Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Fe	0.46689 (5)	0.02001 (7)	0.21446 (4)	3.11 (3)
O(1)	0.4836 (3)	0.1002 (3)	0.2961 (2)	3.8 (2)
O(2)	0.3271 (2)	0.0079 (3)	0.1853 (2)	4.1 (2)
O(3)	0.4719 (2)	−0.1427 (3)	0.2647 (2)	3.5 (2)
O(4)	0.4668 (3)	0.1736 (3)	0.1656 (2)	4.0 (2)
N(1)	0.6296 (3)	0.0190 (4)	0.2268 (2)	3.3 (2)
N(2)	0.4782 (3)	−0.0957 (4)	0.1302 (2)	3.3 (2)
N(3)	0.6634 (5)	0.1029 (7)	0.6522 (3)	7.9 (4)
C(1)	0.6526 (4)	−0.0838 (5)	0.1855 (3)	3.8 (3)
C(2)	0.5803 (4)	−0.0912 (5)	0.1223 (3)	3.8 (3)
C(3)	0.4117 (4)	−0.0407 (5)	0.0722 (3)	4.2 (3)
C(4)	0.3069 (4)	−0.0401 (5)	0.0727 (3)	4.1 (3)
C(5)	0.2403 (5)	−0.0682 (6)	0.0160 (3)	5.2 (3)
C(6)	0.1415 (6)	−0.0681 (7)	0.0116 (4)	6.4 (4)
C(7)	0.1055 (4)	−0.0420 (7)	0.0640 (4)	6.0 (3)
C(8)	0.1677 (4)	−0.0162 (6)	0.1216 (3)	4.9 (3)
C(9)	0.2705 (4)	−0.0139 (5)	0.1281 (3)	3.9 (2)
C(10)	0.4436 (4)	−0.2262 (5)	0.1351 (3)	4.0 (3)
C(11)	0.4935 (4)	−0.3022 (5)	0.1911 (3)	3.5 (2)
C(12)	0.5209 (4)	−0.4239 (6)	0.1828 (3)	4.3 (3)
C(13)	0.5541 (4)	−0.5024 (5)	0.2339 (3)	4.9 (3)
C(14)	0.5610 (4)	−0.4586 (6)	0.2953 (3)	4.9 (3)
C(15)	0.5357 (4)	−0.3383 (6)	0.3051 (3)	4.1 (3)
C(16)	0.4997 (4)	−0.2576 (5)	0.2543 (3)	3.3 (2)
C(17)	0.6719 (4)	−0.0119 (5)	0.2949 (3)	3.8 (2)
C(18)	0.6547 (4)	0.0856 (5)	0.3418 (2)	3.6 (2)
C(19)	0.7336 (4)	0.1285 (6)	0.3896 (3)	5.0 (3)
C(20)	0.7217 (5)	0.2160 (7)	0.4346 (3)	5.6 (3)
C(21)	0.6294 (5)	0.2643 (6)	0.4317 (3)	5.0 (3)
C(22)	0.5511 (4)	0.2239 (5)	0.3851 (3)	4.2 (3)
C(23)	0.5618 (4)	0.1352 (5)	0.3398 (2)	3.2 (2)
C(24)	0.6706 (4)	0.1416 (5)	0.2123 (3)	3.6 (3)
C(25)	0.6265 (4)	0.2006 (5)	0.1489 (2)	3.7 (3)
C(26)	0.6874 (4)	0.2500 (6)	0.1120 (3)	4.6 (3)
C(27)	0.6487 (7)	0.3195 (6)	0.0584 (3)	6.0 (4)
C(28)	0.5504 (7)	0.3398 (6)	0.0409 (3)	5.9 (4)
C(29)	0.4882 (5)	0.2899 (5)	0.0765 (3)	4.6 (3)
C(30)	0.5255 (4)	0.2198 (5)	0.1312 (3)	3.7 (3)
C(31)	0.610 (1)	0.1786 (8)	0.5960 (6)	10.9 (7)
C(32)	0.512 (1)	0.120 (1)	0.5687 (4)	10.5 (6)
C(33)	0.6813 (6)	−0.0250 (8)	0.6364 (4)	7.7 (5)
C(34)	0.7422 (7)	−0.0395 (9)	0.5858 (5)	9.7 (5)
C(35*a*)	0.740 (1)	0.207 (1)	0.6738 (7)	5.9 (3)
C(35)	0.783 (1)	0.125 (1)	0.6994 (7)	5.4 (3)
C(36*a*)	0.765 (1)	0.233 (1)	0.7437 (7)	5.8 (3)
C(36)	0.798 (1)	0.177 (2)	0.7365 (9)	7.4 (4)

