7.6 (6)
C(102)	-0.645 (2)	-0.758 (2)	0.423 (2)	8.3 (6)
C(103)	-0.782 (2)	-0.637 (2)	0.421 (1)	6.8 (5)
C(104)	-0.839 (2)	-0.544 (2)	0.460 (1)	6.0 (5)
C(105)	-0.831 (2)	-0.367 (1)	0.419 (1)	5.8 (5)
C(106)	-0.755 (2)	-0.285 (2)	0.367 (1)	6.1 (5)
C(201)	-0.423 (2)	-0.642 (2)	0.463 (2)	8.7 (7)
C(202)	-0.346 (2)	-0.563 (2)	0.426 (2)	8.5 (7)
C(203)	-0.329 (2)	-0.412 (2)	0.324 (2)	9.2 (7)
C(204)	-0.357 (2)	-0.377 (2)	0.255 (2)	10.0 (8)
C(205')	-0.535 (3)	-0.218 (3)	0.277 (3)	6 (1)
C(205)	-0.472 (3)	-0.283 (3)	0.211 (2)	5.5 (9)
C(206)	-0.592 (2)	-0.245 (2)	0.231 (1)	5.8 (5)

Table 1 (cont.)

	x	y	z	<i>B</i> (Å <sup>2</sup> )
C(301)	-0.370 (1)	-0.712 (1)	0.317 (1)	5.2 (4)
C(302)	-0.418 (1)	-0.734 (1)	0.251 (1)	5.3 (4)
C(303)	-0.526 (1)	-0.653 (1)	0.173 (1)	4.4 (4)
C(304)	-0.541 (1)	-0.550 (1)	0.129 (1)	4.7 (4)
C(305)	-0.663 (1)	-0.410 (1)	0.153 (1)	5.4 (4)
C(306)	-0.741 (1)	-0.352 (1)	0.226 (1)	4.8 (4)

\*  $B_{eq} = \frac{1}{3}(\Sigma_i \beta_{11} + \Sigma_i \beta_{22} + \Sigma_i \beta_{33})$ .

have been resolved into two sets of positions with an occupation factor of 0.5. Moreover, in the final difference Fourier map one residual peak was observed near the carbon of the same ligand, C(71), showing two positions for the non-bonded oxygen, O(72) and O(72') introduced with occupation factors of 0.66 and 0.34 respectively. In addition, the final difference Fourier map showed disorder in one chain of the cryptand ligand: alternate positions were introduced in the least-squares refinement with occupation factors fixed from their relative intensities, 0.5 for C(205) and C(205'). H atoms were not included in the refinement. At the final stage: 476 variables,  $R = 0.038$ ,  $wR = 0.043$  ( $w = 1$ ),  $(\Delta/\sigma)_{max} = 0.12$ ,  $S = 4.65$ , residual electron density in the final difference Fourier map  $0.92 e \text{ \AA}^{-3}$ ; atomic scattering factors including anomalous-dispersion terms were from *International Tables for X-ray Crystallography* (1974); calculations were performed on a MicroVAX II using the *SDP* set of programs (Frenz, 1985).

**Discussion.** The final positional coordinates and equivalent isotropic temperature factors are listed in Table 1.\* The structure analysis shows that the neutral  $U^{IV}$  unit with 16 trifluoroacetato groups (tfa) is a cluster involving four U atoms joined by ten bridging carboxylate ligands. A view of the tetranuclear unit, excluding F atoms, is shown in Fig. 1 while selected bond lengths and angles are listed in Table 2. This tetrameric unit is centrosymmetric and can be seen as formed from two triangles of U atoms [U(1)—U(2) 4.0769 (4), U(1)—U(1') 3.5959 (6) Å] sharing one edge [U(1)—U(1')], with each central O atom of a water molecule trigonally bonded to three U atoms. These two O atoms are coplanar with the four U atoms; values of O—O contacts and angles suggest hydrogen bonds exist between the  $H_2O$  molecules and some tfa oxygens [O(1), O(2) and O(22)]. Such a triangular U-atom arrangement about a central O atom is not unusual for uranyl compounds  $\{[(UO_2)_3(DPA)_3O]^{2-}$  (DPA = 2,2',8,8'-tetramethyl-3,5,7-nonanetricionate) (Lintvedt, Heeg,

