

Systematic absences suggested the space group unambiguously. The structure was solved by direct methods (*SHELXS*; Sheldrick, 1990). All atomic parameters were independently refined. The space group choice was confirmed by successful convergence of the full-matrix least-squares refinement on  $F^2$  (*SHELXL93*; Sheldrick, 1993). The highest peaks in the final difference Fourier map were in the vicinity of the nitromethane solvate molecule; the final map had no other significant features. A final analysis of variance between observed and calculated structure factors showed no dependence on amplitude or resolution. The nitromethane molecule is disordered. In addition to the crystallographic model presented here, a second structural model was refined with disordered positions for the nitromethane solvate. The following restraints were imposed on the disordered solvate parameters: (i) chemically equivalent bond lengths were restrained to remain equal, with an effective s.u. of 0.01 Å; (ii) anisotropic displacement parameters in bonding directions were restrained to be equal (s.u. 0.01 Å<sup>2</sup>); (iii) disordered positions closer than 1.2 Å were restrained to have similar displacement parameters (s.u. 0.02 Å<sup>2</sup>); (iv) disordered methyl H atoms were refined as idealized rotating groups. This model converged with  $R1 = 0.039$  [for 3294 data with  $I > 2\sigma(I)$ ] and  $wR2 = 0.099$  (for all 4146 data) against 408 parameters with 297 restraints. The five highest peaks in the final difference Fourier map were located between aromatic C atoms; the final map had no other significant features. The [Cr(N)(salen)] host molecule for this alternative refinement was identical (within 1 s.u.) to the first refinement. The N—O bond lengths for the disordered solvate model were closer to expected values; however, the excessive restraints required for convergence undermine confidence in the significance of this improvement.

Data collection: *SMART* (Siemens, 1994a). Cell refinement: *SAINT* (Siemens, 1994a). Data reduction: *SAINT*. Molecular graphics: *SHELXTL* (Siemens, 1994b). Software used to prepare material for publication: *CIFTAB* in *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1529). Services for accessing these data are described at the back of the journal.

## References

- Ashankow, S. I. & Poznjak, A. L. (1981). *Z. Anorg. Allg. Chem.* **481**, 201–206.  
 Che, C.-M., Ma, J.-X., Wong, W.-T., Lai, T.-F. & Poon, C.-K. (1988). *Inorg. Chem.* **27**, 2547–2548.  
 Chiu, H.-T., Chen, Y.-P., Chuang, S.-H., Jen, J.-S., Lee, G.-H. & Peng, S.-M. (1996). *J. Chem. Soc. Chem. Commun.* pp. 139–140.  
 Driessen, W. L. & Groeneveld, W. L. (1969). *Rec. Trav. Chim. Pays-Bas*, **88**, 491–498.  
 Du Bois, J., Hong, J., Carreira, E. M. & Day, M. W. (1996). *J. Am. Chem. Soc.* **118**, 915–916.  
 Du Bois, J., Tomooka, C. S., Hong, J. & Carreira, E. M. (1997). *Acc. Chem. Res.* **30**, 364–372.  
 Gahan, B., Garner, D. C., Hill, L. H., Mabbs, F. E., Hargrave, K. D. & McPhail, A. T. (1977). *J. Chem. Soc. Dalton Trans.* pp. 1726–1729.  
 Groves, J. T., Takahashi, T. & Butler, W. M. (1983). *Inorg. Chem.* **22**, 884–887.

- Niemann, A., Bossek, U., Haselhorst, G., Wieghardt, K. & Nuber, B. (1996). *Inorg. Chem.* **35**, 906–915.  
 Odom, A. L., Cummings, C. C. & Protasiewicz, J. D. (1995). *J. Am. Chem. Soc.* **117**, 6613–6614.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.  
 Siddal, T. L., Miyaura, N., Huffman, J. C. & Kochi, J. K. (1983). *J. Chem. Soc. Chem. Commun.* pp. 1185–1186.  
 Siemens (1994a). *SMART and SAINT. Data Collection and Processing Software*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1994b). *SHELXTL. Version 5*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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## Methyl 5-Deoxy-5-C-[(iododiphenylstannyl)methyl]-2,3-O-isopropylidene- $\alpha$ -D-lyxofuranoside†

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## Abstract

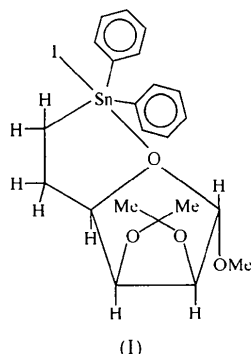
A strong intramolecular Sn $\cdots$ O interaction is present in the title molecule, [SnI(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>17</sub>O<sub>4</sub>)], resulting in a distorted trigonal-bipyramidal geometry about the Sn atom.

