

$\omega$ -2 $\theta$  scans  
Absorption correction:  
refined from  $\Delta F$   
(DIFABS; Walker &  
Stuart, 1983)  
 $T_{\min} = 0.411$ ,  $T_{\max} = 0.675$   
3857 measured reflections  
3685 independent reflections

$R_{\text{int}} = 0.0215$   
 $\theta_{\text{max}} = 60^\circ$   
 $h = 0 \rightarrow 15$   
 $k = 0 \rightarrow 14$   
 $l = -15 \rightarrow 15$   
3 standard reflections  
every 150 reflections  
intensity decay: 12.5%

### Refinement

Refinement on  $F$   
 $R = 0.0637$   
 $wR = 0.0777$   
 $S = 2.114$   
2720 reflections  
316 parameters  
H atoms not refined  
 $w = 1/[\sigma^2(F_o)$   
 $+ 0.00016|F_o|^2]$

$(\Delta/\sigma)_{\text{max}} = 0.0756$   
 $\Delta\rho_{\text{max}} = 0.43 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.39 \text{ e } \text{\AA}^{-3}$   
Extinction correction: none  
Scattering factors from  
*International Tables for*  
*Crystallography* (Vol. C)

Table 1. Selected bond lengths ( $\text{\AA}$ )

|                       |           |                      |           |
|-----------------------|-----------|----------------------|-----------|
| Na1—Na2 <sup>i</sup>  | 3.596 (3) | Na2—O10 <sup>v</sup> | 2.356 (4) |
| Na1—O4 <sup>ii</sup>  | 2.343 (3) | Na2—O12              | 2.352 (4) |
| Na1—O7 <sup>iii</sup> | 2.401 (4) | O1—C1                | 1.378 (5) |
| Na1—O9 <sup>iv</sup>  | 2.498 (4) | O1—C13               | 1.371 (4) |
| Na1—O10               | 2.517 (4) | O2—C11               | 1.297 (5) |
| Na1—O11 <sup>v</sup>  | 2.709 (4) | O3—C3                | 1.285 (5) |
| Na1—O13               | 2.342 (5) | O4—C20               | 1.251 (5) |
| Na2—O2                | 2.568 (4) | O5—C20               | 1.256 (5) |
| Na2—O4 <sup>i</sup>   | 2.433 (4) | C7—C14               | 1.500 (6) |
| Na2—O8 <sup>iii</sup> | 2.439 (4) | C19—C20              | 1.509 (6) |

Symmetry codes: (i)  $\frac{1}{2} + x, \frac{1}{2} - y, z$ ; (ii)  $1 + x, y, z$ ; (iii)  $x, y, z - 1$ ; (iv)  $\frac{1}{2} + x, \frac{1}{2} - y, z - 1$ ; (v)  $x - \frac{1}{2}, \frac{1}{2} - y, z$ .

The  $\omega$ -scan width was  $(1.3 + 0.14 \tan \theta)^\circ$  and the scan speed was  $32^\circ \text{ min}^{-1}$ . Refinement was by full-matrix least-squares methods. The H atoms associated with the O13 water molecule were not located.

Data collection: *MSCIAFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCIAFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SAPI91* (Fan, 1991). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: OA1007). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1997). **C53**, 285–287

## Tetracarbonyl- $1\kappa^2\text{C}, 2\kappa^2\text{C}-\mu$ -diphenylphosphido- $P:P-\mu$ -iodo-bis(triphenylphosphine)- $1\kappa\text{P}, 2\kappa\text{P}$ -diosmium(Os—Os) Benzene Solvate

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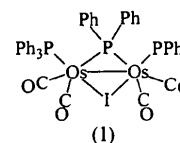
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### Abstract