\* Lists of structure factors and anisotropic thermal parameters, and fuller lists of bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52911 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

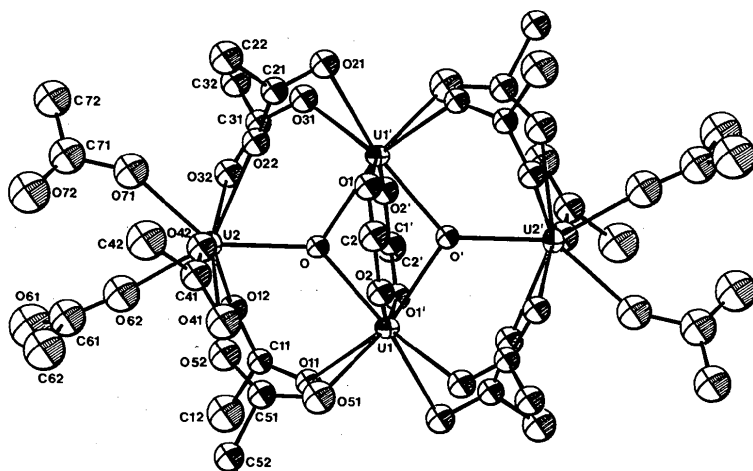


Fig. 1. ORTEP drawing (Johnson, 1976) of the tetranuclear complex with F atoms omitted for clarity. The primed atoms are related by the center of symmetry.

Table 2. Selected bond lengths (Å) and bond angles (°)

U environment

U(1)—O	2.236 (7)	U(2)—O	2.223 (8)
U(1)—O'	2.282 (7)	U(2)—O(12)	2.450 (15)
U(1)—O(1)	2.431 (10)	U(2)—O(22)	2.501 (10)
U(1)—O(2)	2.474 (10)	U(2)—O(32)	2.428 (11)
U(1)—O(11)	2.501 (7)	U(2)—O(42)	2.252 (10)
U(1)—O(21)	2.537 (7)	U(2)—O(52)	2.447 (11)
U(1)—O(31)	2.451 (6)	U(2)—O(62)	2.264 (12)
U(1)—O(51)	2.381 (10)	U(2)—O(71)	2.253 (14)

U(1)—U(2)—U(1')	53.25 (1)	U(2)—U(1)—U(2')	126.75 (1)
U(2)—U(1)—U(1')	61.47 (1)	U(2)—U(1')—U(1)	65.28 (1)
U(2)—O—U(1)	132.2 (3)	U(2)—O—U(1')	122.1 (3)
U(1)—O—U(1')	105.5 (3)		

Possible hydrogen bonding

O—O'	2.74 (2)	O—O(1)	2.90 (1)
O—O(2)	2.92 (1)	O—O(1')	3.00 (1)
O—O(22)	2.71 (1)	O—O(2')	2.94 (1)
O'—O—O(22)	113.0 (4)	O(1)—O—O(2)	105.1 (4)
O(22)—O—O(2')	101.2 (3)		
O(1)—O'—O(2)	107.5 (4)		

Carboxylate groups (mean values)

C—O	1.23 (2)	C—O—C	128 (2)
C—C	1.54 (2)	C—C—F	115 (2)
C—F	1.28 (2)	C—C—F	112 (3)

