

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Na(1)	0.4734 (1)	0.3787 (4)	0.1169 (2)	0.041 (1)
S(1)	0.10053 (7)	0.3722 (3)	0.2621 (2)	0.036 (1)
O(1)	0.0818 (2)	0.5065 (7)	0.2113 (6)	0.064 (8)
O(2)	0.0962 (2)	0.3753 (8)	0.3854 (4)	0.062 (1)
O(3)	0.0822 (2)	0.2324 (7)	0.2152 (6)	0.063 (2)
O(4)	1/2	0.5673 (8)	1/4	0.040 (2)
O(5)	1/2	0.1900 (8)	1/4	0.046 (2)
O(6)	0.4055 (2)	0.4072 (7)	0.2721 (5)	0.054 (1)
O(7)	0.4491 (2)	0.6008 (6)	-0.0094 (4)	0.048 (1)
O(8)	0.4350 (2)	0.1853 (7)	0.0245 (5)	0.067 (2)
N(1)	0.3334 (2)	0.2394 (8)	0.1508 (6)	0.051 (2)
C(1)	0.3037 (2)	0.3347 (8)	0.0953 (5)	0.034 (2)
C(2)	0.3203 (3)	0.4307 (9)	0.0088 (6)	0.038 (2)
C(3)	0.2915 (3)	0.5348 (9)	-0.0431 (6)	0.042 (2)
C(4)	0.2449 (3)	0.5431 (8)	-0.0058 (6)	0.033 (1)
C(5)	0.1782 (3)	0.4572 (8)	0.1257 (6)	0.028 (2)
C(6)	0.1604 (2)	0.3674 (8)	0.2136 (5)	0.025 (2)
C(7)	0.1882 (2)	0.2590 (8)	0.2674 (6)	0.032 (2)
C(8)	0.2347 (3)	0.2495 (8)	0.2298 (6)	0.029 (2)
C(9)	0.2548 (2)	0.3429 (7)	0.1388 (5)	0.025 (2)
C(10)	0.2264 (3)	0.4468 (7)	0.0841 (5)	0.025 (2)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Na(1)—O(4)	2.323 (7)	C(1)—C(2)	1.42 (1)
Na(1)—O(5)	2.321 (8)	C(1)—C(9)	1.54 (1)
Na(1)—O(6)	2.80 (1)	C(2)—C(3)	1.33 (1)
Na(1)—O(7)	2.473 (7)	C(3)—C(4)	1.45 (1)
Na(1)—O(7')	2.666 (8)	C(4)—C(10)	1.47 (1)
Na(1)—O(8)	2.217 (8)	C(5)—C(6)	1.42 (1)
S(1)—O(1)	1.385 (7)	C(6)—C(7)	1.34 (1)
S(1)—O(2)	1.451 (6)	C(7)—C(8)	1.45 (1)
S(1)—O(3)	1.405 (7)	C(8)—C(9)	1.49 (1)
N(1)—C(1)	1.31 (1)	C(9)—C(10)	1.33 (1)
O(1)—S(1)—O(2)	109.7 (5)	C(6)—C(5)—C(10)	127.1 (8)
O(1)—S(1)—O(3)	115.8 (4)	S(1)—C(6)—C(5)	128.0 (6)
O(1)—S(1)—C(6)	102.9 (4)	S(1)—C(6)—C(7)	113.6 (6)
O(2)—S(1)—O(3)	109.5 (5)	C(5)—C(6)—C(7)	118.4 (8)
O(2)—S(1)—C(6)	118.1 (3)	C(6)—C(7)—C(8)	115.0 (8)
O(3)—S(1)—C(6)	100.7 (4)	C(7)—C(8)—C(9)	128.1 (8)
N(1)—C(1)—C(2)	117.7 (9)	C(1)—C(9)—C(8)	129.6 (7)
N(1)—C(1)—C(9)	116.1 (8)	C(1)—C(9)—C(10)	114.4 (7)
C(2)—C(1)—C(9)	125.9 (7)	C(8)—C(9)—C(10)	116.0 (8)
C(1)—C(2)—C(3)	118.4 (9)	C(4)—C(10)—C(5)	126.8 (7)
C(2)—C(3)—C(4)	116.9 (9)	C(4)—C(10)—C(9)	117.9 (8)
C(3)—C(4)—C(10)	126.4 (8)	C(5)—C(10)—C(9)	115.3 (7)