In the title compound,  $[\text{Os}_2\text{I}(\text{C}_{12}\text{H}_{10}\text{P})(\text{C}_{18}\text{H}_{15}\text{P})_2(\text{CO})_4] \cdot \text{C}_6\text{H}_6$ , the Os—Os bond distance is unusually short [2.781 (1)  $\text{\AA}$ ]. The two Os atoms are symmetrically bridged by both an iodo and a phosphido ligand. Two carbonyl groups and a triphenylphosphine moiety complete the Os-atom coordination spheres. There are only two other reported structures with an iodo ligand bridging two Os atoms [Sutton, Nivin & Moss (1983). *Inorg. Chim. Acta*, **70**, 207–210; Geoffroy, Rosenberg, Herlinger & Rheingold (1986). *Inorg. Chem.* **25**, 2916–2919].

### Comment

The coordination geometries about the Os atoms in the title compound, (1), are octahedral, with distortions probably due to steric factors. The compound is diamagnetic with the octadecet count being completed by an unusually short Os—Os bond [2.781 (1)  $\text{\AA}$ ]. The asymmetric unit contains a severely distorted solvent molecule that according to the synthesis and recrystallization information should be benzene, but toluene cannot be ruled out. The solvent molecule is statistically disordered over two positions with a 63/37% occupancy distribution.



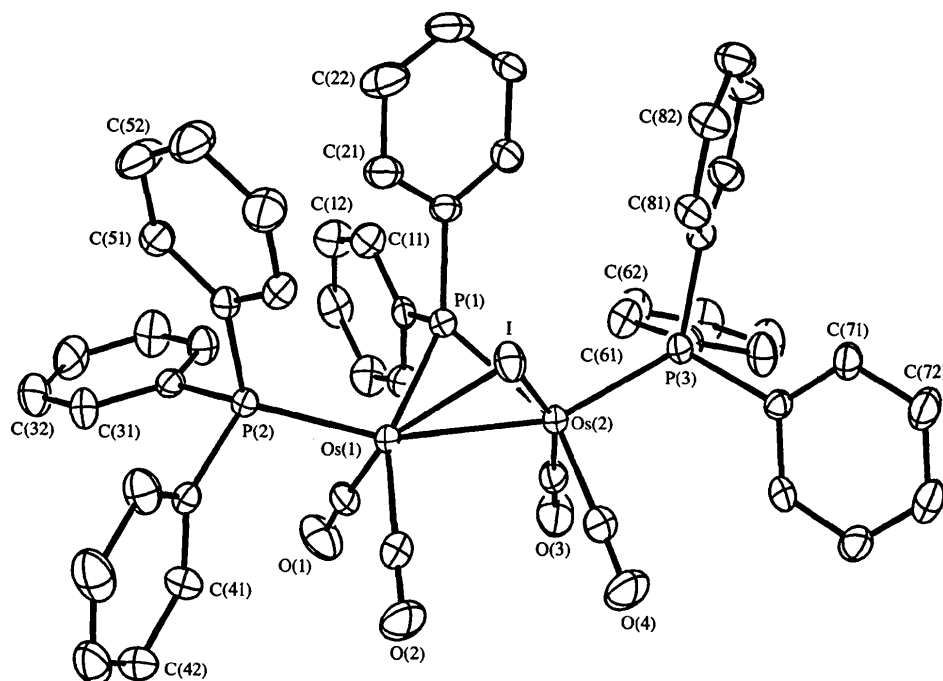


Fig. 1. The structure of the title compound with 30% displacement ellipsoids. H atoms and solvent molecules have been omitted for clarity.

The substitution of two PPh<sub>3</sub> ligands for CO in the binuclear carbonyl complex [Os<sub>2</sub>(μ-PPh<sub>2</sub>)(μ-I)(CO)<sub>6</sub>] occurs symmetrically. This structure is less sterically hindered than that of the isomer which has both PPh<sub>3</sub> ligands on the same Os atom. The presence of PPh<sub>3</sub> on an Os atom would also be expected to deactivate the metal center towards further phosphine substitution.