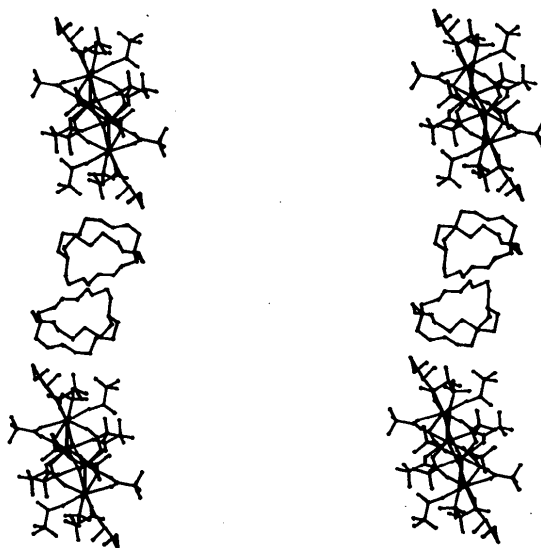


Fig. 2. Stereoscopic view of the packing.

Ahmad & Glick, 1982), [(UO<sub>2</sub>)<sub>4</sub>O<sub>2</sub>Cl<sub>8</sub>(H<sub>2</sub>O)<sub>2</sub>]<sup>4-</sup> (Perrin, 1977), [(UO<sub>2</sub>)<sub>6</sub>(N<sub>3</sub>)<sub>16</sub>O<sub>2</sub>]<sup>8-</sup> (Charpin, Lance, Nierlich, Vigner, Livet & Musikas, 1986)); only one complex with U<sup>IV</sup> has been reported [U<sub>3</sub>O(OCMe<sub>3</sub>)<sub>10</sub> (Cotton, Marler & Schwotzer, 1984)]. In all these complexes, the central oxygen has been identified as O<sup>2-</sup>, so the compound described here is the first one involving a central water bridge.

U(1) and U(2) are both eight coordinate and achieve their coordination number with O atoms of carboxylate groups and water molecules. However, the U(1) environment, made up of six bridging carboxylates and two water molecules, differs from that

of U(2) which involves four bridging and three monodentate carboxylates, and one water molecule. The U(1) polyhedron is best described as a distorted dodecahedron in which the dihedral angle between the two trapezoids O(1)—O'—O(11)—O(51) (max. and min. deviations from the least-squares plane -0.125, 0.119 Å) and O—O(2)—O(21)—O(31) (-0.109, 0.114 Å) is 82 (2)°, while the U(2) polyhedron can be seen as a distorted bicapped trigonal prism.

The carboxylates are coordinated in the expected manner: the U—O(bridging carboxylate) distances 2.459 (8) Å (mean) are significantly longer than the shorter U—O bonds involving unidentate carboxylate, 2.256 (12) Å (mean). Moreover, the geom-

etry and the bond lengths of these groups are in the correct ranges, except those of the disordered monodentate carboxylate: in particular, the C(72)—F distances, which are very inaccurate, range from 1.09 to 1.44 Å [mean value = 1.315 (3) Å] whereas the other C—F distances are dispersed within the standard deviations [mean value 1.276 (8) Å].\*

The two cryptand molecules, related by an inversion center, have an *endo-endo* conformation. The distances and angles have correct values. One of the three chains appears disordered in one carbon position only. The crystal packing is shown in Fig. 2; no significant contact is observed between the tetramer and cryptand units since all the C...F contacts are greater than 3.14 Å.

\* See deposition footnote.

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## Structures of $[(C_2H_5)_4N]_2[In_2Fe_6(CO)_{24}]$ and $[(C_2H_5)_4N][(2,2'-bipyridine)InFe_2(CO)_8]$

