$w = 1/[\sigma^2(F_o^2) + (0.0822P)^2 + 0.3787P]$ where $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ni1—O1	1.860 (4)	C8—N1	1.442 (7)
Ni1—N2	1.867 (5)	C8—C9	1.485 (9)
Ni1—N1	1.872 (5)	C9—N2	1.476 (7)
Ni1—S1	2.163 (3)	C10—N2	1.313 (7)
S1—C15	1.725 (6)	C10—C14	1.440 (8)
C1—O1	1.311 (7)	C10—C11	1.517 (8)
C1—C2	1.412 (8)	C13—C14	1.509 (8)
C6—C7	1.410 (9)	C14—C15	1.343 (8)
C7—N1	1.295 (7)	C15—S2	1.775 (7)
O1—Ni1—N2	178.1 (2)	C15—S1—Ni1	110.3 (2)
O1—Ni1—N1	94.0 (2)	O1—C1—C6	124.4 (5)
N2—Ni1—N1	86.3 (2)	N1—C7—C6	127.0 (6)
O1—Ni1—S1	81.87 (15)	N1—C8—C9	109.0 (5)
N2—Ni1—S1	98.0 (2)	N2—C9—C8	109.1 (5)
N1—Ni1—S1	174.09 (14)	N2—C10—C14	127.3 (5)

Data was corrected for Lorentz–polarization effects. The structure was solved by direct and Fourier methods and refined by full-matrix least-squares techniques. All H atoms were calculated for geometric idealized positions, except for H7 and H11 which were located in the difference Fourier map. H atoms, when calculated in idealized positions, were riding in the refinement with site-occupancy factors and U (or U^{ij}) fixed, but with coordinates free to refine. Structural calculations were carried out on an IBM-PC.

Data collection: Stoe IPDS software. Cell refinement: Stoe IPDS software (*CELL*). Data reduction: Stoe IPDS software (*INTEGRATE*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1482). Services for accessing these data are described at the back of the journal.

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(Ketoprofenato)trimethyltin(IV)†

M. Nawaz Tahir,[a] Dinçer Ülkü,[a]* Saqib Ali,[b] Tariq Masood,[b] Mohammad Danish[b] and Mohammad Mazhar[b]

[a]*Hacettepe University, Department of Engineering Physics, Beytepe 06532, Ankara, Turkey, and* [b]*Quaid-i-Azam University, Department of Chemistry, Islamabad 45320, Pakistan. E-mail: dulku@eti.cc.hun.edu.tr*

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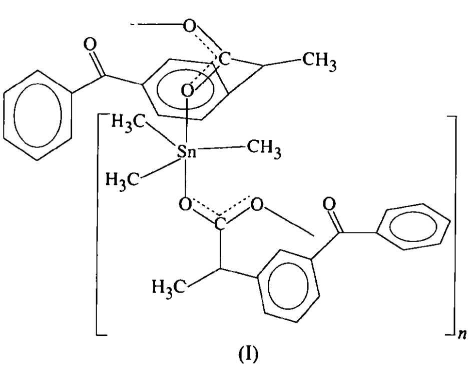
Abstract

In the title compound, [Sn(CH_3)$_3$($C_{16}H_{13}O_3$)], the Sn atom has fivefold coordination involving three methyl C atoms defining the equatorial plane of a trigonal bipyramid and two O atoms from two carboxylate groups occupying the axial positions. The Sn—C bond lengths [2.106 (6), 2.110 (5) and 2.116 (5) Å] are very similar. The two carboxylate O atoms have notably different distances to the metal atom [2.184 (3) and 2.433 (4) Å], with an O—Sn—O angle of 174.0 (1)°. Bidentate bridging of the carboxylate ligands *via* trimethyltin groups forms a polymeric structure.

Comment

There are basically two major structure types for compounds with the general formula [R_3Sn(O_2CR')] (Tiekink, 1994). The *trans*-R_3SnO_2 structure in which the bidentate carboxylate ligand bridges two Sn atoms in a distorted trigonal-bipyramidal coordination is polymeric (Danish *et al.*, 1995; Stocco *et al.*, 1996; Basu Baul & Tiekink, 1996; Ng & Kumar Das, 1996; Tahir *et al.*, 1997). The second most frequent structure type is monomeric, with an essentially fourfold coordinated Sn atom involving a secondary interaction with a second O atom (Tiekink, 1994). The title compound, (I), is a new example of a *trans* type of structure.



(I)

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† Alternative name: *catena*-poly[trimethyltin(IV)-μ-(3-benzoyl-α-methylbenzeneacetato-O^1,$O^{1'}$)].

