

**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

(Received 16 April 1996; accepted 9 October 1996)

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**

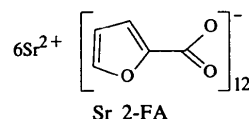
- Geoffroy, G. L., Rosenberg, S., Herlinger, A. W. & Rheingold, A. L. (1986). *Inorg. Chem.* **25**, 2916–2919.  
 Sheldrick, G. M. (1985). *SHELXTL Users Manual*. Revision 4.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.  
 Sutton, E. E., Nivin, M. L. & Moss, J. R. (1983). *Inorg. Chim Acta*, **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.

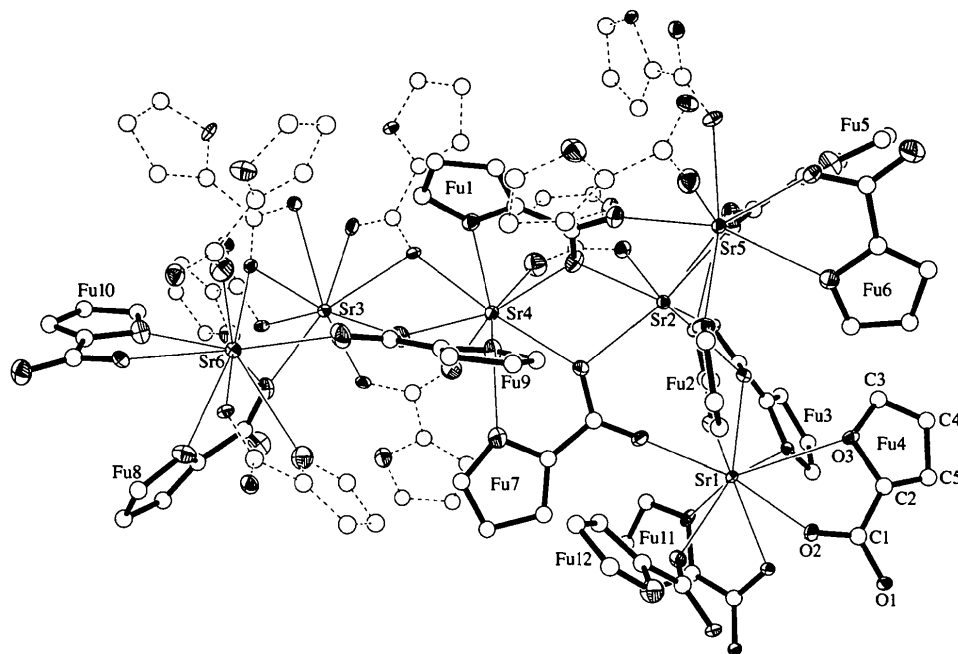


Fig. 1. Perspective view of the title structure. The six independent Sr cations are labelled. The 12 furancarboxylate ligands are indicated with solid lines and depicted as FuX (where X = 1–12). Symmetry-related ligands are drawn with dashed lines. Sr and O atoms are shown as 20% probability ellipsoids, whereas C atoms are visualized as circles. The numbering scheme of all the 2-furancarboxylate residues is shown for the Fu4 residue. The coordination bonds to the Sr atoms are shown as thin lines.

The striking feature of the Sr 2-FA structure is that unlike Sr 3-FA (Paluchowska, Maurin & Leciejewicz, 1996a), the Sr<sup>2+</sup> cations are coordinated not only by carboxylate O atoms, but also by hetero-ring O atoms (Fig. 1). The 2-FA anions, as in the case of Ca 2-FA, act as tetra- or pentadentate [Fu3, Fu4 and Fu11, where FuX (X = 1–12) are the furan rings] chelating ligands. The Sr—O<sub>carboxyl</sub> and Sr—O<sub>ring</sub> bonding distances lie in the ranges 2.406 (7)–2.906 (8) Å [ave 2.568 (7) Å] and 2.737 (7)–2.988 (9) Å [ave 2.859 (8) Å], respectively, and are consistent with the values reported for other strontium carboxylate structures (Schauer & Anderson, 1987, 1988; Schmidbaur, Bach, Wilkinson & Müller, 1989; Schmidbaur, Mikulcik & Müller, 1990). In Sr 2-FA, as in the papers cited above, the Sr atoms exhibit high coordination numbers, *i.e.* eight for Sr2, Sr3 and Sr5, and nine for Sr1, Sr4 and Sr6.

The present results confirm that strontium, as a coordination centre, has considerable bonding flexibility and can thus coordinate 2-furancarboxylate anions in a similar way to calcium.

## Experimental

Strontium bis(2-furancarboxylate) was obtained by the reaction of strontium hydroxide with a hot aqueous solution of 2-furancarboxylic acid in a 1:2 molar ratio. Air-stable crystals suitable for X-ray diffraction measurements were formed at room temperature by slow evaporation.

