Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1073). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Chih, H. & Penfold, B. R. (1975). *J. Cryst. Mol. Struct.* **3**, 285–297.

Enraf–Nonius (1988). *CAD-4 VAX/PC Fortran System. Operator's Guide to the Enraf–Nonius CAD-4 Diffractometer Hardware, its Software and the Operating System.* Enraf–Nonius, Delft, The Netherlands.

Hall, S. R. & Stewart, J. M. (1990). Editors. *Xtal3.0 Reference Manual.* Universities of Western Australia, Australia and Maryland, USA.

Holmes, R. R., Day, R. O., Chandrasekhar, V. & Holmes, J. M. (1986). Unpublished work cited in Smith, Day, Chandrasekhar, Holmes & Holmes (1986).

Molloy, K. C., Quill, K. & Nowell, I. W. (1987). *J. Chem. Soc. Dalton Trans.* pp. 101–106.

Ng, S. W. (1992). *J. Crystallogr. Spectrosc. Res.* **22**, 615–618.

Ng, S. W. (1995). *Z. Kristallogr.* **210**, 287–289.

Ng, S. W., Chen, W. & Kumar Das, V. G. (1988). *J. Organomet. Chem.* **345**, 59–64.

Ng, S. W. & Kumar Das, V. G. (1991). *J. Organomet. Chem.* **409**, 143–156.

Ng, S. W. & Kumar Das, V. G. (1995). *Acta Cryst.* C**51**, 2489–2491.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Sandhu, G. K., Verma, S. P. & Tiekink, E. R. T. (1990). *J. Organomet. Chem.* **393**, 195–200.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Smith, P. J., Day, R. O., Chandrasekhar, V., Holmes, J. M. & Holmes, R. R. (1986). *Inorg. Chem.* **25**, 2495–2499.

Tiekink, E. R. T. (1991). *Appl. Organomet. Chem.* **5**, 1–23.

Tiekink, E. R. T., Sandhu, G. K. & Verma, S. P. (1989). *Acta Cryst.* C**45**, 1810–1811.

Zsolnai, L. & Pritzkow, H. (1994). *ZORTEP. Interactive Graphics Program.* University of Heidelberg, Germany.

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# [3-(Diethylphosphono)propionato]triphenyltin

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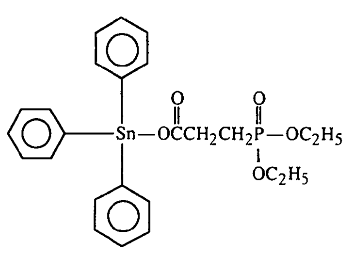
## Abstract

In the title compound, $[Sn(C_7H_{14}O_5P)(C_6H_5)_3]$, the planar triphenyltin cations are axially bridged through the carboxy and phosphoryl O atoms of the 3-(diethylphosphono)propionate anions into zigzag chains that run parallel to the *c* axis {*i.e. catena*-poly[triphenyltin-$\mu$-3-(diethylphosphono)propionato-$O$:$O^{P=O}$]}.

## Comment

Triphenyltin alkanoates generally adopt carboxylate-bridged structures in which the triphenyltin cations are linked axially by the carboxylate anions into linear chains (Ng, Chen & Kumar Das, 1988; Tiekink, 1991, 1994). A second donor O atom in the carboxylate anion allows the possibility of bridging through this site, a feature that has been documented for (diethylphosphonoacetato)triphenyltin [Sn—O = 2.129 (3), Sn←O = 2.420 (3) Å], which propagates by translations along the *b* axis (Ng & Kumar Das, 1994). The introduction of another methylene linkage in this ester results in no significant differences in the coordination geometry of the Sn atom in the title compound, (I) [Sn—O = 2.116 (2), Sn←O = 2.397 (3) Å]. However, the 3-(diethylphosphono)propionate propagates the chain by a glide along the *c* axis, so that the chain has a zigzag conformation.