Symmetry code: (i) $1 - x, 1 - y, -z$.Table 3. Hydrogen-bonding geometry (\AA , $^\circ$)

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
O(6)—H(61)···N(1)	0.75	2.11 (6)	2.79 (1)	137.8 (6)
O(4)—H(41)···O(3')	0.75	2.10 (6)	2.82 (1)	161.5 (6)
O(5)—H(51)···O(1 ⁱⁱ)	0.68	2.23 (6)	2.91 (1)	172.2 (6)
O(6)—H(62)···O(3 ⁱⁱⁱ)	0.72	2.26 (7)	2.83 (1)	136.5 (7)
O(7)—H(72)···O(2 ⁱⁱⁱ)	0.96	2.59 (6)	3.12 (1)	170.2 (6)
O(8)—H(82)···O(3 ⁱⁱⁱ)	0.81	2.11 (6)	2.87 (1)	157.4 (5)
O(8)—H(81)···O(2 ⁱⁱⁱ)	0.78	2.27 (6)	3.04 (1)	168.9 (6)

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

The title structure was solved by direct methods. Full-matrix least squares were used for refinement on *F*, with anisotropic displacement parameters for non-H atoms and isotropic displacement parameters for H atoms. Calculations were performed on a VAX 3100 computer using the *MolEN* (Fair, 1990) program package for structure solution and refinement.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1205). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Chlorotris(*p*-methylsulfonylphenyl)tin(IV)

IVOR WHARF, ANNE-MARIE LEBUIS AND
HENRY LAMPARSKI

Chemistry Department, McGill University, 801 Sherbrooke St. W., Montreal, Quebec, Canada H3A 2K6. E-mail: wharf@omc.lan.mcgill.ca

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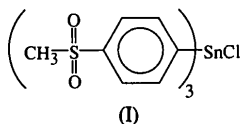
Abstract

The title compound, $[\text{SnCl}(\text{C}_7\text{H}_7\text{O}_2\text{S})_3]$, crystallizes in an unusual space group for Ar_3SnCl compounds, namely, *Pna*₂. The structure has $[-\text{O}=\text{S}(\text{CH}_3)(\text{O})-\text{C}_6\text{H}_4(\text{Ar})_2\text{Sn}(\text{Cl})-]_n$ polymer chains zigzagging along the screw axis parallel to *c*, which are then closely packed to create a dense structure, unlike the open lattice found for the *tert*-butyl analogue. The molecular unit has distorted *C*_s symmetry, with one aryl group forming the pseudo-mirror plane. The environment around the Sn atom is quasi-five-coordinate, compared with that found in either four-coordinate monomeric Ph_3SnCl or in five-coordinate $\text{Ph}_3\text{SnCl}\cdot\text{L}$.

Comment

Triaryltin halides (Ar₃SnX) usually crystallize in one of the ubiquitous monoclinic space groups (*P2₁/a*, *P2₁/c* or *P2₁/n*) for a variety of *para* and *ortho* substituents (Preut & Huber, 1979; Tse, Lee & Gabe, 1986; Reuter & Puff, 1989; Wharf & Simard, 1991; Howie, Ross, Wardell & Low, 1994), although recently an unexpected trigonal polymorph of Ph₃SnCl has been reported (Ng, 1995*b*) and triphenyltin iodide has a triclinic (*P* $\bar{1}$) unit cell for the two polymorphs identified so far (Simard & Wharf, 1994; Ng, 1995*a*).