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The coordination around the Sn atom is a distorted trigonal bipyramid, with the three methyl C atoms forming the trigonal plane and two carboxylate O atoms occupying the axial positions (Fig. 1). The Sn atom is 0.1329 (3) Å out of the equatorial plane (C17, C18 and C19) towards the carboxylate O atom with the shorter Sn—O bond length. The Sn—C bond lengths [2.106 (6), 2.110 (5) and 2.116 (5) Å] in the equatorial plane are equal within experimental error, but the Sn—O1 [2.184 (3) Å] and Sn—O2($\frac{1}{2}-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$) [2.433 (4) Å] bond distances in the *trans* coordination are very different. In the carboxylate groups, somewhat unequal C—O bond lengths are also observed [C1—O1 1.276 (6) and C1—O2 1.255 (6) Å], although the difference of 0.021 (8) Å is not significant. From these observations one could conclude that the shorter Sn—O distance indicates a covalent bond and the longer Sn—O distance is a coordination bond. The O1—Sn—O2($\frac{1}{2}-x$, $y-\frac{1}{2}$, $\frac{1}{2}-z$) angle is 174.0 (1)°. Each trimethyltin group bridges two neighbouring carboxylate ligands and each carboxylate group binds two trimethyltin moieties, forming a polymeric structure. The interplanar angle between the equatorial plane (C17, C18 and C19) and the carboxylate fragment (O1, C1 and O2) is 88.4 (3)°. The 3-benzoyl-α-methylbenzeneacetic acid (ketoprofen) ligand is not planar, the two six-membered rings, C4–C9 and C11–C16, forming an interplanar angle of 59.6 (2)°.

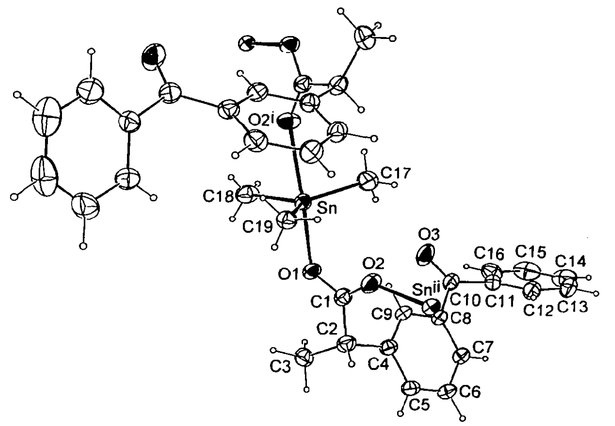

Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles with arbitrary radii.

Experimental

Trimethyltin chloride (1.99 g, 0.01 mol) and the silver salt of 3-benzoyl-α-methylbenzeneacetic acid (3.65 g, 0.01 mol) were refluxed with constant stirring in dry dichloromethane under argon for 8 h. The reaction mixture was then cooled to room temperature and the solid silver chloride removed by filtration under argon. The filtrate was treated with activated charcoal and concentrated under reduced pressure. The solid mass obtained after a week was crystallized from a dichloromethane/hexane (80/20) solvent mixture by slow evaporation under argon at room temperature (yield 83%). IR(KBr): (C—H aromatic) 3004 (*s*), (C—H aliphatic) 2927 (*m*), (C═O) 1676 (*s*), (C═C) 1600 (*s*), (COO asym) 1574 (*s*), (COO sym) 1421 (*m*), (C—C) 546 (*w*), (Sn—O) 417 (*w*) cm^{-1}; ^1H NMR ($CDCl_3$, δ = p.p.m., ^nJ = Hz): 3.76 (*q*) H2, 1.47 (*d*) H3, 7.4 (*t*) H5, 7.38 (*t*) H6, 7.4 (*t*) H7, 7.76 (*s*) H9, 7.77 (*d*) H12, 7.63 (*d*) H13, 7.54 (*d,d,d*) H14, 7.63 (*d*) H15, 7.77 H16, Hα 0.5 (*s*), ^2J [57.0]; ^{13}C NMR (δ = p.p.m., ^nJ = Hz). 179 C1, 19.68 C2, 19.37 C3, 132.4 C4, 132.4 C5, 128.4 C6, 128.6 C7, 143 C8, 130.0 C9, 196.6 C10, 138 C11, 129.2 C12, 128.3 C13, 131.6 C14, 128.3 C15, 129.2 C16, Cα −2.26, ^1J [400].