(I)

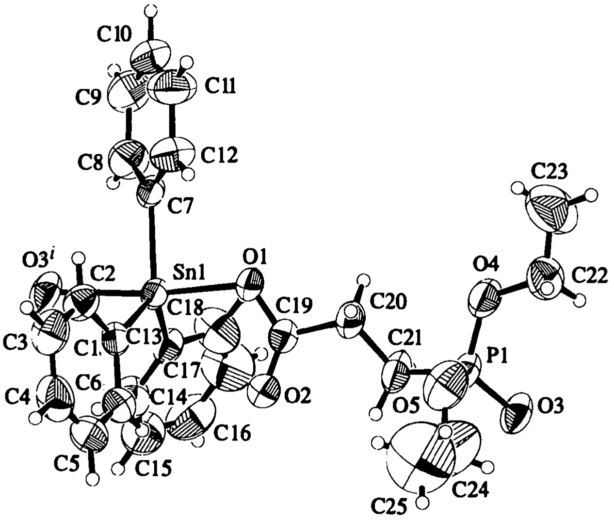


Fig. 1. Atomic labelling scheme for the title compound. *ZORTEP* (Zsolnai & Pritzkow, 1994) displacement ellipsoids are plotted at the 50% probability level.

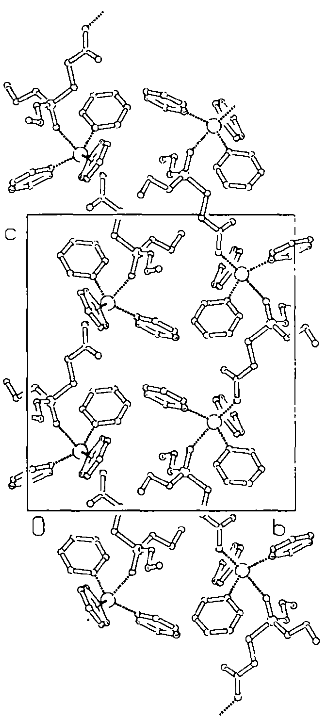


Fig. 2. *PLUTON* (Spek, 1994) plot of the packing.

## Experimental

A commercial sample of ethyl 3-(diethylphosphono)propionate was hydrolyzed to 3-(diethylphosphono)propionic acid, duplicating the literature procedure of Clayton, Luk & Rogers (1979) for the hydrolysis of 3-(diethylphosphono)acetic acid. A slight molar excess of the acid was briefly heated with triphenyltin hydroxide in ethanol; slow cooling of the filtered solution afforded crystals of the organotin ester.

*Crystal data*

$[Sn(C_7H_{14}O_5P)(C_6H_5)_3]$
$M_r = 559.14$
Monoclinic
$P2_1/n$
$a = 9.170\,(1)$ Å
$b = 16.1350\,(9)$ Å
$c = 17.697\,(2)$ Å
$\beta = 96.980\,(6)°$
$V = 2598.9\,(5)$ Å$^3$
$Z = 4$
$D_x = 1.429$ Mg m$^{-3}$
$D_m$ not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10.0–12.5°$
$\mu = 1.076$ mm$^{-1}$
$T = 300$ K
Block
$0.36 \times 0.29 \times 0.22$ mm
Colourless

*Data collection*

Enraf–Nonius CAD-4 diffractometer
$\omega$–$2\theta$ scans
Absorption correction: $\psi$ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.952$, $T_{max} = 1.000$
4850 measured reflections
4546 independent reflections