Our interest, to date, has been in the effect of *para* substituents on the properties and structures of both Ar₄Sn and Ar₃SnX systems. Usually, increasing the size of the *para* substituent (e.g. CH₃ or C₂H₅) causes a decrease in melting point, but with *tert*-butyl as the *para* substituent, both Ar₄Sn and Ar₃SnCl have high melting points. The crystal structure of [*p*-(CH₃)₃C(C₆H₄)₃SnCl shows a relatively open but well organized lattice due to strong interactions between overlapping phenyl rings (Wharf, Simard & McGinn, 1995). Since the CH₃SO₂ group is isosteric with *tert*-butyl, the crystal structure of the title compound, (1), which also has a high melting point, was determined for comparison with its *tert*-butyl analogue.



The title compound crystallizes in a space group so far unreported for triaryltin compounds, *Pna2₁*, with a tightly packed lattice, contrasting with the much more open structure found for the *tert*-butyl analogue, in spite of their similar physical properties. In fact, the density of compound (1) is even higher than that of another closely packed structure, Ph₃SnCl (1.544 Mg m⁻³; Tse, Lee & Gabe, 1986). All intermolecular interatomic distances are greater than the sum of the van der Waals radii, except for Sn—O11ⁱ of 3.046(5) Å which is indicative of a weak tin–oxygen coordinative bond. The principal structural motif has an [—O=S(CH₃)(O)C₆H₄(Ar)₂Sn(Cl)—]_n polymer chain zigzagging along the screw axis parallel to *c* (Fig. 1), giving an example of a Class 3 organotin polymer (Molloy & Quill, 1985). The methylsulfonyl groups on rings 2 and 3 pack with molecules in adjacent chains, with the CH₃ on group 3 interacting with its symmetry-related equivalent.

The environment around the Sn atom (Fig. 2) shows a distorted *C_s* geometry for the molecule, with the pseudo-mirror plane comprised of an O=S(CH₃)C₆H₄SnCl moiety formed using aryl group 3. This contrasts with the distorted or regular *C₃* propeller geometry usually found for Ar₃SnX systems which molecular-mechanics calculations show is indeed the most stable

conformation. A metastable conformer close in energy, however, has *C_s* symmetry, which although distorted in compound (1), may be required for the polymer chain to form. All three aryl groups have an S=O bond almost coplanar with the phenyl ring, with no disorder of either

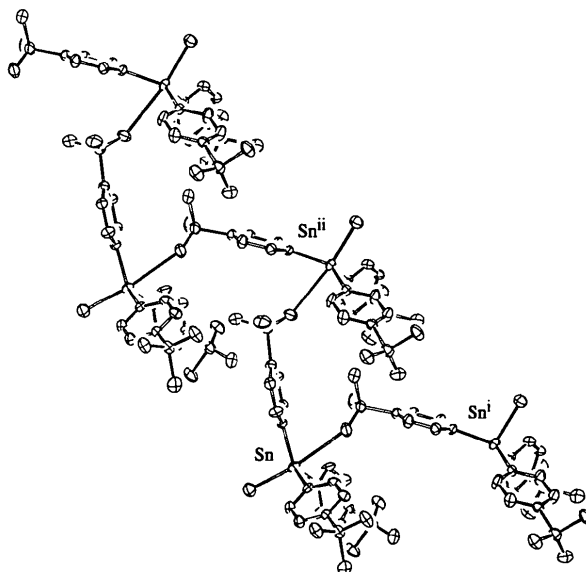


Fig. 1. ORTEP plot (Johnson, 1976) showing one chain of molecules along the 2₁ axis. Ellipsoids are shown at the 30% probability level. [Symmetry codes: (i) 1 - *x*, 2 - *y*, *z* - ½; (ii) 1 - *x*, 2 - *y*, *z* + ½.]

