After preliminary X-ray photography, lattice parameters were refined from 16 reflections with $11° < \theta < 14°$ and about 3300 X-ray intensities were measured on a Hilger & Watts computer-controlled, four-circle diffractometer up to $\theta = 25°$ using a $\theta/2\theta$ scan technique in the range $-11 \leq h \leq 9$, $-12 \leq k \leq 12$, $0 \leq l \leq 12$. Of these, 3067 were regarded as significant $[I > 3\sigma(I)]$. No corrections were made for absorption, in view of the low absorption coefficient and small crystal size (maximum dimension ca $0·5$ mm). The structure was solved by the heavy-atom Patterson method, and refined by least-squares calculations (on F), using anisotropic thermal parameters, to a final R value of $0·032$, $wR = 0·044$. H atoms were included in calculated positions with fixed isotropic thermal parameters, and not refined. A weighting scheme of the form of a four-term Chebyshev polynomial was applied in the last few cycles of refinement (235 parameters refined). $\Delta/\sigma_{max} = 0·14$. The CRYSTALS system of programs was employed.

Discussion. A drawing of the molecular structure showing the atomic numbering scheme is given in Fig. 1. Table 1 gives the atomic coordinates and U_{eq} values for the non-H atoms and Fig. 2 shows the crystal structure projected along the b axis.*

The bond distances and angles found for the present complex (Table 2) correspond closely to those found for the related chiral benzamidinato complexes, except for features relating to the chiral

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53448 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

centres, and the substituent at the skeletal C atom. The skeletal NCN angle is generally lower $(108·0°)$ when the latter substituent is methyl, than when it is phenyl (usually $110-112°$), except for the case where each N atom substituent is $CH(CH_3)C_6H_5$. Also for this acetamidinato complex the Mo atom is positioned, within experimental error, in the skeletal NCN plane whereas for the chiral complexes dihedral angles up to $5·7°$ occur. The planes of the phenyl rings of the present complex are twisted so that two ortho-H atoms lie below the plane containing the metal and skeletal NCN atoms, whereas the methyl C atom lies above this plane, thus minimizing repulsion. The packing of the molecules in the crystal is in layers parallel to $(10\bar{1})$, with adjacent molecules within the layers in opposite orientations.

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1-Carbonyl-1-η5-cyclopentadienyl-2,2,2-triphenyl-1-triphenylphosphine-μ-carboxylato-1κC:2κO:2κO′-irontin

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Abstract. $[FeSn(C_5H_5)(C_6H_5)_3(CO)(CO_2)\{P(C_6H_5)_3\}]$, $M_r = 805·27$, orthorhombic, Pbca, $a = 17·972$ (2), $b = 20·254$ (4), $c = 20·148$ (3) Å, $V = 7334·0$ Å^3, $Z = 8$, $D_x = 1·46$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0·71073$ Å), $\mu = 11·60$ cm^{-1}, $F(000) = 3264$, $T = 296$ K, 5282 unique reflections, $R = 0·038$, $wR =$

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0·040 for 3843 observed reflections with $I > 3\sigma(I)$. The coordination environment about the iron consists of a triphenylphosphine, a cyclopentadienyl ring, a carbonyl and a C-bound carboxylate. A triphenyltin unit is bound to the iron center through the O atoms of the carboxylate group giving a bimetallic complex. The Sn—O bond lengths differ by only 0·219 Å [2·123 (4) and 2·342 (4) Å] which is less than that observed for monomeric triaryltin organocarboxylates (0·45–0·81 Å). The geometry about the Sn atom is best described as distorted trigonal bipyramidal.

Introduction. Metal CO_2 complexes are of interest because of their proposed intermediacy in catalytic CO_2 activation processes. The need for binding to two different types of metal centers to achieve CO_2 activation has been noted previously (Gambarotta, Arena, Floriani & Zanuzzi, 1982). As part of a general study of bimetallic CO_2 complexes (Gibson & Ong, 1987), we have prepared the title compound (see Fig. 1): its structure determination is reported herein.

