Discussion. Fig. 2 shows the environment of the K^+ ion and Fig. 3 the contents of the unit cell. The pimelate residue has *mm* symmetry and the K^+ cation is at a site of symmetry 222. The acidic H atom H(O1) occupies a site of $2/m$ symmetry. The pimelate anions are linked into infinite chains by very short, symmetric hydrogen bonds [O—H(O1)···O 2·449 (3) Å]. There are two such chains lying in mirror planes at $y = \frac{1}{4}$ and $y = \frac{3}{4}$, related by a glide plane. Layers of pimelate residues are interspersed by layers of K^+ ions at $y = 0$ and $y = \frac{1}{2}$, such that each K^+ is eight-coordinate. The K^+ ion makes contact with two sets of four equivalent O atoms. Because of the crystal symmetry there are only two independent K^+ contacts: $K^+\cdots$O(1) 2·808 (2) and $K^+\cdots$O(2) 3·024 (2) Å. The structure is that of a typical A_2 salt, as predicted by Speakman (1972) and the IR spectrum (Hadzi, 1965), and is isostructural with potassium hydrogen glutarate. A comparison of the unit-cell parameters for KH glutarate and KH pimelate is given in Table 3. Two cell dimensions, *b* and *c*, are nearly the same, but the *a* dimension in KH pimelate is increased to accommodate the extra methylene groups.

Table 3. *Comparison of crystal data of potassium hydrogen glutarate and potassium hydrogen pimelate*

	KH glutarate	KH pimelate
a (Å)	18·476 (4)	23·551 (3)
b (Å)	7·282 (3)	7·449 (1)
c (Å)	5·133 (1)	5·069 (1)
Z	4	4
Space group	*Cmma*	*Cmma*
OH···O (Å)	2·445 (3)	2·449 (3)

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Structure of Chlorobis(*N*,*N*-dimethyldithiocarbamato)(3-methoxy-3-oxopropyl)tin(IV)

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Abstract. $[SnCl(C_3H_6NS_2)_2(C_4H_7O_2)]$, $M_r = 469·57$, monoclinic, $P2_1/n$, $a = 9·656$ (2), $b = 18·478$ (2), $c = 10·519$ (3) Å, $\beta = 101·04$ (2)°, $V = 1842$ (7) Å^3, $Z = 4$, $D_x = 1·693$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 18·34$ cm^{-1}, $F(000) = 964·00$, $T = 293$ K, final $R = 0·0488$ for 2518 unique observed reflections. Intramolecular carbonyl oxygen-to-tin coordination is not allowed in order to accommodate the two bidentate dithiocarbamate ligands, and thus the coordination geometry around the Sn^{IV} atom is a distorted octahedral arrangement with the ester group and Cl atom occupying mutually *cis* positions.

Introduction. Details of intramolecular carbonyl-to-tin coordination and the Lewis acidity of β-alkoxycarbonylethyltin(IV) chlorides (so-called 'estertins') have been published by Howie, Paterson, Wardell & Burley (1983, 1986). In a continuing effort to understand the coordination geometry of such complexes we have studied the estertin complexes with the dithiocarbamate (dtc) ligand, R_2NCS_2—, which is potentially bidentate (Jung, Jeong & Sohn, 1989*a*,*b*) and here report the crystal structure of $[CH_3OOCCH_2CH_2Sn(S_2CNMe_2)_2Cl]$. From ^{119}Sn NMR spectroscopy the authors (Jung *et al.*, 1989*a*) suggested that in this complex the Sn center is six-coordinate.