## Experimental

[Os<sub>2</sub>(μ-PPh<sub>2</sub>)(μ-I)(CO)<sub>6</sub>] was prepared according to literature methods. Benzene-*d*<sub>6</sub> and triphenylphosphine (PPh<sub>3</sub>; Aldrich) were used as received without further purification. All manipulations were performed using standard Schlenk techniques under an atmosphere of prepurified N<sub>2</sub>. For the synthesis of [Os<sub>2</sub>(μ-PPh<sub>2</sub>)(μ-I)(CO)<sub>4</sub>(PPh<sub>3</sub>)<sub>2</sub>], (1), a three-neck round-bottomed flask was charged with [Os<sub>2</sub>(μ-PPh<sub>2</sub>)(μ-I)(CO)<sub>6</sub>] and dry heptane. The reaction mixture was stirred for 4 h while heating to reflux under nitrogen. The solvent was removed leaving a yellow solid which was purified by chromatography on silica gel. The mass spectrum of compound (1) showed a parent ion and fragment ions corresponding to progressive loss of PPh<sub>3</sub> and four carbonyls. MS (EI): *m/z* 1332 (*M*<sup>+</sup>, <sup>192</sup>Os), 1276 (*M*<sup>+</sup> - 2CO), 1070 (*M*<sup>+</sup> - PPh<sub>3</sub>), 1042 (*M*<sup>+</sup> - PPh<sub>3</sub> - CO), 1014 (*M*<sup>+</sup> - PPh<sub>3</sub> - 2CO), 986 (*M*<sup>+</sup> - PPh<sub>3</sub> - 3CO), 958 (*M*<sup>+</sup> - PPh<sub>3</sub> - 4CO). The <sup>1</sup>H NMR spectrum (benzene-*d*<sub>6</sub>) showed two signals with relative intensities of 3:1 at δ 8.80 and 8.35 that were assigned to the PPh<sub>3</sub> and μ-PPh<sub>2</sub> ligands, respectively. The IR spectrum (benzene-*d*<sub>6</sub>) in the carbonyl stretching region showed ν<sub>CO</sub> bands at 2012 (*vs*), 1998 (*sh*), 1976 (*s*), 1939 (*s*) and 1928 (*sh*) cm<sup>-1</sup>. Yellow crystals of (1) were grown from benzene-*d*<sub>6</sub> by slow

evaporation at room temperature. Magnetic susceptibility measurements made with a Johnson Matthey magnetic susceptibility balance showed the title compound to be diamagnetic.

## Crystal data

[Os<sub>2</sub>I(C<sub>12</sub>H<sub>10</sub>P)(C<sub>18</sub>H<sub>15</sub>P)<sub>2</sub>(CO)<sub>4</sub>].C<sub>6</sub>H<sub>6</sub>  
*M<sub>r</sub>* = 1407.16  
 Triclinic  
*P* $\bar{1}$   
*a* = 10.788 (2) Å  
*b* = 13.754 (4) Å  
*c* = 19.164 (5) Å  
 $\alpha$  = 79.13 (2)°  
 $\beta$  = 81.39 (2)°  
 $\gamma$  = 69.45 (2)°  
*V* = 2604 (1) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 1.79 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 10.0–12.5°  
 $\mu$  = 5.61 mm<sup>-1</sup>  
*T* = 298 K  
 Plate  
 0.36 × 0.36 × 0.25 mm  
 Yellow

## Data collection

Nicolet R3m diffractometer with graphite monochromator  
 $\omega/2\theta$  scans  
 Absorption correction: semi-empirical (SHELXTL; Sheldrick, 1985)  
 $T_{\min}$  = 0.153,  $T_{\max}$  = 0.246  
 9167 measured reflections  
 7071 independent reflections

6810 reflections with  $F_o \geq 5\sigma(F_o)$   
 $R_{\text{int}}$  = 0.022  
 $\theta_{\text{max}}$  = 25°  
 $h$  = -12 → 12  
 $k$  = -16 → 16  
 $l$  = 0 → 22  
 3 standard reflections every 197 reflections  
 intensity decay: <1.0%

