

Triethylammonium (3-carboxyacrylato)-  
dichlorodimethyltin(IV)Brett D. Chandler,<sup>a</sup> Masood  
Parvez,<sup>a\*</sup> Viqar Uddin Ahmad,<sup>b</sup>  
Javid Hussain,<sup>b</sup> Hidayat Hussain<sup>b</sup>  
and Mohammad Zubair<sup>b</sup><sup>a</sup>Department of Chemistry, The University of  
Calgary, 2500 University Drive NW, Calgary,  
Alberta, Canada T2N 1N4, and <sup>b</sup>HEJ Research  
Institute of Chemistry, University of Karachi,  
Karachi 75270, Pakistan

Correspondence e-mail: parvez@ucalgary.ca

## Key indicators

Single-crystal X-ray study  
 $T = 173\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
 $R$  factor = 0.020  
 $wR$  factor = 0.048  
Data-to-parameter ratio = 15.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The crystal structure of the title compound,  $(\text{C}_6\text{H}_{16}\text{N})[\text{SnCl}_2(\text{CH}_3)(\text{C}_5\text{H}_6\text{O}_4)]$ , is composed of strongly hydrogen-bonded pairs of anions and cations. The metal centre exhibits a distorted trigonal-bipyramidal geometry, with inequivalent Sn—Cl distances of 2.5252 (4) and 2.3954 (4) Å, identical Sn—C distances within  $3\sigma$  limits [mean 2.107 (3) Å] and an Sn—O distance of 2.3077 (13) Å. The carboxyacrylate ligand forms an essentially planar seven-membered ring involving an intramolecular hydrogen bond.

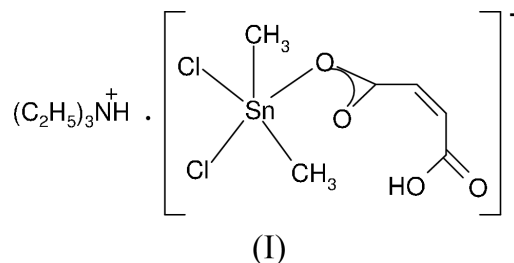
Received 24 April 2002

Accepted 3 May 2002

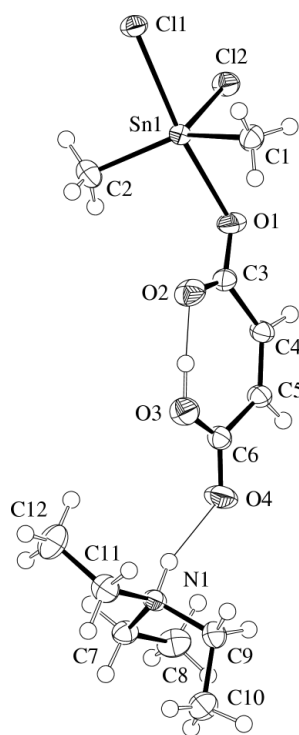
Online 11 May 2002

## Comment

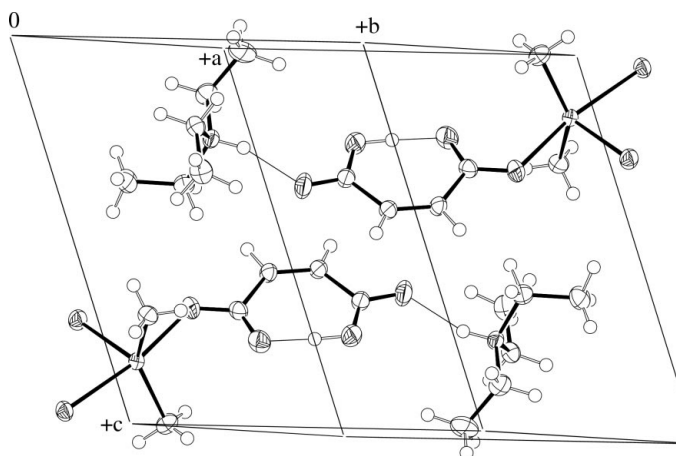
Organotin compounds have been found to be of great importance, as many have shown biocidal activities (Davies & Smith, 1982; Parulekar *et al.*, 1990; Molloy *et al.*, 1986, 1987), while others have been used as homogeneous catalysts in industry (Parulekar *et al.*, 1989; Jousseau *et al.*, 1993; Lockhart *et al.*, 1987). Organotin compounds are also used extensively as fungicides, pesticides, antifouling coatings, polymer stabilizers and preservatives of wood (Jousseau *et al.*, 1993). Accordingly, in view of our interest in the synthesis, characterization, biological applications and crystal structures of organotin carboxylates, we have prepared triethylammonium (3-carboxyacrylato)dichlorodimethyltin(IV), (I). The crystal structure of (I) is reported here.