## Crystal data

[Sr(C<sub>5</sub>H<sub>3</sub>O<sub>3</sub>)<sub>2</sub>]

*M<sub>r</sub>* = 309.77

Monoclinic

*P*2<sub>1</sub>/*n*

*a* = 23.024 (3) Å

*b* = 13.343 (3) Å

*c* = 23.035 (3) Å

*β* = 107.00 (3)°

*V* = 6767 (2) Å<sup>3</sup>

*Z* = 24

*D<sub>x</sub>* = 1.824 Mg m<sup>-3</sup>

*D<sub>m</sub>* not measured

## Data collection

Kuma KM-4 computer-

controlled four-circle

*κ*-axis diffractometer

*ω*/2*θ* scans

Absorption correction:

*ψ* scans (Kuma Diffrac-

tion, 1989)

*T<sub>min</sub>* = 0.786, *T<sub>max</sub>* = 0.998

12 076 measured reflections

11 501 independent

reflections

## Refinement

Refinement on *F*<sup>2</sup>

*R*(*F*) = 0.0457

*wR*(*F*<sup>2</sup>) = 0.1408

Mo *Kα* radiation

*λ* = 0.71073 Å

Cell parameters from 40

reflections

*θ* = 9–13°

*μ* = 4.793 mm<sup>-1</sup>

*T* = 293 (2) K

Irregular

0.30 × 0.25 × 0.20 mm

Colourless

3792 reflections with

*I* > 2 $\sigma$ (*I*)

*R<sub>int</sub>* = 0.1558

*θ<sub>max</sub>* = 26.03°

*h* = 0 → 27

*k* = 0 → 16

*l* = -27 → 27

3 standard reflections

every 200 reflections

intensity decay: none

( $\Delta/\sigma$ )<sub>max</sub> = 0.001

$\Delta\rho$ <sub>max</sub> = 0.862 e Å<sup>-3</sup>

$\Delta\rho$ <sub>min</sub> = -1.075 e Å<sup>-3</sup>

S = 0.885

11 499 reflections

920 parameters

H atoms not refined

$$w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 33.5904P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

Extinction correction:

SHELXL93

Extinction coefficient:

0.00109 (8)

Scattering factors from

International Tables for  
Crystallography (Vol. C)

Table 1. Selected bond lengths (Å)

Sr1—O17	2.456 (7)	Sr5—O16	2.588 (7)
Sr1—O12	2.488 (7)	Sr5—O22	2.615 (6)
Sr1—O112	2.510 (6)	Sr5—O32	2.737 (7)
Sr1—O13	2.551 (6)	Sr5—O36	2.751 (7)
Sr1—O24	2.564 (6)	Sr6—O28 <sup>v</sup>	2.412 (8)
Sr1—O211	2.642 (6)	Sr6—O19	2.469 (8)
Sr1—O33	2.919 (7)	Sr6—O111 <sup>ii</sup>	2.534 (6)
Sr1—O311	2.939 (7)	Sr6—O212 <sup>vi</sup>	2.548 (6)
Sr1—O34	2.948 (6)	Sr6—O18	2.550 (7)
Sr2—O27	2.543 (6)	Sr6—O210	2.629 (6)
Sr2—O16 <sup>i</sup>	2.567 (7)	Sr6—O38	2.802 (7)
Sr2—O22	2.584 (7)	Sr6—O310	2.812 (7)
Sr2—O21	2.586 (7)	Sr6—O312 <sup>vi</sup>	2.888 (8)
Sr2—O15	2.602 (7)	O11—C11	1.300 (12)
Sr2—O23	2.640 (7)	O21—C11	1.230 (12)
Sr2—O13	2.678 (6)	O12—C12	1.185 (12)
Sr2—O25	2.906 (8)	O22—C12	1.246 (12)
Sr3—O212 <sup>ii</sup>	2.530 (6)	O13—C13	1.326 (11)
Sr3—O29	2.554 (7)	O23—C13	1.210 (11)
Sr3—O210 <sup>iii</sup>	2.555 (7)	O14—C14	1.231 (11)
Sr3—O14 <sup>ii</sup>	2.607 (6)	O24—C14	1.250 (11)
Sr3—O18	2.610 (7)	O15—C15	1.239 (12)
Sr3—O111 <sup>ii</sup>	2.716 (6)	O25—C15	1.258 (12)
Sr3—O211 <sup>ii</sup>	2.718 (6)	O16—C16	1.246 (11)
Sr3—O24 <sup>ii</sup>	2.801 (6)	O26—C16	1.217 (11)
Sr4—O26 <sup>i</sup>	2.434 (7)	O17—C17	1.230 (11)
Sr4—O110 <sup>iii</sup>	2.476 (7)	O27—C17	1.291 (11)
Sr4—O27	2.519 (7)	O18—C18	1.260 (12)
Sr4—O14 <sup>ii</sup>	2.525 (6)	O28—C18	1.266 (12)
Sr4—O29	2.573 (7)	O19—C19	1.227 (12)
Sr4—O21	2.613 (7)	O29—C19	1.298 (12)
Sr4—O31	2.810 (7)	O110—C110	1.199 (12)
Sr4—O39	2.827 (7)	O210—C110	1.283 (12)
Sr4—O37	2.988 (9)	O111—C111	1.235 (12)
Sr5—O11	2.406 (7)	O211—C111	1.190 (11)
Sr5—O23 <sup>v</sup>	2.455 (7)	O112—C112	1.258 (11)
Sr5—O15	2.459 (7)	O212—C112	1.239 (12)
Sr5—O25 <sup>v</sup>	2.526 (8)		