**Refinement**Refinement on  $F^2$  $R = 0.029$  $wR = 0.032$  $S = 1.0$ 

6810 reflections

673 parameters

H atoms not refined

$$w = 1/[\sigma^2(F_o) + 0.001F_o^2]$$

$$(\Delta/\sigma)_{\max} = 0.12$$

$$\Delta\rho_{\max} = 0.84 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.59 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Scattering factors from

SHELXTL

*Acta Cryst.* (1997). **C53**, 287–289**Hetero-Ring Oxygen Coordination to Strontium in Strontium Bis(2-furan-carboxylate)**BEATA PALUCHOWSKA,<sup>a</sup> JAN K. MAURIN<sup>a</sup> AND JANUSZ LECIEJEWICZ<sup>b</sup><sup>a</sup>*Institute of Atomic Energy, 05-400 Otwock-Świerk, Poland, and* <sup>b</sup>*Institute of Nuclear Chemistry and Technology, Dorodna 16, 03-195 Warsaw, Poland. E-mail: e08bp@cx1.cyf.gov.pl*

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Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

|                  |            |                  |            |
|------------------|------------|------------------|------------|
| Os(1)—Os(2)      | 2.781 (1)  | Os(2)—I          | 2.771 (1)  |
| Os(1)—I          | 2.758 (1)  | Os(2)—P(1)       | 2.399 (2)  |
| Os(1)—P(1)       | 2.403 (2)  | Os(2)—P(3)       | 2.362 (2)  |
| Os(1)—P(2)       | 2.352 (1)  | Os(2)—C(3)       | 1.833 (6)  |
| Os(1)—C(1)       | 1.843 (6)  | Os(2)—C(4)       | 1.902 (10) |
| Os(1)—C(2)       | 1.907 (10) |                  |            |
| Os(2)—Os(1)—I    | 60.0 (1)   | Os(2)—Os(1)—P(1) | 54.6 (1)   |
| I—Os(1)—P(1)     | 79.2 (1)   | Os(2)—Os(1)—P(2) | 160.4 (1)  |
| I—Os(1)—P(2)     | 107.5 (1)  | P(1)—Os(1)—P(2)  | 110.5 (1)  |
| Os(2)—Os(1)—C(1) | 101.4 (2)  | I—Os(1)—C(1)     | 160.6 (2)  |
| P(1)—Os(1)—C(1)  | 94.6 (3)   | P(2)—Os(1)—C(1)  | 91.8 (2)   |
| Os(2)—Os(1)—C(2) | 99.1 (2)   | I—Os(1)—C(2)     | 86.9 (2)   |
| P(1)—Os(1)—C(2)  | 153.7 (2)  | P(2)—Os(1)—C(2)  | 94.9 (2)   |
| C(1)—Os(1)—C(2)  | 91.2 (3)   | Os(1)—Os(2)—I    | 59.6 (1)   |
| Os(1)—Os(2)—P(1) | 54.7 (1)   | I—Os(2)—P(1)     | 79.0 (1)   |
| Os(1)—Os(2)—P(3) | 157.6 (1)  | I—Os(2)—P(3)     | 106.4 (1)  |
| P(1)—Os(2)—P(3)  | 107.9 (1)  | Os(1)—Os(2)—C(3) | 103.1 (2)  |
| I—Os(2)—C(3)     | 161.1 (2)  | P(1)—Os(2)—C(3)  | 97.4 (2)   |
| P(3)—Os(2)—C(3)  | 92.5 (2)   | Os(1)—Os(2)—C(4) | 96.6 (2)   |
| I—Os(2)—C(4)     | 85.7 (2)   | P(1)—Os(2)—C(4)  | 151.3 (2)  |
| P(3)—Os(2)—C(4)  | 99.7 (2)   | C(3)—Os(2)—C(4)  | 89.3 (3)   |
| Os(1)—I—Os(2)    | 60.4 (1)   | Os(1)—P(1)—Os(2) | 70.8 (1)   |
| Os(1)—P(1)—C(16) | 119.4 (2)  | Os(2)—P(1)—C(16) | 124.0 (1)  |
| Os(1)—P(1)—C(26) | 121.3 (2)  | Os(2)—P(1)—C(26) | 119.8 (2)  |
| Os(1)—P(2)—C(36) | 115.4 (1)  | Os(1)—P(2)—C(46) | 113.0 (2)  |
| Os(1)—P(2)—C(56) | 119.7 (1)  | Os(2)—P(3)—C(66) | 113.0 (1)  |
| Os(2)—P(3)—C(76) | 114.9 (2)  | Os(2)—P(3)—C(86) | 120.0 (1)  |
| Os(1)—C(1)—O(1)  | 177.5 (7)  | Os(1)—C(2)—O(2)  | 179.3 (5)  |
| Os(2)—C(3)—O(3)  | 176.1 (7)  | Os(2)—C(4)—O(4)  | 177.7 (6)  |