Table 2. *Selected geometric parameters* (Å, °)

Fe—O(1)	1.917 (3)	Fe—N(2)	2.227 (4)
Fe—O(2)	1.930 (3)	N(1)—C(1)	1.496 (6)
Fe—O(3)	2.050 (4)	N(2)—C(2)	1.481 (6)
Fe—O(4)	1.958 (4)	C(1)—C(2)	1.502 (7)
Fe—N(1)	2.243 (4)		

O(1)—Fe—O(2)	103.6 (1)	O(2)—Fe—N(2)	87.2 (2)
O(1)—Fe—O(3)	85.7 (1)	O(3)—Fe—O(4)	177.8 (1)
O(1)—Fe—O(4)	95.2 (2)	O(3)—Fe—N(1)	91.1 (1)
O(1)—Fe—N(1)	88.9 (1)	O(3)—Fe—N(2)	86.9 (1)
O(1)—Fe—N(2)	167.0 (1)	O(4)—Fe—N(1)	86.9 (2)
O(2)—Fe—O(3)	91.5 (1)	O(4)—Fe—N(2)	91.9 (2)
O(2)—Fe—O(4)	90.3 (2)	N(1)—Fe—N(2)	80.7 (1)
O(2)—Fe—N(1)	167.4 (1)		

The asymmetric unit contains one [Fe^{III}(tben)]$^-$ anion and one triethylammonium cation. With the exception of the disordered C atoms [C(35), C(35*a*), C(36) and C(36*a*)], all non-H atoms were refined anisotropically. H atoms on the anion were included in idealized positions. For the cation, H atoms on resolved disordered atoms were omitted. Data collection: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SAPI91* (Fan Hai-Fu, 1991). Program(s) used to refine structure: *DIRDIF92* (Beurskens *et al.*, 1992). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71640 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1080]

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Cd(alloxanthine)$_2$(NO$_3$)$_2$(H$_2$O)$_2$

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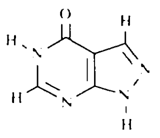
Abstract

The title complex, diaquadinitratobis{[1*H*]pyrazolo-[3,4-*d*]pyrimidine-4,6(5*H*,7*H*)-dione-*N*^{1}}cadmium-(II), was prepared as part of a study of the coordinating properties of the xanthine oxidase inhibiting molecule, alloxanthine. [Cd(NO$_3$)$_2$(C$_5$H$_4$-

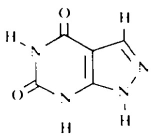
$N_4O_2)_2(H_2O)_2]$ contains monomeric centrosymmetric units with the Cd ion octahedrally coordinated by two NO_3 groups, two H_2O molecules and by two alloxanthine ligands. Alloxanthine coordinates monodentately through the pyrazole N atom N(9). The H atoms of the neutral alloxanthine molecule are bonded to the other atoms N(1), N(3) and N(8). The molecular structure is stabilized by intramolecular bifurcated hydrogen-bonding contacts of the type N—H···O, forming indirect chelates.