The asymmetric unit of (I) is composed of a triethylammonium cation and a (3-carboxyacrylato)dichlorodimethyltin(IV) anion (Fig. 1). The coordination environment of the Sn atom can best be described as distorted trigonal bipyramidal, with atoms O1 and Cl1 occupying the axial and atoms C1, C2 and Cl2 the equatorial positions. The Sn—Cl1 and Sn—Cl2 distances [2.5252 (4) and 2.3954 (4) Å, respectively] are significantly different from one another, and are in agreement with the corresponding values found for similar structures contained in the Cambridge Structural Database (Allen & Kennard, 1993). The Sn—C distances (Table 1) are identical within  $3\sigma$  limits [mean 2.107 (3) Å] and the Sn—O1 distance of 2.3077 (13) Å is also unexceptional. The angle O1—Sn1—Cl1 is close to being linear [169.56 (4)°] and C2—



**Figure 1**  
ORTEP (Johnson, 1976) drawing of the cation and anion pair of (I), with displacement ellipsoids plotted at the 50% probability level.



**Figure 2**  
The unit-cell packing for (I), showing the hydrogen-bonding pattern.

Sn1—C1 is widened [ $141.93(9)^\circ$ ] from an ideal value of  $120^\circ$  at the expense of angles C2—Sn1—Cl2 [ $107.84(6)^\circ$ ] and C1—Sn1—Cl2 [ $109.06(6)^\circ$ ].

The 3-carboxyacrylate anion forms an essentially planar seven-membered ring involving an intramolecular O—H...O hydrogen bond (details are given in Table 2) that can be described in terms of graph set S(7) (Bernstein *et al.*, 1994). This kind of hydrogen-bonding pattern is typical of an unsaturated carboxylic acid. The deprotonation of the carboxyl group coordinated to the Sn atom has also led to significant electron delocalization, resulting in the C3—O1 and C3—O2 bonds [mean  $1.260(6) \text{ \AA}$ ] lying between a double [C6=O4

$1.226(2) \text{ \AA}$ ] and a single O—Csp<sup>2</sup> bond distance [C6—O3  $1.298(2) \text{ \AA}$ ].

The C—N and C—C distances in the cation are identical within  $3\sigma$  limits, with mean values of  $1.506(3)$  and  $1.510(3) \text{ \AA}$ , respectively. The anion and cation pairs exhibit strong N—H...O hydrogen bonds (details are given in Table 2), thus giving stability to the structure (Fig. 2).

## Experimental

3-Carboxyacrylic acid (1.6 g, 0.1 mol) was dissolved in 50 ml chloroform in a round-bottomed flask, and dichloromethyltin (1.098 g, 0.05 mol) and triethylamine (1.4 ml, 0.1 mol) were added. The mixture was refluxed for 3 h. On completion of the reaction, the solvent was removed by rotary evaporation and the residue was left to crystallize. The compound was recrystallized, by slow evaporation from ether at room temperature, to yield crystals suitable for X-ray diffraction studies.

### Crystal data

(C<sub>6</sub>H<sub>16</sub>N)[SnCl<sub>2</sub>(CH<sub>3</sub>)(C<sub>6</sub>H<sub>6</sub>O<sub>4</sub>)]  
*M<sub>r</sub>* = 436.92  
 Triclinic, *P* $\bar{1}$   
*a* = 8.9962 (2)  $\text{\AA}$   
*b* = 10.1856 (2)  $\text{\AA}$   
*c* = 10.8439 (3)  $\text{\AA}$   
 $\alpha$  = 69.9082 (9) $^\circ$   
 $\beta$  = 83.7866 (10) $^\circ$   
 $\gamma$  = 75.1111 (13) $^\circ$   
*V* = 901.63 (4)  $\text{\AA}^3$