Interpretative Patterson (PATT; Sheldrick, 1985) structure solution and least-squares refinement of 673 parameters were performed with all non-H atoms anisotropic, except for the solvent C atoms. All H atoms were calculated, except for those of the solvent, which were ignored because of disorder ( $C-H = 0.960 \text{ \AA}$ ,  $U = 1.2U$  of attached C atom), and phenyl rings attached to P atoms, which were constrained as rigid planar hexagons ( $C-C = 1.395 \text{ \AA}$ ). SHELXTL (Sheldrick, 1985) was used for all computations.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: BK1268). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**References**

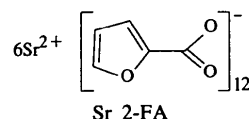
- Geoffroy, G. L., Rosenberg, S., Herlinger, A. W. & Rheingold, A. L. (1986). *Inorg. Chem.* **25**, 2916–2919.  
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 Sutton, E. E., Nivin, M. L. & Moss, J. R. (1983). *Inorg. Chim Acta*, **70**, 207–210.

**Abstract**

The title compound,  $[\text{Sr}(\text{C}_5\text{H}_3\text{O}_3)_2]$ , contains six independent formula units linked to form a three-dimensional polymer. The metal cations are eight and nine coordinate and show contacts with the carboxylate and furan O atoms. All the 2-furancarboxylate ligands are tetra- or pentadentate, similar to the structure of the corresponding calcium salt.

**Comment**

In the complexes of divalent cations with 2-furancarboxylate (2-FA), the carboxylate group donates its O atoms to the coordination sphere of the central ion, acting mainly as a bridging ligand and giving rise to a variety of coordination schemes (Paluchowska, Lis & Leciejewicz, 1994). In the case of the calcium complex with 2-FA (Paluchowska, Maurin & Leciejewicz, 1996b), the additional participation of the hetero-ring oxygen in the chelation of the cation was observed. As a continuation of our research on alkaline earth metal complexes with 2-FA, we have now studied the complex with strontium (henceforth Sr 2-FA), in which the furan O atom is also directly bonded to the cation.



Similar to Ca 2-FA, Sr 2-FA exhibits a three-dimensional polymeric structure. All the  $\text{Sr}^{2+}$  cations are located in general positions and create a network of cross-linked zigzag chains [ $\text{Sr} \cdots \text{Sr}$  distances  $3.948(2)$ – $4.456(2) \text{ \AA}$ ]. The carboxylate bridges display a tendency towards unequal Sr—O bond lengths; of 15 such bridges, six differ by more than  $0.1 \text{ \AA}$  and ten by more than  $0.05 \text{ \AA}$ . This may arise from steric effects caused by ligand packing within the network.