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**Abstract.** Bis(tetraethylammonium) bis[[di- $\mu$ -carbonyl-bis[tricarbonylferrio(*Fe-Fe*)]- $\mu$ -(tetracarbonylferrio)-indiumate(1<sup>-</sup>)],  $2C_8H_{20}N^+ \cdot [Fe_6In_2(CO)_{24}]^{2-}$ ,  $M_r = 1497.48$ , orthorhombic, *Cmca*,  $a = 18.928$  (2),  $b = 17.051$  (8),  $c = 17.245$  (4) Å,  $V = 5566$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.79$  g cm<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71069$  Å,  $\mu = 23.95$  cm<sup>-1</sup>,  $F(000) = 2960$ ,  $T = 296$  K,  $R = 0.042$ ,  $wR = 0.054$  for 1794 unique reflections with  $I > 3\sigma(I)$ .  $[(C_2H_5)_4N]_2[In_2Fe_6(CO)_{24}]$  consists of a central  $In_2Fe_2$  parallelogram with each In atom linked to an additional  $[Fe_2(CO)_6(\mu-CO)_2]$  group in a distorted tetrahedral array. The  $In_2Fe_2$  parallelogram sits on a crystallographic site of  $2/m$  symmetry. Tetraethylammonium (2,2'-bipyridyl)bis-(tetracarbonylferrio)indiumate(1<sup>-</sup>),  $C_8H_{20}N^+ \cdot [Fe_2In(CO)_8(C_{10}H_8N_2)]^-$ ,  $M_r = 737.04$ , triclinic, *P1*,  $a = 10.060$  (4),  $b = 11.567$  (7),  $c = 14.024$  (6) Å,  $\alpha = 93.79$  (9),  $\beta = 95.04$  (3),  $\gamma = 107.65$  (6)°,  $V = 1542$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.59$  g cm<sup>-3</sup>,  $\lambda(Mo K\alpha) = 0.71069$  Å,  $\mu = 17.06$  cm<sup>-1</sup>,  $F(000) = 740$ ,  $T = 296$  K,  $R = 0.063$ ,  $wR = 0.087$  for 4853 unique observed reflections with  $I > 3\sigma(I)$ .  $[(C_2H_5)_4N] \cdot [(bpy)InFe_2(CO)_8]$  (*bpy* = 2,2'-bipyridine) crystallizes with the  $Fe(CO)_4$  groups and the bipyridine ligand in

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a distorted tetrahedral arrangement around the In atom. The  $[Fe(CO)_4]$  groups exhibit trigonal bipyramidal geometries.

**Introduction.** A number of different iron-carbonyl-containing indium compounds have been synthesized, including  $[(C_2H_5)_4N][Fe(CO)_2CpInPh_3]$  (Burlitch, Leonowicz, Petersen & Hughes, 1979),  $\{[(C_6H_5)_3P]_2N\}_2[Fe(CO)_4InBr_3]$ ,  $\{[(C_6H_5)_3P]_2N\} \cdot [Fe(CO)_4InBr_2]$ ,  $\{[(C_6H_5)_3P]_2N\} \cdot [Fe(CO)_4InBr_2(L)]$  [ $L = N(C_2H_5)_3$ , pyridine] (Ruff, 1968),  $\{[Fe(CO)_2Cp]_2InX(L)\}$  ( $X = Br, Cl$ ;  $L = THF, p$ -dioxan),  $[Fe(CO)_2CpInBr_2(L)]$  ( $L = THF, p$ -dioxan) (Hsieh & Mays, 1971) and  $[Fe_2(CO)_8\{\mu-InMn(CO)_5\}_2]$  (Preut & Haupt, 1979). Recently several novel Tl-Fe carbonyl compounds have been synthesized and structurally characterized. These compounds include  $[(C_2H_5)_4N]_2[Tl_2Fe_4(CO)_{16}]$  (Whitmire, Cassidy, Rheingold & Ryan, 1987), and its Lewis base adducts (Cassidy & Whitmire, 1989),  $[(C_2H_5)_4N]_4 \cdot [Tl_4Fe_8(CO)_{30}]$ ,  $[(C_2H_5)_4N]_6[Tl_6Fe_{10}(CO)_{36}]$  (Whitmire, Cassidy, Rheingold & Ryan, 1987), and  $\{[(C_6H_5)_3P]_2N\}_2[Tl_2Fe_6(CO)_{24}]$  (Cassidy & Whitmire, 1989). Consequently we decided to try to synthesize some In-Fe carbonyl complexes by methods similar to those developed for thallium. The structures of

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