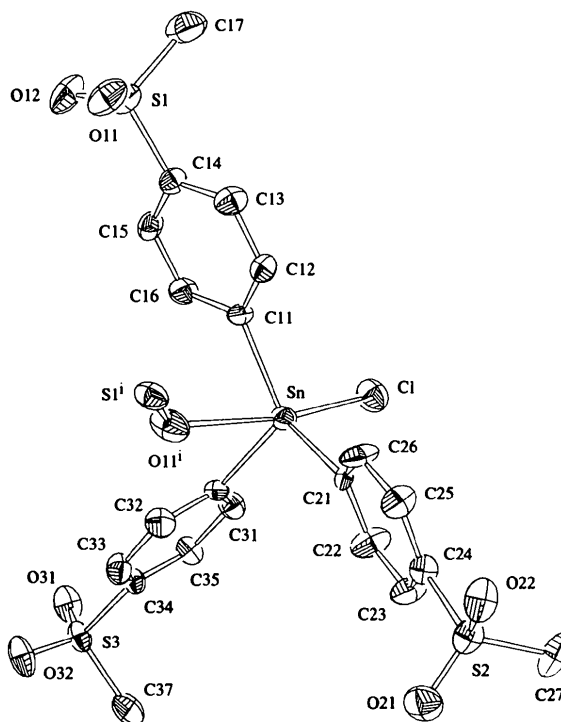


Fig. 2. ORTEP plot (Johnson, 1976) of the molecular unit showing the atom-numbering scheme. Ellipsoids are shown at the 50% probability level.

the methyl group or the second O atom, which is not the case for the Ar₄Sn analogue, tetrakis(*p*-methylsulfonylphenyl)tin (Wharf, Simard & Lamparski, 1990).

We believe compound (1) to be the first reported stable intermolecular organotin–sulfone adduct, although the intramolecular adduct *p*-MeC₆H₄SO₂(CH₂)₃SnPh₂I is known (Howie & Wardell, 1994) and has a smaller tin–oxygen distance of 2.625 Å. Even this Sn–O bond, however, does not persist in solution, consistent with the earlier results of Davies & Price (1983) and the fact that sulfones, *e.g.* sulfolane (DN = 14.8) (Gutmann, 1976), are weaker donors than sulfoxides, *e.g.* Me₂SO (DN = 29.8), or phosphine oxides, *e.g.* Ph₃PO (Spencer *et al.*, 1989). The detailed molecular geometry around the Sn atom (Table 2) for compound (1) is between that of four-coordinate Ph₃SnCl and five-coordinate neutral adducts such as Ph₃SnCl.L, where L is Ph₃P=O (Ng & Kumar Das, 1992) or O=S(Pr)CH₂ (Filgueiras, Holland, Johnson & Raithby, 1982). Thus, in compound (1), tin may be considered quasi-five-coordinate, as in Ph₃SnNCO (Wharf, Piehler, Sailofsky, Onyszczuk & Simard, 1987), with the weak Sn–O interaction stabilized in the solid state by a polymeric structure.

Experimental

All experimental procedures and materials were as reported previously (Wharf, 1989; Wharf, Simard & Lamparski, 1990). Solid *m*-chloroperbenzoic acid (85%, 4.95 g) was added over a period of 1.5 h to chlorotris(*p*-methylthiophenyl)tin (1.88 g) in dichloromethane (20 ml) at 265 K. The mixture was further diluted (100 ml, CH₂Cl₂) and then stirred at room temperature for 4 h. Further dichloromethane was added to dissolve the solid that formed, followed by addition of dry ether to the filtrate which gave the title product (yield 80%, m.p. 630 K). Analysis found, C 40.53, H 3.39%, calculated for C₂₁H₂₁ClO₆S₃Sn, C 40.70, H 3.42%; IR (Nujol mull, cm⁻¹): 1301 [ν_{as}(SO₂)], 1151–1145 [ν_s(SO₂)], 771 [ν(CH₃S)]. Slow evaporation of an acetone–chlorobenzene solution gave the crystals used in this study.