Experimental. The title compound was prepared by the authors' procedure (Jung *et al.*, 1989*a*). Analysis: calculated for $C_{10}H_{19}ClN_2O_2S_4Sn$: C 25·58; H 4·09; N 5·97; found: C 25·34; H 4·01; N 5·86%. A colorless crystal, 0·33 × 0·52 × 0·68 mm, was sealed in a glass capillary and mounted on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated molybdenum radiation. The unti-cell parameters were determined by least-squares refinement of 25 intense reflections ($11 \leq \theta \leq 15°$). Data were collected at ambient temperature [296 (2) K] in $\omega/2\theta$ scan mode using variable rates, $\theta_{max} = 25°$ for the range $0 \leq h \leq 11$, $0 \leq k \leq 22$, $-12 \leq l \leq 12$ and two standard reflections measured after every hour did not reveal any systematic variations in intensity. Max. $\sin\theta/\lambda$ reached in intensity measurement was

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Table 1. *Positional and equivalent isotropic thermal parameters for* $[CH_3OOCCH_2CH_2Sn(S_2CNMe_2)_2Cl]$

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}(Å^2)
Sn	0·2067 (1)	0·3414 (<1)	0·9147 (1)	0·0343 (4)
Cl	0·3755 (3)	0·4268 (2)	0·8497 (3)	0·0555 (18)
S(1)	0·0006 (3)	0·4130 (1)	0·7904 (3)	0·0463 (16)
S(2)	−0·0331 (3)	0·2750 (1)	0·9222 (3)	0·0453 (16)
C(5)	−0·1090 (10)	0·3458 (6)	0·8275 (9)	0·0379 (56)
N(1)	−0·2453 (9)	0·3494 (5)	0·7853 (8)	0·0467 (55)
C(6)	−0·3429 (13)	0·2915 (7)	0·8114 (14)	0·0651 (93)
C(7)	−0·3117 (13)	0·4098 (8)	0·7028 (13)	0·0651 (89)
S(3)	0·2179 (3)	0·2623 (2)	0·7195 (2)	0·0495 (21)
S(4)	0·3551 (3)	0·2259 (2)	0·9857 (3)	0·0503 (16)
C(8)	0·3057 (11)	0·1995 (6)	0·8279 (11)	0·0433 (62)
N(2)	0·3346 (11)	0·1345 (5)	0·7881 (10)	0·0559 (54)
C(9)	0·2913 (17)	0·1113 (7)	0·6523 (13)	0·0753 (79)
C(10)	0·4070 (19)	0·0796 (8)	0·8786 (15)	0·0880 (96)
C(1)	0·2313 (13)	0·3859 (6)	1·1105 (10)	0·0494 (74)
C(2)	0·3871 (13)	0·3877 (7)	1·1745 (10)	0·0541 (85)
C(3)	0·4113 (13)	0·4047 (6)	1·3200 (10)	0·0446 (71)
O(1)	0·5273 (10)	0·4168 (5)	1·3808 (8)	0·0659 (52)
O(2)	0·2953 (9)	0·4031 (5)	1·3687 (7)	0·0546 (55)
C(4)	0·3157 (16)	0·4139 (8)	1·5101 (11)	0·0684 (107)

Table 2. *Bond distances* (Å) *and bond angles* (°) *for* $[CH_3OOCCH_2CH_2Sn(S_2CNMe_2)_2Cl]$

Sn—Cl	2·458 (3)	S(3)—C(8)	1·730 (11)
Sn—S(1)	2·535 (3)	S(4)—C(8)	1·708 (11)
Sn—S(2)	2·635 (3)	N(2)—C(8)	1·320 (13)
Sn—S(3)	2·540 (3)	N(2)—C(9)	1·474 (16)
Sn—S(4)	2·599 (3)	N(2)—C(10)	1·472 (16)
Sn—C(1)	2·188 (10)	C(1)—C(2)	1·526 (16)
S(1)—C(5)	1·725 (11)	C(2)—C(3)	1·535 (14)
S(2)—C(5)	1·720 (11)	O(1)—C(3)	1·200 (13)
N(1)—C(5)	1·307 (13)	O(2)—C(3)	1·319 (14)
N(1)—C(6)	1·485 (15)	O(2)—C(4)	1·476 (12)
N(1)—C(7)	1·483 (15)		