Experimental. Under nitrogen, 1·00 g of Cp-Fe(CO)(PPh₃)COOK.3H₂O (Cp = cyclopentadienyl) was added to tetrahydrofuran maintained at 195 K; 0·78 g of Ph₃SnCl was added to the slurry and the temperature of the mixture was increased to 263 K. The mixture was stirred for 30 min then chilled to 243 K and filtered. The filtrate was warmed to 268 K and the solvent removed under vacuum. The crude product was recrystallized from CH₂Cl₂/hexane; yellow crystals, m.p. 383–385 K, were collected by filtration.

X-ray diffraction data were obtained with an Enraf–Nonius CAD-4H diffractometer, Mo Kα radiation, incident-beam graphite monochromator, from a yellow block crystal (cut from a larger sample), 0·40 × 0·50 × 0·50 mm, sealed (with solvents) in a thin-walled glass capillary. The crystal was grown from a mixture of CH₂Cl₂ and pentane. The cell dimensions were taken from a least-squares refinement of the setting angles of 25 reflections whose θ angles ranged from 18 to 20°. Data were
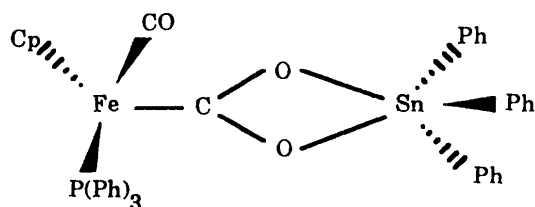

Fig. 1. Sketch of the title complex (Cp = cyclopentadienyl ring, Ph = phenyl ring).

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

	x	y	z	$B(\text{Å}^2)$
Sn	0·10721 (2)	0·21862 (2)	0·03469 (2)	3·527 (7)
Fe	0·00517 (4)	0·03735 (3)	0·13642 (4)	3·13 (1)
P1	−0·01981 (8)	0·09559 (7)	0·22597 (7)	3·41 (3)
O1	−0·1413 (2)	0·0495 (2)	0·0782 (2)	5·7 (1)
O2	0·1061 (2)	0·1409 (2)	0·1053 (2)	4·48 (8)
O3	0·0073 (2)	0·1451 (2)	0·0451 (2)	4·60 (9)
C1	−0·0836 (3)	0·0465 (3)	0·1032 (3)	3·9 (1)
C2	0·0414 (3)	0·1151 (3)	0·0913 (3)	3·6 (1)
C3	0·1081 (3)	−0·0065 (3)	0·1582 (3)	4·5 (1)
C4	0·0511 (3)	−0·0405 (3)	0·1925 (3)	4·3 (1)
C5	0·0008 (4)	−0·0664 (3)	0·1468 (3)	4·6 (1)
C6	0·0268 (3)	−0·0501 (3)	0·0830 (3)	4·7 (1)
C7	0·0935 (3)	−0·0135 (3)	0·0908 (3)	4·5 (1)
C11	0·2055 (3)	0·2628 (3)	0·0775 (3)	4·1 (1)
C12	0·2249 (3)	0·2558 (3)	0·1440 (3)	5·0 (1)
C13	0·2877 (4)	0·2845 (3)	0·1688 (3)	6·0 (2)
C14	0·3312 (4)	0·3219 (3)	0·1283 (4)	6·2 (2)
C15	0·3124 (4)	0·3305 (4)	0·0628 (4)	6·8 (2)
C16	0·2504 (4)	0·2999 (3)	0·0380 (3)	5·9 (2)
C21	0·0281 (3)	0·2963 (3)	0·0270 (3)	4·8 (1)
C22	0·0443 (4)	0·3585 (3)	0·0532 (3)	6·1 (2)
C23	−0·0097 (5)	0·4101 (3)	0·0472 (4)	8·3 (2)
C24	−0·0777 (5)	0·3962 (4)	0·0161 (4)	8·9 (2)
C25	−0·0939 (5)	0·3358 (4)	−0·0081 (4)	8·6 (2)
C26	−0·0408 (4)	0·2868 (3)	−0·0029 (3)	6·1 (2)
C31	0·1465 (3)	0·1787 (3)	−0·0569 (3)	3·7 (1)
C32	0·1363 (3)	0·2109 (3)	−0·1168 (3)	5·2 (1)
C33	0·1678 (4)	0·1863 (4)	−0·1749 (3)	6·1 (2)
C34	0·2088 (4)	0·1306 (3)	−0·1734 (3)	6·2 (2)
C35	0·2206 (4)	0·0967 (3)	−0·1157 (4)	6·7 (2)
C36	0·1890 (4)	0·1212 (3)	−0·0574 (3)	5·5 (2)
C41	−0·0450 (3)	0·1819 (3)	0·2115 (3)	3·9 (1)
C42	−0·1054 (3)	0·1953 (3)	0·1715 (3)	5·0 (1)
C43	−0·1256 (4)	0·2603 (3)	0·1567 (3)	5·4 (2)
C44	−0·0852 (4)	0·3111 (3)	0·1825 (3)	6·2 (2)
C45	−0·0256 (4)	0·2977 (3)	0·2216 (4)	7·2 (2)
C46	−0·0057 (4)	0·2330 (3)	0·2365 (3)	5·5 (2)
C51	0·0577 (3)	0·0995 (3)	0·2855 (3)	3·6 (1)
C52	0·1286 (3)	0·1171 (3)	0·2621 (3)	4·7 (1)
C53	0·1896 (3)	0·1171 (4)	0·3040 (3)	5·8 (2)
C54	0·1808 (4)	0·0979 (3)	0·3693 (3)	5·8 (2)
C55	0·1129 (4)	0·0818 (3)	0·3927 (3)	5·7 (2)
C56	0·0504 (4)	0·0820 (3)	0·3522 (3)	4·7 (1)
C61	−0·0973 (3)	0·0662 (3)	0·2783 (3)	4·1 (1)
C62	−0·1136 (3)	−0·0015 (3)	0·2818 (3)	5·5 (2)
C63	−0·1651 (4)	−0·0252 (4)	0·3262 (3)	6·6 (2)
C64	−0·2023 (4)	0·0169 (4)	0·3658 (3)	6·3 (2)
C65	−0·1913 (4)	0·0827 (4)	0·3611 (3)	6·8 (2)
C66	−0·1385 (3)	0·1090 (3)	0·3178 (3)	5·3 (2)