Crystal data

$[Sn(CH_3)_3(C_{16}H_{13}O_3)]$
$M_r = 417.075$
Monoclinic
$C2/c$
$a = 12.4924\ (2)$ Å
$b = 9.8064\ (2)$ Å
$c = 30.4234\ (3)$ Å
$\beta = 98.944\ (5)°$
$V = 3681.5\ (2)$ Å^3
$Z = 8$
$D_x = 1.5048$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.06–17.89°$
$\mu = 1.402$ mm^{-1}
$T = 295$ K
Prismatic
0.40 × 0.30 × 0.12 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*MolEN*; Fair, 1990)
$T_{min} = 0.833$, $T_{max} = 0.999$
3615 measured reflections
3442 independent reflections

2659 reflections with $I > 3\sigma(I)$
$R_{int} = 0.011$
$\theta_{max} = 25.01°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 11$
$l = -36 \rightarrow 36$
3 standard reflections
frequency: 120 min
intensity variation: ±1.1%

Refinement

Refinement on F
$R = 0.029$
$wR = 0.046$
$S = 1.17$
2659 reflections
208 parameters
H atoms: see below
$w = [1/\sigma(F)^2]$

$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.69$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Sn—O1	2.184 (3)	Sn—C19	2.116 (5)
Sn—O2^i	2.433 (4)	O1—C1	1.276 (6)
Sn—C17	2.110 (5)	O2—C1	1.255 (6)
Sn—C18	2.106 (6)		
O1—Sn—O2^i	174.0 (1)	O2^i—Sn—C19	89.0 (2)
O1—Sn—C17	94.2 (2)	C17—Sn—C18	117.0 (2)
O1—Sn—C18	90.4 (2)	C17—Sn—C19	124.7 (2)
O1—Sn—C19	95.9 (2)	C18—Sn—C19	117.1 (2)
O2^i—Sn—C17	85.8 (2)	Sn—O1—C1	118.8 (3)
O2^i—Sn—C18	84.2 (2)	Sn^{ii}—O2—C1	146.5 (3)

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

All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically 0.95 Å from their parent C atoms. The H atoms of the methyl C atoms and of C2 were refined for a few cycles and then a riding model was used for all H atoms, with $U_{\rm iso}(H) = 1.3U_{\rm eq}(C)$.

Data collection and cell parameters: *CAD*-4 *Express* (Enraf–Nonius, 1994). Data reduction, structure solution and refinement, molecular graphics and preparation of material for publication: *MolEN* (Fair, 1990). Other programs include *PLATON* (Spek, 1990).

The authors wish to acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1153). Services for accessing these data are described at the back of the journal.

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Calcium Potassium Tris(oxalato-O^1,O^2)-chromate(III) Pentahydrate

Vanessa Masters, Lawrence R. Gahan and
Colin H. L. Kennard

Department of Chemistry, The University of Queensland, Brisbane, Q 4072, Australia. E-mail: c.kennard@mailbox.uq.edu.au

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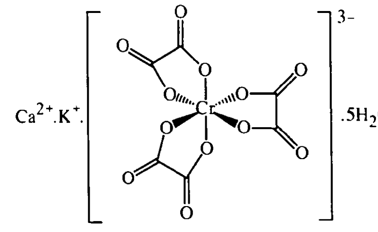
Abstract

The Cr^{III} atom in $CaK[Cr(C_2O_4)_3].5H_2O$, has a regular octahedral geometry with three oxalato groups completing the coordination. Both the calcium and potassium cations are coordinated to the O atom of the oxalate group.

Comment

This is another example of a $[Cr(ox)_3]^{3-}$ complex (ox is oxalato) but with different cations (Bulc, Golic & Siftar, 1982, 1985; Merrachi, Mentzen & Chassagneux, 1986, 1987; van Niekerk & Schoening, 1952; Taylor, 1978). This compound is a complex existing as a double salt of monovalent potassium and divalent calcium cations and the chromium(III) cation.



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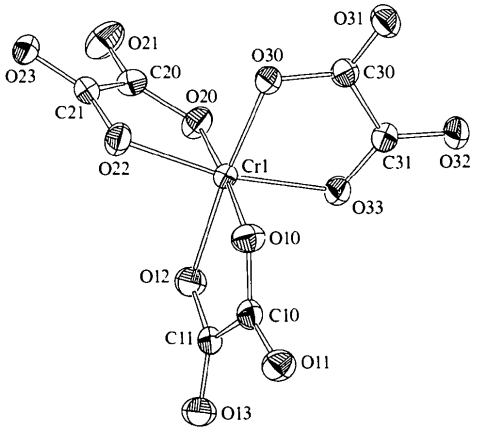

Fig. 1. *PLATON*96 (Spek, 1996) view of $[Cr(ox)_3]^{3-}$ with displacement ellipsoids drawn at the 30% probability level.

Experimental

Crystals of (I) were obtained by slow evaporation of an aqueous solution of the complex.

Crystal data

$CaK[Cr(C_2O_4)_3].5H_2O$
$M_r = 485.32$
Monoclinic
$P2_1/n$
$a = 10.923\,(4)$ Å
$b = 11.114\,(1)$ Å
$c = 13.548\,(4)$ Å
$\beta = 99.52\,(1)°$
$V = 1622.1\,(8)$ Å^3
$Z = 4$
$D_x = 1.996$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–$13°$
$\mu = 1.368$ mm^{-1}
$T = 293\,(2)$ K
Well formed needle
0.53 × 0.33 × 0.27 mm
Purple