S(1)—Sn—Cl	91·0 (1)	N(1)—C(5)—S(2)	121·6 (8)
S(2)—Sn—Cl	160·1 (1)	C(6)—N(1)—C(5)	122·3 (10)
S(2)—Sn—S(1)	69·6 (1)	C(7)—N(1)—C(5)	122·1 (10)
S(3)—Sn—Cl	90·7 (1)	C(7)—N(1)—C(6)	115·6 (10)
S(3)—Sn—S(1)	92·0 (1)	C(8)—S(3)—Sn	86·8 (4)
S(3)—Sn—S(2)	86·0 (1)	C(8)—S(4)—Sn	85·4 (4)
S(4)—Sn—Cl	104·4 (1)	S(4)—C(8)—S(3)	117·4 (6)
S(4)—Sn—S(1)	155·8 (1)	N(2)—C(8)—S(3)	120·5 (9)
S(4)—Sn—S(2)	92·8 (1)	N(2)—C(8)—S(4)	122·1 (9)
S(4)—Sn—S(3)	69·7 (1)	C(9)—N(2)—C(8)	122·3 (11)
C(1)—Sn—Cl	93·6 (3)	C(10)—N(2)—C(8)	121·7 (11)
C(1)—Sn—S(1)	102·8 (3)	C(10)—N(2)—C(9)	116·0 (10)
C(1)—Sn—S(2)	94·8 (3)	C(2)—C(1)—Sn	110·1 (7)
C(1)—Sn—S(3)	164·5 (3)	C(3)—C(2)—C(1)	113·2 (10)
C(1)—Sn—S(4)	94·8 (3)	O(1)—C(3)—C(2)	121·2 (11)
C(5)—S(1)—Sn	87·8 (3)	O(2)—C(3)—C(2)	113·8 (10)
C(5)—S(2)—Sn	84·7 (3)	O(2)—C(3)—O(1)	125·0 (10)
S(2)—C(5)—S(1)	117·8 (6)	C(4)—O(2)—C(3)	115·4 (9)
N(1)—C(5)—S(1)	120·5 (8)		

0·63 Å^{-1}; 3627 measured intensities, 2810 unique, 2518 unique observed $[I > 3\sigma(I)]$. The crystal structure was determined by the conventional heavy-atom method and Fourier techniques. All non-H atoms were refined anisotropically and H atoms were omitted from the refinement. The final cycle of refinement with 181 refined parameters assigning unit weights to all reflections yielded $R(F) = 0{\cdot}0488$, $R_G = 0{\cdot}0645$, $S = 3{\cdot}42$. Maximum height in final difference electron density map was 2·27 e Å^{-3} at 1·04 Å from the Sn atom. The maximum shift-to-e.s.d. in the final cycle of least squares was 0·1 for all parameters. All calculations were performed on an IBM 3083 computer using *SHELX76* (Sheldrick, 1976) and atomic scattering factors for all non-H atoms were supplied by the *SHELX76* system.

Discussion. Atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1, and selected bond distances and angles in Table 2.* The molecular geometry and atom-labeling scheme for the complex are shown in Fig. 1. The two dtc ligands in the present complex exhibit a bidentate bonding mode: according to X-ray studies the dtc ligands usually act in an anisobidentate manner in most mono- and diorganotin(IV) dithiocarbamate complexes (Morris & Schlemper, 1978; Kim, Ibers, Jung & Sohn, 1987; Kumar Das, Wei & Sinn, 1985; Lockhart, Manders & Schlemper, 1985), except for $[PhSnCl(S_2CNEt_2)_2]$ (Harrison & Mangia, 1976), in which four tin—sulfur bond distances [Sn—S(1), 2·528 (6); Sn—S(2), 2·661 (7); Sn—S(3), 2·551 (5); Sn—S(4), 2·593 (4) Å] are similar to those in the present complex [Sn—S(1), 2·535 (3); Sn—S(2), 2·635 (3); Sn—S(3), 2·540 (3); Sn—S(4), 2·599 (3) Å]. The bonding fashion of the ester group in the present complex is different from that in the monodithiocarbamate analog $[CH_3OOCCH_2CH_2Sn(S_2CNMe_2)Cl_2]$ (Jung *et al.*, 1989*a*) which exhibited a carbonyl oxygen-to-tin coordination resulting in a five-membered chelate ring. However, in the present compound the carbonyl group seems not to be allowed to coordinate to the Sn^{IV} atom presumably