Crystal data

[SnCl(C₇H₇O₂S)₃]

M_r = 619.70

Orthorhombic

*Pna*2₁

a = 10.6814 (15) Å

b = 20.220 (5) Å

c = 11.467 (2) Å

V = 2476.6 (8) Å³

Z = 4

D_x = 1.662 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.7107 Å

Cell parameters from 24 reflections

θ = 12–14°

μ = 1.427 mm⁻¹

T = 293 (2) K

Plate

0.47 × 0.28 × 0.05 mm

Colourless

Data collection

Rigaku AFC-6 diffractometer

3213 observed reflections
[*I* > 2σ(*I*)]

ω/2θ scans

Absorption correction:

ψ scan (NRCVAX; Gabe, Le Page, Charland, Lee & White, 1989)

T_{min} = 0.6919, *T_{max}* = 0.9991

16 670 measured reflections

4373 independent reflections

Refinement

Refinement on *F*²

R(*F*) = 0.0478

w*R*(*F*²) = 0.0827

S = 1.027

4373 reflections

295 parameters

H atoms riding, C–H =

0.93–0.96 Å

w = 1/[σ²(*F_o*²) + (0.0297*P*)²]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.002

Δρ_{max} = 0.550 e Å⁻³

Δρ_{min} = -0.428 e Å⁻³

R_{int} = 0.073

θ_{max} = 24.94°

h = -12 → 12

k = -23 → 24

l = -13 → 13

3 standard reflections

monitored every 200

reflections

intensity decay: 1.2%

Extinction correction:

SHELXL93

Extinction coefficient:

0.00078 (12)

Atomic scattering factors

from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Absolute configuration:

Flack (1983)

Flack parameter = -0.05 (3)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
Sn	0.86063 (4)	0.98001 (2)	0.00140 (4)	0.03611 (14)
Cl	1.0684 (2)	0.95201 (11)	0.0572 (2)	0.0560 (6)
S1	0.5391 (2)	1.02347 (12)	0.4911 (3)	0.0461 (5)
S2	0.7447 (2)	0.75409 (11)	-0.4071 (2)	0.0469 (5)
S3	0.9432 (2)	1.25979 (10)	-0.2968 (2)	0.0406 (5)
O12	0.5456 (5)	1.0921 (3)	0.5219 (6)	0.068 (2)
O11	0.4181 (5)	0.9961 (3)	0.4644 (4)	0.062 (2)
O21	0.7170 (5)	0.7941 (3)	-0.5067 (7)	0.065 (2)
O22	0.6512 (5)	0.7083 (3)	-0.3670 (5)	0.058 (2)
O32	0.8223 (5)	1.2816 (3)	-0.3345 (5)	0.056 (2)
O31	1.0175 (5)	1.3029 (3)	-0.2268 (5)	0.052 (2)
C11	0.7694 (7)	0.9902 (4)	0.1639 (6)	0.033 (2)
C12	0.7066 (7)	0.9391 (4)	0.2163 (7)	0.038 (2)
C13	0.6389 (7)	0.9476 (4)	0.3184 (7)	0.044 (2)
C14	0.6349 (7)	1.0098 (4)	0.3683 (6)	0.035 (2)
C15	0.6998 (7)	1.0619 (4)	0.3196 (6)	0.039 (2)
C16	0.7644 (8)	1.0518 (4)	0.2178 (6)	0.042 (2)
C17	0.6048 (9)	0.9765 (5)	0.6041 (8)	0.073 (4)
C21	0.8203 (7)	0.9013 (4)	-0.1186 (6)	0.031 (2)
C22	0.8931 (8)	0.9013 (5)	-0.2174 (7)	0.056 (3)
C23	0.8707 (8)	0.8555 (4)	-0.3055 (7)	0.055 (2)
C24	0.7812 (7)	0.8084 (4)	-0.2890 (7)	0.037 (2)
C25	0.7103 (8)	0.8069 (4)	-0.1895 (8)	0.047 (2)
C26	0.7314 (8)	0.8530 (4)	-0.1035 (6)	0.052 (2)
C27	0.8842 (7)	0.7106 (4)	-0.4353 (8)	0.066 (3)
C31	0.8892 (7)	1.0702 (4)	-0.0947 (6)	0.032 (2)
C32	0.7910 (8)	1.0994 (4)	-0.1515 (7)	0.043 (2)
C33	0.8072 (7)	1.1560 (4)	-0.2146 (7)	0.044 (2)
C34	0.9240 (7)	1.1837 (4)	-0.2197 (7)	0.032 (2)
C35	1.0262 (7)	1.1560 (4)	-0.1650 (6)	0.037 (2)
C36	1.0084 (8)	1.0978 (4)	-0.1022 (7)	0.042 (2)
C37	1.0290 (8)	1.2387 (4)	-0.4209 (7)	0.056 (2)