*Z* = 2  
*D<sub>x</sub>* = 1.609 Mg m<sup>-3</sup>  
 Mo *K* $\alpha$  radiation  
 Cell parameters from 7194 reflections  
 $\theta$  = 1.0–27.4 $^\circ$   
 $\mu$  = 1.72 mm<sup>-1</sup>  
*T* = 173 (2) K  
 Prism, colourless  
 0.25 × 0.20 × 0.15 mm

### Data collection

Nonius KappaCCD diffractometer  
 $\omega$  and  $\varphi$  scans  
 Absorption correction: multi-scan (SORTAV; Blessing, 1997)  
*T*<sub>min</sub> = 0.673, *T*<sub>max</sub> = 0.782  
 7194 measured reflections  
 4031 independent reflections

3828 reflections with *I* > 2 $\sigma$ (*I*)  
*R*<sub>int</sub> = 0.016  
 $\theta$ <sub>max</sub> = 27.4 $^\circ$   
*h* = -11 → 11  
*k* = -13 → 13  
*l* = -14 → 14

### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.020  
*wR*(*F*<sup>2</sup>) = 0.048  
*S* = 1.08  
 4031 reflections  
 257 parameters  
 All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.015P)^2 + 0.38P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.50 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Sn1—Cl1	2.5252 (4)	O2—C3	1.254 (2)
Sn1—Cl2	2.3954 (4)	O3—C6	1.298 (2)
Sn1—O1	2.3077 (13)	O4—C6	1.226 (2)
Sn1—C1	2.109 (2)	N1—C7	1.505 (2)
Sn1—C2	2.104 (2)	N1—C9	1.510 (2)
O1—C3	1.267 (2)	N1—C11	1.504 (2)
C2—Sn1—C1	141.93 (9)	C1—Sn1—Cl1	94.78 (6)
C2—Sn1—O1	89.96 (7)	O1—Sn1—Cl1	169.56 (4)
C1—Sn1—O1	87.58 (7)	Cl2—Sn1—Cl1	89.807 (15)
C2—Sn1—Cl2	107.85 (6)	C3—O1—Sn1	120.95 (12)
C1—Sn1—Cl2	109.06 (6)	C11—N1—C7	112.35 (15)
O1—Sn1—Cl2	79.82 (4)	C11—N1—C9	111.33 (15)
C2—Sn1—Cl1	94.37 (6)	C7—N1—C9	113.72 (14)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O3–H3···O2	0.98 (3)	1.47 (3)	2.451 (2)	177 (3)
N1–H1···O4	0.89 (3)	1.89 (3)	2.748 (2)	162 (3)

The H atoms were located from difference Fourier syntheses and were included in the refinement with an overall isotropic displacement parameter.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALE-PACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976).

## References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
- Bernstein, J., Etter, M. C. & Leiserowitz, L. (1994). *Structure Correlation*, edited by H.-B. Bürgi & J. D. Dunitz, Vol. 2, pp. 431–507. New York: VCH.
- Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
- Davies, A. G. & Smith, P. J. (1982). *Comprehensive Organometallic Chemistry, The Synthesis, Reactions, and Structures of Organometallic Compounds*, Vol. 2, edited by G. Wilkinson, F. G. Stone & E. W. Able, pp. 519–627. New York: Pergamon.
- Fan, H.-F. (1991). *SAPI91*. Rigaku Corporation, Tokyo, Japan.
- Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Jousseau, B., Guillou, V., Noiret, N., Pereyre, M. & Frances, J. M. (1993). *J. Organomet. Chem.* **450**, 97–102.
- Lockhart, T. P., Calabrese, J. C. & Davidson, F. (1987). *Organometallics*, **6**, 2479–2483.
- Molloy, K. C., Purcell, T. G., Hahn, E., Schumann, H. & Zuckerman, J. J. (1986). *Organometallics*, **5**, 85–89.
- Molloy, K. C., Quill, K. & Nowell, I. W. (1987). *J. Chem. Soc. Dalton Trans.* pp. 101–106.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Parulekar, C. S., Jain, V. K., Das, T. K., Gupta, A. R., Hoskins, B. F. & Tiekink, E. R. T. (1989). *J. Organomet. Chem.* **372**, 193–199.
- Parulekar, C. S., Jain, V. K., Kesvadas, T. & Tiekink, E. R. T. (1990). *J. Organomet. Chem.* **387**, 163–173.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.