collected using the $\omega/2\theta$ scan technique to a maximum 2θ of 46° over the range −19 to 0, 0 to 22, 0 to 22 for h, k, l. Three standard reflections ($\overline{4}89$, 763, 449) were measured every 60 min of exposure time and showed only statistical variation. A total of 5579 reflections were collected of which all non-standard data were unique (5282); 3843 reflections were considered observed with $I > 3\sigma(I)$ where $\sigma(I)$ was determined from counting statistics (1439 unobserved). The structure was solved by Patterson techniques (Sn atom) and subsequent least-squares and difference Fourier cycles; H atoms were included in calculated positions (C—H = 0·97 Å) with isotropic thermal parameters set to $1\cdot2B_{\text{eq}}$ of the bonded atom. Data were corrected for absorption by the empirical method (ψ scans) with minimum and maximum corrections of 0·940 and 0·999. The structure was refined by full-matrix least squares, minimizing the function $\sum w(|F_o| - |F_c|)^2$; $w = 4F_o^2/[\sigma(F_o)^2]^2$. Atomic scattering factors used were from *Interna-*

tional Tables for X-ray Crystallography (1974, Vol. IV, Table 2.2B) and were corrected for anomalous-dispersion effects [*International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)]. The model converged [max. $\Delta/\sigma = 0.90$ for a thermal parameter of C(36); av. $\Delta/\sigma < 0.2$] for 3843 reflections and 442 variables with $R = 0.038$, $wR = 0.040$, $S = 2.15$ and highest and lowest peaks on a difference Fourier map of 0.55 (7) and -0.37 (7) e Å^{-3}. All calculations were performed on a VAX 750 computer using *SDP/VAX* (Frenz, 1978). Final positional and isotropic equivalent thermal parameters for non-H atoms are given in Table 1.*

Discussion. Fig. 2 gives a view of the molecule with the atomic numbering focused on the bonding mode of the bridging CO_2 to the two metals. Selected bond lengths (Å) and angles (°) are given in Table 2.