Table 2. Selected geometric parameters (Å, °)

Sn–C11	2.113 (7)	S2–O21	1.431 (7)
Sn–C21	2.147 (7)	S2–O22	1.437 (6)
Sn–C31	2.152 (7)	S2–C27	1.760 (7)
Sn–Cl	2.378 (2)	S2–C24	1.786 (8)
Sn–O11 ¹	3.046 (5)	S3–O31	1.427 (6)

S1—O12	1.434 (5)	S3—O32	1.431 (6)
S1—O11	1.439 (5)	S3—C37	1.746 (8)
S1—C17	1.753 (10)	S3—C34	1.786 (7)
S1—C14	1.762 (8)		
C11—Sn—C21	123.0 (3)	C31—Sn—Cl	101.9 (2)
C11—Sn—C31	115.8 (3)	C11—Sn—O11 ¹	69.9 (2)
C21—Sn—C31	109.2 (3)	C21—Sn—O11 ¹	80.3 (2)
C11—Sn—Cl	102.5 (2)	C31—Sn—O11 ¹	86.2 (2)
C21—Sn—Cl	100.6 (2)	Cl—Sn—O11 ¹	170.92 (11)

Symmetry code: (i) $1 - x, 2 - y, z - \frac{1}{2}$.

Data collection: *TEXSAN* (Molecular Structure Corporation, 1985). Cell refinement: *TEXSAN*. Data reduction: *DATRD2* in *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976) in *NRCVAX*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: FG1190). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Triclinic Form of Tetraqua(orotato-*N,O*)-magnesium(II) Hydrate at 153 K

ILPO MUTIKAINEN,^a REIJO HÄMÄLÄINEN,^a MARTTI KLINGA,^a OLLI ORAMA^b AND URHO TURPEINEN^a

^aLaboratory of Inorganic Chemistry, Department of Chemistry, PO Box 55 (A. I. Virtasen aukio 1), FIN-00014 University of Helsinki, Finland, and ^bVTT Chemical Technology, Department of Chemistry, PO Box 1401 (Biologinkuja 7, Espoo), FIN-0244 VTT, Finland. E-mail: ilpo.mutikainen@helsinki.fi

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Abstract

In the title complex, [Mg(C₅H₂N₂O₄)(H₂O)₄].H₂O, orotic acid (uracil-6-carboxylic acid or 1,2,3,6-tetrahydro-2,6-dioxo-4-pyrimidinecarboxylic acid) coordinates to Mg through the deprotonated heterocyclic N1 atom and the O atom of the adjacent carboxylate group. The coordination sphere of the Mg atom is octahedral with water molecules occupying the four remaining coordination positions. The bond lengths in the coordination sphere are in the range 2.0128 (13) to 2.1295 (13) Å for the Mg—O bonds and 2.1663 (14) Å for the Mg—N bond. The C=O bond lengths for the exocyclic O2 and O4 atoms of the orotato ligand are 1.247 (2) and 1.249 (2) Å, respectively.

Comment

Orotic acid (uracil-6-carboxylic acid, vitamin B₁₃) occupies a unique position biologically by being the only effective precursor in the biosynthesis of the pyrimidine bases of nucleic acids. It has been established that during these processes the presence of metal ions, especially magnesium, is necessary, particularly during the phosphoribosylation of orotic acid (Victor, Greenberg & Sloan, 1979). Besides the biological interest, orotic acid is also interesting as a ligand because the tautomerism between the ketonic and enolic forms makes multifaceted coordination possible. Orotic acid is potentially a polydentate ligand, since, especially above