## Comment

A trigonal-bipyramidal arrangement about the Sn atom is present in methyl 5-deoxy-5-C-[(iododiphenylstannyl)methyl]-2,3-O-isopropylidene- $\alpha$ -D-lyxofuranoside, (I), with the non-C atoms, O1 and O3, in axial positions. The distortion from ideal geometry is shown by the angles in Table 1. In particular, the O3—Sn1—C7 angle of 69.6(2)°, which forms the bite angle of the bidentate ligand, is considerably less than 90°. The Sn1—O3 interaction of 2.720(4) Å is longer than the sum of the covalent radii (2.06 Å) but shorter than the sum of the van der Waals radii (3.78 Å) (Bondi, 1964). A five-membered chelate ring is formed by this interaction and Sn—O bond lengths for pentacoordinate C,O-

† Alternative name: (5,6-dideoxy-2,3-O-isopropylidene-1-O-methyl- $\alpha$ -D-lyxofuranosyl-C<sup>6</sup>,O)iododiphenyltin.

chelated triorganotin iodides are in the range 2.391 (5)–2.759 (5) Å (Cox *et al.*, 1996). The Sn1—I1 bond length is 2.7745 (7) Å and is comparable to the observed Sn—I range of 2.713 (2)–2.857 (1) Å found in similar compounds (Howie & Wardell, 1994), while the I1—Sn1—O3 valence angle is 165.94 (9)°.



The furanose and isopropylidene rings adopt envelope conformations, with C5 and O3, respectively, at the flaps. The ring-puckering parameters (Cremer & Pople, 1975) for the furanose ring are  $Q_2 = 0.345$  (7) Å and  $\varphi_2 = 330$  (1)°, and for the isopropylidene ring are  $Q_2 = 0.318$  (7) Å and  $\varphi_2 = 353$  (1)°.

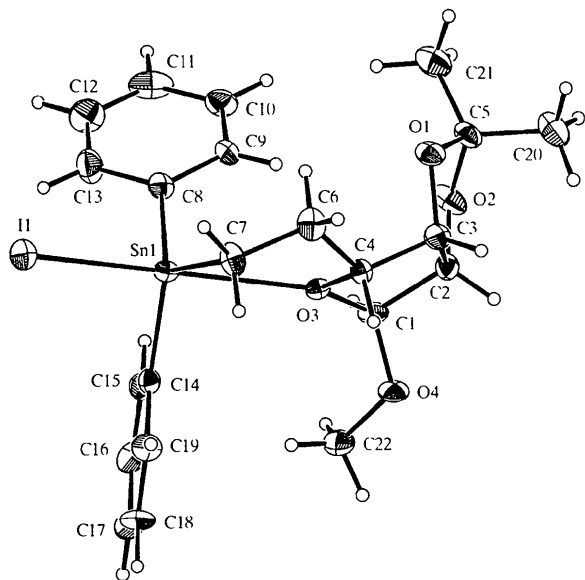


Fig. 1. ZORTEP (Zsolnai, 1997) plot showing the atomic arrangement in the title molecule. Displacement ellipsoids are shown at the 50% probability level and H atoms are represented by small spheres of arbitrary radii.

## Experimental

The title compound was prepared from methyl 5,6-dideoxy-2,3-*O*-isopropylidene- $\alpha$ -*D*-lyxo-hex-5-enofuranoside (Lerner, 1975) on successive treatment with Ph<sub>3</sub>SnH and I<sub>2</sub>. Suitable crystals of (I) (m.p. 358–359 K) were obtained by recrystallization from ethanol.