Comment

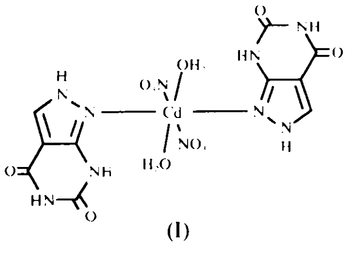
Hypoxanthine and xanthine are intermediates in the biological degradation of nucleic acids to uric acid, a sequence of reactions which are catalyzed by the Mo- and Fe-containing enzyme, xanthine oxidase. Disturbances in purine metabolism result in an increased uric acid level, thus leading to deposits of sodium hydrogen urate monohydrate crystals in joints. This disease, known as gout, is clinically treated by the antihyperuricemic drug, allopurinol, which *in vivo* is oxidized to alloxanthine (Hille & Massey, 1985; Elion, 1989). Alloxanthine is believed to remain bound to the reduced form of the Mo center of the enzyme, thus inhibiting the action of xanthine oxidase (Hille, George, Eidsness & Cramer, 1989; Hawkes, George & Bray, 1984). In this context, the coordination properties of the pyrazolopyrimidine alloxanthine are of current interest. The ligand alloxanthine (pyrazolo[3,4-*d*]pyrimidine-4,6-dione) was prepared according to the method described by Robins (1956), except that precipitation of alloxanthine dissolved in 2*N* NaOH was carried out using 2*N* H_2SO_4 instead of acetic acid. The complex $Cd(alloxanthine)_2(NO_3)_2(H_2O)_2$ (I) was synthesized by adding an excess of $Cd(NO_3)_2.4H_2O$ dissolved in water to a hot aqueous solution of alloxanthine. Transparent single crystals were obtained from the resulting mixture by slow evaporation at a temperature of 313 K.



Allopurinol



Alloxanthine



(I)

The complex (Fig. 1) is isostructural with the corresponding Cu and Co compounds (Hänggi, Schmalle & Dubler, 1993). The Cd ion exhibits a markedly distorted octahedral coordination polyhedron including two NO_3 groups, two H_2O molecules and two alloxanthine ligands. The neutral alloxanthine molecule, with the H atoms bonded to the N atoms N(1), N(3) and N(8), coordinates monodentately through the pyrazole N atom N(9). The coordination geometry is markedly distorted as displayed by the octahedron-defining angles varying from 85.40 (6) to 94.60 (6)°. Intramolecular bonding distances and angles are listed in Table 2.

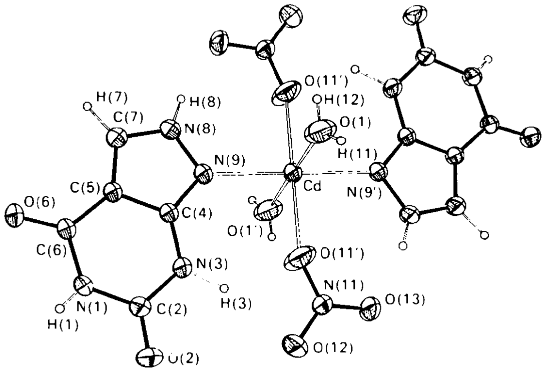


Fig. 1. Coordination polyhedron of the monomeric complex $[Cd(NO_3)_2(alloxanthine)_2(H_2O)_2]$. The thermal ellipsoids shown are drawn at the 50% probability level.

The monomeric complex unit is stabilized by two pairs of indirect chelates formed *via* intramolecular bifurcated hydrogen-bonding contacts. A relatively strong hydrogen bond N(3)—H(3)···O(11) is involved in the formation of a six-membered indirect chelate, whereas the weaker interaction N(3)—H(3)···O(12) results in an eight-membered chelate. Indirect chelation also occurs *via* the hydrogen-bonding contact N(8)—H(8)···O(11), forming a five-membered ring system. Finally, the interaction N(8)—H(8)···O(13) results in the formation of a seven-membered chelate ring. The H atoms of the H_2O molecule participate in hydrogen-bonding interactions to the exocyclic O atoms O(2) and O(6) of alloxanthine, thus interconnecting the planes containing the Cd ion; the coordinating alloxanthine ligands and the NO_3 groups are perpendicular to these planes. In addition, monomeric complex units which are related by a center of inversion at position $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$, are connected *via* two N(1)—H(1)···O(6) hydrogen-bonding contacts. A summary of bond distances and angles involving hydrogen-bonding contacts is given in Table 3.