The structure is isomorphous with the related compound $CpRe(NO)(PPh_3)CO_2SnPh_3$ (Senn, Gladysz, Emerson & Larsen, 1987) (space group *Pcab*) with some minor differences. The Sn—O bond lengths in this molecule differ by 0.219 Å [2.123 (4) and 2.342 (4) Å]. The difference is considerably greater than observed (Senn *et al.,* 1987) for the related compound $CpRe(NO)(PPh_3)CO_2SnPh_3$ (0.082 Å) but smaller than that observed for monomeric triaryltin organocarboxylates (Holmes, Day, Chandrasekhar, Vollano & Holmes, 1986) which show a difference of 0.45–0.81 Å, suggesting a

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete list of bond distances, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53408 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
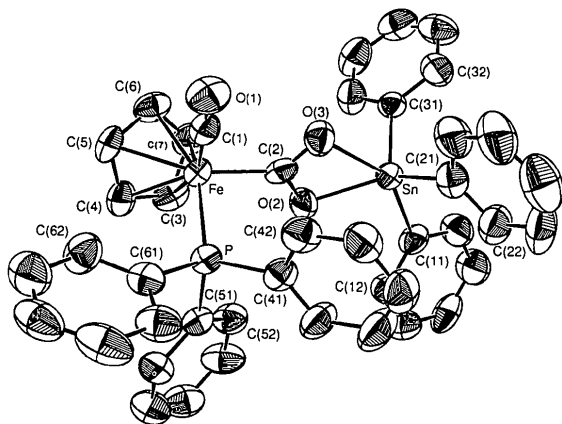
Table 2. *Selected bond lengths (Å) and angles (°)*

Sn—O2	2·123 (4)	Fe—C2	1·931 (5)
Sn—O3	2·342 (4)	P1—C41	1·829 (6)
Sn—C11	2·159 (5)	P1—C51	1·839 (5)
Sn—C21	2·128 (6)	P1—C61	1·845 (6)
Sn—C31	2·136 (5)	O2—C2	1·305 (6)
Fe—P1	2·203 (2)	O3—C2	1·270 (6)
Fe—C1	1·741 (6)		
O2—Sn—O3	57·4 (1)	C1—Fe—C2	92·4 (2)
O2—Sn—C11	92·7 (2)	Fe—P1—C41	115·6 (2)
O2—Sn—C21	126·3 (2)	Fe—P1—C51	113·8 (2)
O2—Sn—C31	107·5 (2)	Fe—P1—C61	116·7 (2)
O3—Sn—C11	148·7 (2)	C41—P1—C51	104·5 (2)
O3—Sn—C21	87·9 (2)	C41—P1—C61	102·3 (3)
O3—Sn—C31	95·2 (2)	C51—P1—C61	102·3 (2)
C11—Sn—C21	105·6 (2)	Sn—O2—C2	99·2 (3)
C11—Sn—C31	103·4 (3)	Sn—O3—C2	90·0 (4)
C21—Sn—C31	116·0 (2)	Fe—C2—O2	121·6 (4)
P1—Fe—C1	94·1 (2)	Fe—C2—O3	124·9 (4)
P1—Fe—C2	91·0 (2)	O2—C2—O3	113·4 (4)

slightly distorted bidentate carboxylate binding mode. The carboxylate C—O distances in the two bimetallic compounds are nearly the same, however [1·269 (11) and 1·313 (11) Å in the Re/Sn compound, 1·270 (6) and 1·305 (6) Å in ours].

The geometry about the Sn atom can be described as a distorted trigonal bipyramid; the sum of the equatorial bond angles is 349·8° (quite similar to the value of 351·2° found for the Re/Sn complex). Displacement of the Sn atom from the equatorial plane was 0·393 Å towards the shorter Sn—O2 bond (compared to 0·364 Å found for the Re/Sn complex). In triaryltin organocarboxylates, the value is somewhat larger with a range of 0·543–0·693 Å. The angle between the O2—C2—O3 plane and the Fe—C1—O1 vector is 9 (3)°, only slightly greater than that in the Re/Sn compound and consistent with expected bonding modes.

Fig. 2. *ORTEP* (Johnson, 1965) plot of compound showing the atomic numbering system. The thermal ellipsoids are drawn at the 50% probability level.

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