## Crystal data

[SnI(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>(C<sub>10</sub>H<sub>17</sub>O<sub>4</sub>)]

$M_r = 601.03$

Orthorhombic

$P2_12_1$

$a = 10.7138$  (11) Å

$b = 11.5397$  (14) Å

$c = 18.747$  (3) Å

$V = 2317.7$  (5) Å<sup>3</sup>

$Z = 4$

$D_x = 1.722$  Mg m<sup>-3</sup>

$D_m$  not measured

## Data collection

Delft Instruments FAST diffractometer

Area-detector scans

Absorption correction:

refined from  $\Delta F$

(DIFABS; Walker &

Stuart, 1983)

$T_{min} = 0.571$ ,  $T_{max} = 0.709$

9820 measured reflections

3589 independent reflections

## Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.038$

$wR(F^2) = 0.093$

$S = 1.120$

3589 reflections

258 parameters

H atoms: see text

$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

Mo  $K\alpha$  radiation

$\lambda = 0.71073$  Å

Cell parameters from 250

reflections

$\theta = 2.07$ – $25.02^\circ$

$\mu = 2.458$  mm<sup>-1</sup>

$T = 150$  (2) K

Prism

$0.18 \times 0.14 \times 0.14$  mm

Colourless

3426 reflections with

$I > 2\sigma(I)$

$R_{int} = 0.088$

$\theta_{max} = 25.02^\circ$

$h = -11 \rightarrow 8$

$k = -11 \rightarrow 13$

$l = -18 \rightarrow 20$

Intensity decay: none

(see text below)

$\Delta\rho_{max} = 0.91$  e Å<sup>-3</sup>

$\Delta\rho_{min} = -0.92$  e Å<sup>-3</sup>

Extinction correction: none

Scattering factors from

*International Tables for*

*Crystallography* (Vol. C)

Absolute structure: Flack

(1983)

Flack parameter =  $-0.02$  (4)

Table 1. Selected geometric parameters (Å, °)

Sn1—C14	2.133 (7)	Sn1—O3	2.720 (4)
Sn1—C8	2.134 (7)	Sn1—I1	2.7745 (7)
Sn1—C7	2.134 (7)		
C14—Sn1—C8	107.6 (3)	C8—Sn1—I1	102.04 (19)
C14—Sn1—C7	121.1 (3)	C7—Sn1—I1	96.52 (19)
C8—Sn1—C7	124.0 (3)	O3—Sn1—I1	165.94 (9)
C14—Sn1—O3	87.0 (2)	C1—O3—C4	108.6 (5)
C8—Sn1—O3	88.2 (2)	C1—O3—Sn1	144.0 (4)
C7—Sn1—O3	69.6 (2)	C4—O3—Sn1	107.3 (3)
C14—Sn1—I1	98.89 (19)		

The unit-cell and intensity data were collected on a Delft Instruments FAST diffractometer using the routines ENDEX, REFIN and MADONL in the MADNES software (Pflugrath & Messerschmidt, 1989), and processed using ABSMAD (Karaulov, 1992); detailed procedures are described by Darr *et al.* (1993). Absence of crystal decay in the X-ray beam was confirmed by checking equivalent reflections at the beginning and end of data collection, which lasted about 8 h. The H atoms were initially placed in calculated positions, and thereafter allowed to ride on their attached C atoms with common isotropic displacement parameters, which refined to 0.024 (9) (non-methyl) and 0.044 (1) Å<sup>2</sup> (methyl).

Program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994). Program(s) used to refine structure: SHELXL97

(Sheldrick, 1997). Molecular graphics: *ZORTEP* (Zsolnai, 1997).

The use of the EPSRC X-ray Crystallographic Service at the University of Wales, Cardiff, is gratefully acknowledged.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1215). Services for accessing these data are described at the back of the journal.