The packing diagram of the complex is presented in Fig. 2. A stacking pattern of type II (Bugg, 1972) occurs, where the stacking bases are rotated about 180° with respect to each other. The alloxanthine ligands are infinitely stacked along the *a* axis of the unit cell with only minor direct overlap of the pyrazole rings. The shortest stacking distance between bases, calculated as the mean distance of all atoms of one molecule from the least-squares plane through the stacking molecules, is 3.382 Å. In addition, the NO_3 groups are stacked along the *a* axis of the unit cell with a mean spacing distance of 3.107 Å on top of the six-membered rings of the alloxanthine ligands.

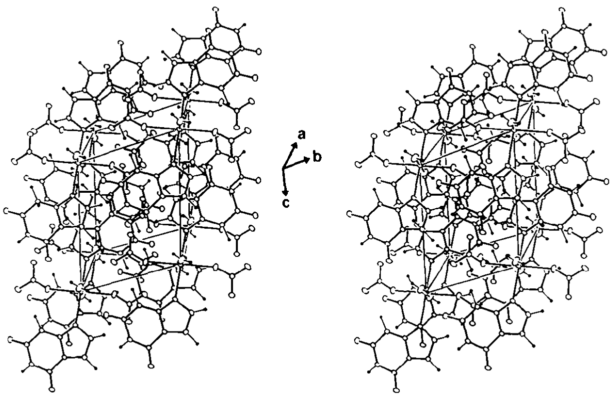


Fig. 2. *ORTEP*II (Johnson, 1976) drawing of the unit-cell contents.

The observed high stability of the enzyme-inhibitory complex of alloxanthine has been explained by monodentate coordination of alloxanthine to Mo through the pyrazole N atom N(9) stabilized by a strong N(8)—H(8)···N(enzyme) hydrogen-bonding contact (Stiefel, 1977). In contrast, Hawkes (Hawkes, George & Bray, 1984) has suggested a metal-binding pattern where alloxanthine is ligated *via* the N atom N(8). A third model of an inhibitory complex with alloxanthine coordinated bidentately through both pyrazole atoms N(8) and N(9) has been proposed by Robins (Robins *et al.*, 1985). Regarding the results of the crystal structure analysis of the alloxanthine–cadmium complex presented here and of the analyses of its Cu and Co analogues, as well as of $Zn(alloxanthine)_2Cl_2$ (Hänggi, Schmalle & Dubler, 1993), it is evident that N(9) is a preferred metal-binding site of neutral alloxanthine. Therefore, these crystallographic data strongly support those structural models for the mechanism of action of alloxanthine inhibiting the enzyme xanthine oxidase which suggest coordination of alloxanthine to the metal *via* N(9).

Experimental

Crystal data

$[Cd(NO_3)_2(C_5H_4N_4O_2)_2(H_2O)_2]$
$M_r = 576.68$
Triclinic
$P\bar{1}$
$a = 7.135$ (2) Å
$b = 7.946$ (4) Å
$c = 9.039$ (1) Å
$\alpha = 109.25$ (3)°
$\beta = 99.62$ (2)°
$\gamma = 104.44$ (2)°
$V = 450.8$ (6) Å^3
$Z = 1$

$D_x = 2.124$ Mg m^{-3}
$D_m = 2.13$ Mg m^{-3}
D_m measured by flotation in $CHCl_3/CH_2I_2$
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8.1$-13.6°
$\mu = 1.295$ mm^{-1}
$T = 295$ K
Plate
0.50 × 0.46 × 0.09 mm
Colorless