Symmetry codes: (i)  $\frac{3}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (ii)  $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$ ; (iii)  $\frac{3}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$ ; (iv)  $\frac{3}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$ ; (v)  $\frac{3}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$ ; (vi)  $1 - x, -y, 1 - z$ .

A preliminary reduction of the unit cell indicated the possibility of higher metric symmetry (orthorhombic C), but the  $R_{\text{int}}$  value was 0.223, cf.  $R_{\text{int}}$  was 0.038 for the chosen monoclinic cell. The intensity statistics indicated centrosymmetry. All non-H atoms were located from  $E$  maps and the subsequent  $\Delta\rho$  syntheses. Since the number of collected reflections satisfying the criterion  $I > 2\sigma(I)$  was not large enough, all furan rings were treated as residues of common geometry in the final steps of refinement [using the SADI command in SHELXL93 (Sheldrick, 1993)], and DELU was used to stabilize the anisotropic displacement parameters by restraining common components of neighbouring atoms to be approximately equal. The H atoms bonded to the furan rings were placed geometrically with a C—H distance of 0.93 Å. Their isotropic displacement parameters were taken to be 1.2 times those of their attached C atoms.

Data collection: *KM-4 Users Guide* (Kuma Diffraction, 1989). Cell refinement: *KM-4 Users Guide*. Data reduction: *KM-4 Users Guide*. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990).

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: JZ1136). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Kuma Diffraction (1989). *KM-4 Users Guide*. Version 3.1. Kuma Diffraction, Wroctaw, Poland.
- Paluchowska, B., Lis, T. & Leciejewicz, J. (1994). *Acta Cryst.* **C50**, 683–686.
- Paluchowska, B., Maurin, J. K. & Leciejewicz, J. (1996a). *Acta Cryst.* **C52**, 342–347.
- Paluchowska, B., Maurin, J. K. & Leciejewicz, J. (1996b). *Acta Cryst.* **C52**, 347–351.
- Schauer, C. K. & Anderson, O. P. (1987). *J. Am. Chem. Soc.* **109**, 3646–3656.
- Schauer, C. K. & Anderson, O. P. (1988). *Inorg. Chem.* **27**, 3118–3130.
- Schmidbaur, H., Bach, I., Wilkinson, D. L. & Müller, G. (1989). *Chem. Ber.* **122**, 1433–1438.
- Schmidbaur, H., Mikulcik, P. & Müller, G. (1990). *Chem. Ber.* **123**, 1599–1602.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

*Acta Cryst.* (1997). **C53**, 289–292

### [Bis(thiodiphenylphosphino-S)amido]-chlorobis(dimethylphenylphosphine-P)-nitridorhenium(V)

ULRICH ABRAM,<sup>a</sup> ERNESTO SCHULZ LANG<sup>a</sup> AND JONATHAN R. DILWORTH<sup>b</sup>

<sup>a</sup>Institut für Anorganische Chemie, Universität Tübingen, Auf der Morgenstelle 18, D-72076 Tübingen, Germany, and <sup>b</sup>University of Essex, Department of Chemistry and Biological Chemistry, Wivenhoe Park, Colchester CO4 3SQ, England. E-mail: ulrich.abram@uni-tuebingen.de

(Received 19 June 1996; accepted 5 November 1996)

## Abstract

The title complex,  $[\text{ReNCl}(\text{C}_{24}\text{H}_{20}\text{NP}_2\text{S}_2)(\text{C}_8\text{H}_{11}\text{P})_2]$ , is formed during the reaction of  $[\text{ReNCl}_2(\text{Me}_2\text{PhP})_3]$  with a stoichiometric amount of  $\text{Na}(\text{SPh}_2\text{PNPPH}_2\text{S})$  in dichloromethane/methanol. The equatorial sphere of the six-coordinate complex is occupied by two  $\text{Me}_2\text{PhP}$  ligands and the tetraphenyldithioimidodiphosphinate anion, which binds in a bidentate manner via the sulfur donors. The chloro ligand is arranged in a position *trans* with respect to the terminal nitrido N atom, with exceptionally long Re—Cl bond lengths [2.598(4) and 2.632(5) Å for the two crystallographically independent molecules] due to the structural *trans* influence of 'N<sup>3-</sup>'.