## References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A. & Polidori, G. (1994). *J. Appl. Cryst.* **27**, 435–436.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Cox, P. J., Garden, S. J., Howie, R. A., Melvin, O. A. & Wardell, J. L. (1996). *J. Organomet. Chem.* **516**, 213–224.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1361.
- Darr, J. A., Drake, S. R., Hursthouse, M. B. & Malik, K. M. A. (1993). *Inorg. Chem.* **32**, 5704–5708.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Howie, R. A. & Wardell, J. L. (1994). *Main Group Met. Chem.* **17**, 571–582.
- Karalov, A. I. (1992). *ABSMAD. Program for FAST Data Processing*. University of Wales, Cardiff, Wales.
- Lerner, L. M. (1975). *Carbohydr. Res.* **44**, 13–17.
- Pflugrath, J. W. & Messerschmidt, A. (1989). *MADNES*. Version of 11 September 1989. Distributed by Delft Instruments, Delft, The Netherlands.
- Sheldrick, G. M. (1997). *SHELXL97. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.
- Zsolnai, L. (1997). *ZORTEP. An Interactive ORTEP Program*. University of Heidelberg, Germany.

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## Hexakis(urea-O)magnesium Dichlorate

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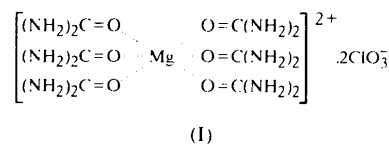
### Abstract

In the title compound,  $[\text{Mg}\{\text{OC}(\text{NH}_2)_2\}_6](\text{ClO}_3)_2$ , Mg atoms are octahedrally coordinated by the O atoms of six urea molecules. Urea1 is in-plane coordinated, while Urea2 and Urea3 are bonded with N21—C2—O2—Mg and N31—C3—O3—Mg torsion angles of 22.2 (4)

and 27.3 (4)°, respectively. The structure is built up of  $[\text{Mg}(\text{urea})_6]^{2+}$  and  $\text{ClO}_3^-$  ions held together by hydrogen bonds.

### Comment

Magnesium chlorate has been used widely as a defoliant in agrochemistry (Ware, 1983), but it is a highly hygroscopic compound. Under ambient conditions, it exists as a hexahydrate and demands special care and handling upon storage and maintenance. The partial or complete substitution of crystallization water molecules makes this salt more stable and resistant to atmospheric moisture. Moreover, the corresponding adducts possess combined activity as defoliant and fertilizing agents. The adducts known so far are  $\text{Mg}(\text{ClO}_3)_2 \cdot 4\text{H}_2\text{O} \cdot 2\text{urea}$ ,  $\text{Mg}(\text{ClO}_3)_2 \cdot 2\text{H}_2\text{O} \cdot 4\text{urea}$  and  $\text{Mg}(\text{ClO}_3)_2 \cdot 6\text{urea}$  (Musaev *et al.*, 1984; Suleimankoulov, 1971). This paper reports the crystal structure of  $\text{Mg}(\text{ClO}_3)_2 \cdot 6\text{urea}$ , (I).



In the title compound, Mg atoms lie on a centre of symmetry and are octahedrally coordinated by the O atoms of six urea molecules. The distances and angles within the urea molecules do not show any unusual values (Table 1). The coordination of the urea molecules can be suitably described with respect to C=O bond direction and N—C—O—Mg torsion angles (Macíček *et al.*, 1995). The values of 136.8 (2), 137.0 (2)

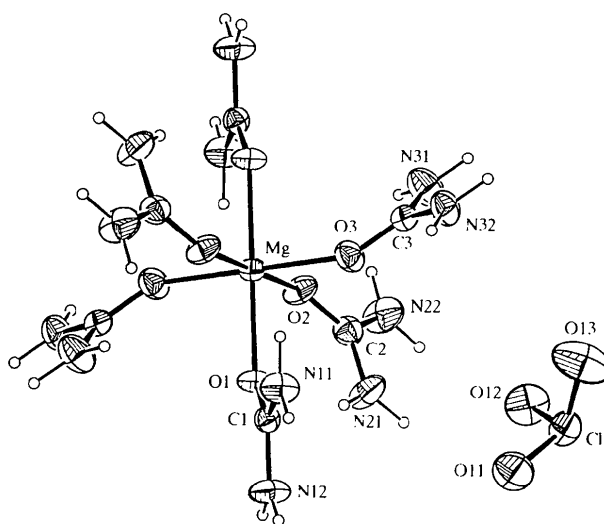


Fig. 1. ORTEP (Johnson, 1976) view of the formula unit of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are arbitrarily reduced.