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: analytical
$T_{min} = 0.440$, $T_{max} = 0.659$
5854 measured reflections
5562 independent reflections
4686 observed reflections
$[I \geq 3\sigma(I)]$

$R_{int} = 0.013$
$\theta_{max} = 40$°
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 0$
$l = -16 \rightarrow 16$
3 standard reflections monitored every 300 reflections
intensity variation: $< \pm 2\%$

Refinement

Refinement on F
$R = 0.033$
$wR = 0.039$
$S = 0.535$
4686 reflections
176 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o) + 0.016F_o^2]$
$(\Delta/\sigma)_{max} = 0.02$

$\Delta\rho_{max} = 0.84$ e Å^{-3}
$\Delta\rho_{min} = -1.82$ e Å^{-3}
Extinction correction: empirical, secondary
Extinction coefficient: 3.7×10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cd	0	0	1	0.03156 (4)
O(1)	0.2839 (3)	−0.0379 (3)	1.1148 (2)	0.0543 (5)
N(11)	0.1343 (3)	0.4745 (2)	1.2325 (2)	0.0303 (4)
O(11)	0.1387 (3)	0.3233 (2)	1.1323 (2)	0.0613 (5)
O(12)	0.2048 (3)	0.6216 (2)	1.2122 (2)	0.0510 (5)
O(13)	0.0674 (3)	0.4763 (2)	1.3505 (2)	0.0460 (4)
N(1)	0.3908 (3)	0.4591 (2)	0.6630 (2)	0.0308 (4)
C(2)	0.3435 (3)	0.5168 (2)	0.8106 (2)	0.0301 (4)
O(2)	0.3769 (3)	0.6840 (2)	0.8901 (2)	0.0445 (4)
N(3)	0.2586 (3)	0.3763 (2)	0.8592 (2)	0.0318 (4)
C(4)	0.2241 (3)	0.1915 (2)	0.7647 (2)	0.0262 (4)
C(5)	0.2785 (3)	0.1369 (2)	0.6177 (2)	0.0260 (4)
C(6)	0.3694 (3)	0.2786 (2)	0.5618 (2)	0.0275 (4)
O(6)	0.4249 (2)	0.2491 (2)	0.4362 (2)	0.0393 (4)
C(7)	0.2200 (3)	−0.0589 (2)	0.5575 (2)	0.0312 (4)
N(8)	0.1378 (3)	−0.1103 (2)	0.6644 (2)	0.0333 (4)
N(9)	0.1373 (3)	0.0407 (2)	0.7943 (2)	0.0320 (4)

Table 2. *Selected geometric parameters* (Å, °)

