

Refinement

Refinement on F
 $R = 0.029$
 $wR = 0.032$
 $S = 1.0$
6810 reflections
673 parameters
H atoms not refined

$w = 1/[\sigma^2(F_o) + 0.001F_o^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.12$
 $\Delta\rho_{\text{max}} = 0.84 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.59 \text{ e } \text{\AA}^{-3}$
Extinction correction: none
Scattering factors from
SHELXTL

Table 1. Selected geometric parameters (\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.

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Acta Cryst. (1997). C53, 287–289**Hetero-Ring Oxygen Coordination to Strontium in Strontium Bis(2-furan-carboxylate)**BEATA PALUCHOWSKA,^a JAN K. MAURIN^a AND JANUSZ LECIEJEWICZ^b

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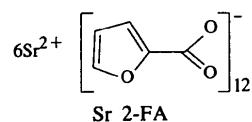
(Received 16 April 1996; accepted 9 October 1996)

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 Sr²⁺ cations are located in general positions and create a network of cross-linked zigzag chains [Sr···Sr distances 3.948 (2)–4.456 (2) \AA]. The carboxylate bridges display a tendency towards unequal Sr—O bond lengths; of 15 such bridges, six differ by more than 0.1 \AA and ten by more than 0.05 \AA . This may arise from steric effects caused by ligand packing within the network.

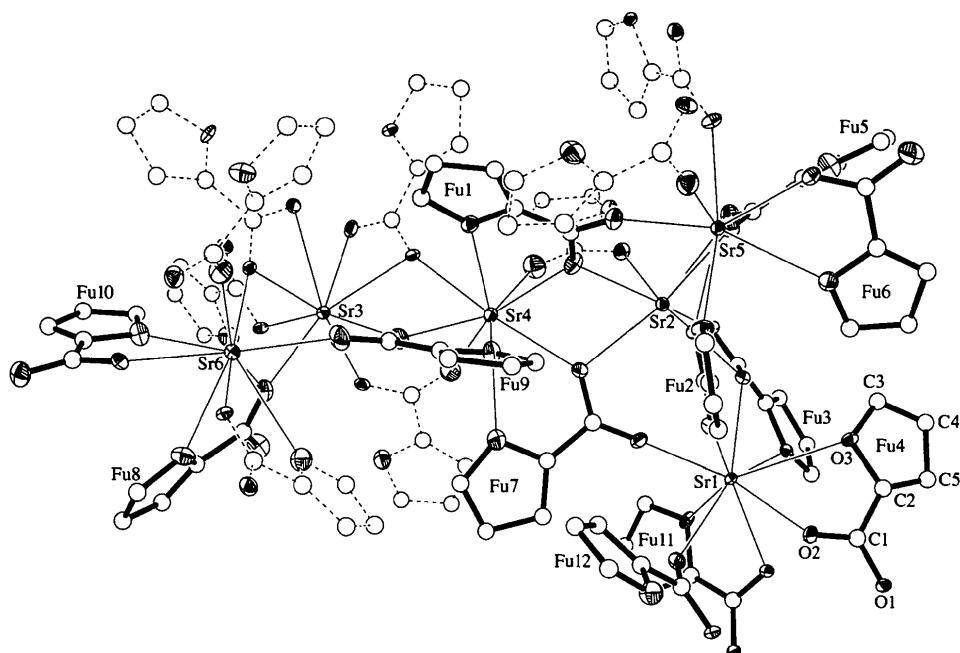


Fig. 1. Perspective view of the title structure. The six independent Sr cations are labelled. The 12 furancarboxylate ligands are indicated with solid lines and depicted as Fu X (where $X = 1-12$). Symmetry-related ligands are drawn with dashed lines. Sr and O atoms are shown as 20% probability ellipsoids, 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 Sr²⁺ 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 Fu X ($X = 1-12$) are the furan rings] chelating ligands. The Sr—O_{carboxyl} and Sr—O_{ring} 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(C₅H₃O₃)₂]

$M_r = 309.77$

Monoclinic

$P2_1/n$

$a = 23.024 (3)$ Å

$b = 13.343 (3)$ Å

$c = 23.035 (3)$ Å

$\beta = 107.00 (3)^\circ$

$V = 6767 (2)$ Å³

$Z = 24$

$D_x = 1.824$ Mg m⁻³

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 40 reflections

$\theta = 9-13^\circ$

$\mu = 4.793$ mm⁻¹

$T = 293 (2)$ K

Irregular

$0.30 \times 0.25 \times 0.20$ mm

Colourless

Data collection

Kuma KM-4 computer-controlled four-circle κ -axis diffractometer

$\omega/2\theta$ scans

Absorption correction:
 ψ scans (Kuma Diffraction, 1989)

$T_{\min} = 0.786$, $T_{\max} = 0.998$

12 076 measured reflections

11 501 independent reflections

3792 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.1558$

$\theta_{\max} = 26.03^\circ$

$h = 0 \rightarrow 27$

$k = 0 \rightarrow 16$

$l = -27 \rightarrow 27$

3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F^2

$R(F) = 0.0457$

$wR(F^2) = 0.1408$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.862$ e Å⁻³

$\Delta\rho_{\min} = -1.075$ e Å⁻³

$S = 0.885$
 11 499 reflections
 920 parameters
 H atoms not refined
 $w = 1/[\sigma^2(F_o^2) + (0.0539P)^2 + 33.5904P]$
 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 ^v | 2.412 (8) |
| Sr1—O211 | 2.642 (6) | Sr6—O19 | 2.469 (8) |
| Sr1—O33 | 2.919 (7) | Sr6—O111 ⁱⁱ | 2.534 (6) |
| Sr1—O311 | 2.939 (7) | Sr6—O212 ^{vi} | 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 ⁱ | 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 ^{vi} | 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 ⁱⁱ | 2.530 (6) | O13—C13 | 1.326 (11) |
| Sr3—O29 | 2.554 (7) | O23—C13 | 1.210 (11) |
| Sr3—O210 ⁱⁱⁱ | 2.555 (7) | O14—C14 | 1.231 (11) |
| Sr3—O14 ⁱⁱ | 2.607 (6) | O24—C14 | 1.250 (11) |
| Sr3—O18 | 2.610 (7) | O15—C15 | 1.239 (12) |
| Sr3—O111 ⁱⁱ | 2.716 (6) | O25—C15 | 1.258 (12) |
| Sr3—O211 ⁱⁱ | 2.718 (6) | O16—C16 | 1.246 (11) |
| Sr3—O24 ⁱⁱ | 2.801 (6) | O26—C16 | 1.217 (11) |
| Sr4—O26 ⁱ | 2.434 (7) | O17—C17 | 1.230 (11) |
| Sr4—O110 ⁱⁱⁱ | 2.476 (7) | O27—C17 | 1.291 (11) |
| Sr4—O27 | 2.519 (7) | O18—C18 | 1.260 (12) |
| Sr4—O14 ⁱⁱ | 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 ^{iv} | 2.455 (7) | O112—C112 | 1.258 (11) |
| Sr5—O15 | 2.459 (7) | O212—C112 | 1.239 (12) |
| Sr5—O25 ^{iv} | 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_{int} value was 0.223, cf. R_{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.

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

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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 Me_2PhP ligands and the tetraphenylthioimidodiphosphinate 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^{3-} .