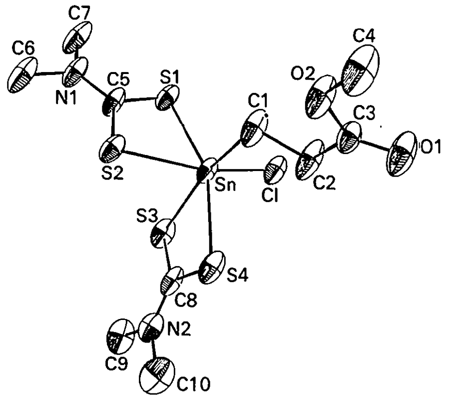

Fig. 1. *ORTEP* drawing (Johnson, 1965) of $[CH_3OOCCH_2CH_2Sn(S_2CNMe_2)_2Cl]$ with the atom-labeling scheme and thermal ellipsoids.

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* Lists of anisotropic thermal parameters and structure amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52142 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

due to chelation of the two dtc ligands. Thus the Sn atom is six-coordinated in a distorted octahedral arrangement by the ester group [C(1)], the Cl atom, and the chelating dtc residues [S(1), S(2), S(3), and S(4)], with the C(1) and Cl occupying mutually *cis* positions [C(1)—Sn—Cl, 93·6 (3)°]. Typically small bite angles [S(1)—Sn—S(2), 69·6 (1); S(3)—Sn—S(4), 69·7 (1)°] of the dtc ligands are in part responsible for the distortion of the geometry. The Sn—C(1) [2·188 (10) Å] and Sn—Cl [2·458 (3) Å] bond distances in the title compound are slightly longer than those [Sn—C(1), 2·121 (7); Sn—Cl, 2·428 (2) Å] of the monodithiocarbamate analog probably owing to relatively low Lewis acidity of the Sn atom due to the partial change of the donor atoms.

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Structure of (2,2′-Bipyridine-3,3′-dicarboxylic acid)dichlorocopper(II) Monohydrate

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Abstract. $[CuCl_2(C_{12}H_8N_2O_4)].H_2O$, $M_r = 396{\cdot}7$, orthorhombic, *Pbca*, $a = 9{\cdot}932\ (1)$, $b = 17{\cdot}560\ (2)$, $c = 17{\cdot}065\ (2)$ Å, $V = 2976{\cdot}2\ (6)$ Å^3, $Z = 8$, $D_x = 1{\cdot}77$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 18{\cdot}54$ cm^{-1}, $F(000) = 1592$, $T = 291$ K, final $R = 0{\cdot}034$ for 2543 unique observed reflections. The coordination of the Cu atom is approximately square planar with the two Cl atoms and the two N atoms of the bipyridyl ligand occupying the four coordination sites. There is an additional weak interaction between the Cu atom and the Cl and carboxylic acid carbonyl O atoms on neighbouring molecules, resulting in an approximately octahedral arrangement of ligands around the metal atom. The uncoordinated carboxylic acid groups and the water molecules form an intermolecular hydrogen-bonding network throughout the structure.

Introduction. While there are numerous studies involving the metal complexes of 2,2′-bipyridine and related ligands (McWhinnie & Miller, 1969; Fronczek, Gupta & Newkome, 1983; McMillin, Kirchoff & Goodwin, 1985), much less is known about the complexes of functionally substituted bipyridines. We are interested in the possibility of synthesizing polynuclear metal complexes with 2,2′-bipyridine-3,3′-dicarboxylic acid. However, the reaction of this ligand with copper(II) chloride resulted in the (mononuclear) title complex (1). We undertook the crystal structure determination in order to establish the nature of the complex, in particular the mode of coordination of 2,2′-bipyridine-3,3′-dicarboxylic acid to the Cu atom.

Experimental. 2,2′-Bipyridine-3,3′-dicarboxylic acid was prepared by the permanganate oxidation of 1,10-phenanthroline (Wimmer & Wimmer, 1983). A green precipitate is immediately obtained when aqueous solutions of the ligand and copper(II) chloride are mixed together in molar ratio. Upon

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