Cd—N(9)	2.317 (1)	C(7)—N(8)	1.332 (2)
Cd—O(1)	2.253 (2)	N(8)—N(9)	1.376 (2)
Cd—O(11)	2.307 (2)	N(9)—C(4)	1.336 (2)
N(1)—C(2)	1.389 (2)	C(2)—O(2)	1.222 (2)
C(2)—N(3)	1.372 (2)	C(6)—O(6)	1.233 (2)
N(3)—C(4)	1.367 (2)	N(3)—N(9)	2.419 (2)
C(4)—C(5)	1.408 (2)	N(11)—O(11)	1.256 (2)
C(5)—C(6)	1.436 (2)	N(11)—O(12)	1.238 (2)
C(6)—N(1)	1.377 (2)	N(11)—O(13)	1.236 (2)
C(5)—C(7)	1.390 (3)		
N(9)—Cd—O(1)	89.65 (8)	C(7)—N(8)—N(9)	112.9 (2)
N(9)—Cd—O(1′)	90.35 (8)	N(8)—N(9)—C(4)	104.4 (1)
N(9)—Cd—O(11)	85.40 (6)	N(9)—C(4)—C(5)	110.8 (2)
N(9)—Cd—O(11′)	94.60 (6)	N(3)—C(4)—N(9)	126.9 (2)
O(1)—Cd—O(11)	90.96 (9)	C(6)—C(5)—C(7)	135.0 (2)
O(1)—Cd—O(11′)	89.04 (9)	N(1)—C(2)—O(2)	120.7 (2)
C(6)—N(1)—C(2)	128.0 (2)	N(3)—C(2)—O(2)	123.2 (2)
N(1)—C(2)—N(3)	116.1 (2)	N(1)—C(6)—O(6)	120.9 (2)
C(2)—N(3)—C(4)	120.5 (2)	C(5)—C(6)—O(6)	125.6 (2)
N(3)—C(4)—C(5)	122.3 (1)	Cd—N(9)—N(8)	121.7 (1)
C(4)—C(5)—C(6)	119.4 (2)	Cd—N(9)—C(4)	133.9 (1)
C(5)—C(6)—N(1)	113.5 (1)	O(11)—N(11)—O(12)	117.9 (2)
C(4)—C(5)—C(7)	105.5 (1)	O(11)—N(11)—O(13)	120.7 (2)
C(5)—C(7)—N(8)	106.3 (2)	O(12)—N(11)—O(13)	121.4 (2)

Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(1)	H(1)	O(6)	0.78 (3)	2.08 (3)	2.856 (2)	175 (3)
N(3)	H(3)	O(11)	1.11 (4)	2.02 (4)	2.866 (3)	131 (3)
N(3)	H(3)	O(12)	1.11 (4)	2.20 (4)	3.288 (2)	166 (3)
N(8)	H(8)	O(11)	0.93 (3)	2.88 (3)	3.425 (3)	119 (2)
N(8)	H(8)	O(13)	0.93 (3)	1.94 (3)	2.859 (2)	166 (3)
O(1)	H(11)	O(2)	0.81 (4)	2.09 (4)	2.770 (3)	141 (4)
O(1)	H(12)	O(6)	0.84 (4)	2.18 (4)	2.857 (3)	138 (4)

The structure determination was started with the atomic coordinates of the non-H atoms of the isostructural Cu complex (Hänggi, Schmalle & Dubler, 1993); refinement was by full-matrix least-squares calculations applying the program *MolEN* (Fair, 1990). After anisoptropic refinement, all H atoms could be located in the difference Fourier maps and were included in the refinement with variable positional and isotropic displacement parameters.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71589 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1062]

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n-Butyltris(2-pyrimidinethiolato-*N*,*S*)tin(IV)

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Abstract

The crystal structure of the title compound contains two crystallographically independent molecules. The 2-pyrimidinethiolato ligands (SPym) are bidentate. Sn—S bond distances range from 2.445 (4) to 2.561 (5) Å and Sn···N coordination is inferred from short Sn—N distances [2.470 (5)–2.622 (6) Å]. The coordination polyhedron around Sn in both molecules can be considered as a distorted pentagonal bipyramid. The pentagonal plane in each molecule is formed by two SPym ligands, with S atoms in *cis* positions, and the N atom of the third SPym ligand. The S atom of this ligand, which is arranged nearly perpendicular to the equatorial plane, and the C(*n*-butyl) atom occupy the axial positions. Intermolecular distances shorter than the sum of the van der Waals radii were not found.

Comment

In diorganotin(IV) complexes of 2-mercaptopyridine (HSPy) and 2-mercaptopyrimidine (HSPym), the ligands form four-membered bidentate chelate rings with strong intramolecular Sn—N coordination (Mullins, 1979; Schmiedgen, 1992). For monoorganotin(IV) complexes with the same ligands, *e.g.* *R*Sn(SPym)$_3$ (*R* = methyl, *n*-butyl, phenyl), an analogous bonding behavior and a distorted pentagonal bipyramidal coordination polyhedron around Sn has