3360 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0213$
$\theta_{max} = 24.96°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 19$
$l = -21 \rightarrow 20$
3 standard reflections
frequency: 60 min
intensity decay: 2.4%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.0355$
$wR(F^2) = 0.0827$
$S = 0.971$
4546 reflections
289 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.426$ e Å$^{-3}$
$\Delta\rho_{min} = -0.270$ e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Sn1 | 0.16084 (3) | 0.19838 (2) | 0.20031 (1) | 0.0422 (1) |
| P1 | −0.1379 (1) | 0.41267 (7) | −0.13090 (6) | 0.0552 (3) |
| O1 | 0.0397 (3) | 0.2841 (2) | 0.1271 (1) | 0.0496 (7) |
| O2 | 0.1234 (4) | 0.2344 (2) | 0.0239 (2) | 0.0658 (8) |
| O3 | −0.2114 (3) | 0.3914 (2) | −0.2060 (1) | 0.0649 (8) |
| O4 | −0.2318 (4) | 0.4689 (2) | −0.0841 (2) | 0.077 (1) |
| O5 | 0.0036 (4) | 0.4637 (2) | −0.1334 (2) | 0.091 (1) |
| C1 | 0.3784 (4) | 0.2340 (2) | 0.1857 (2) | 0.0464 (9) |
| C2 | 0.4600 (5) | 0.2812 (3) | 0.2406 (2) | 0.059 (1) |
| C3 | 0.6074 (5) | 0.2992 (3) | 0.2351 (3) | 0.075 (1) |
| C4 | 0.6710 (5) | 0.2708 (4) | 0.1740 (3) | 0.081 (2) |
| C5 | 0.5914 (5) | 0.2242 (4) | 0.1193 (3) | 0.080 (2) |
| C6 | 0.4460 (5) | 0.2057 (3) | 0.1251 (2) | 0.063 (1) |
| C7 | 0.0664 (4) | 0.2537 (3) | 0.2923 (2) | 0.047 (1) |
| C8 | −0.0011 (6) | 0.2072 (3) | 0.3434 (3) | 0.075 (1) |
| C9 | −0.0675 (7) | 0.2453 (5) | 0.4014 (3) | 0.098 (2) |
| C10 | −0.0649 (7) | 0.3283 (5) | 0.4086 (3) | 0.094 (2) |
| C11 | 0.0004 (7) | 0.3740 (4) | 0.3601 (3) | 0.096 (2) |
| C12 | 0.0663 (6) | 0.3381 (3) | 0.3015 (3) | 0.073 (1) |
| C13 | 0.0673 (5) | 0.0870 (2) | 0.1526 (2) | 0.0475 (9) |
| C14 | 0.1499 (6) | 0.0193 (3) | 0.1404 (3) | 0.072 (1) |
| C15 | 0.0879 (8) | −0.0513 (3) | 0.1057 (3) | 0.088 (2) |
| C16 | −0.0568 (8) | −0.0558 (4) | 0.0845 (3) | 0.093 (2) |
| C17 | −0.1422 (7) | 0.0108 (4) | 0.0959 (4) | 0.110 (2) |
| C18 | −0.0791 (6) | 0.0825 (3) | 0.1294 (3) | 0.088 (2) |
| C19 | 0.0464 (4) | 0.2814 (2) | 0.0541 (2) | 0.047 (1) |
| C20 | −0.0480 (6) | 0.3472 (3) | 0.0111 (2) | 0.066 (1) |
| C21 | −0.0937 (6) | 0.3261 (3) | −0.0700 (2) | 0.067 (1) |
| C22 | −0.3099 (7) | 0.5398 (3) | −0.1188 (3) | 0.084 (2) |
| C23 | −0.3957 (9) | 0.5754 (4) | −0.0640 (4) | 0.137 (3) |
| C24 | 0.101 (1) | 0.4504 (7) | −0.1906 (5) | 0.176 (4) |
| C25 | 0.225 (1) | 0.4932 (7) | −0.1828 (6) | 0.196 (4) |

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

| | | | |
|---|---|---|---|
| Sn1—O1 | 2.116 (2) | Sn1—C7 | 2.131 (4) |
| Sn1—O3$^i$ | 2.397 (3) | Sn1—C13 | 2.123 (4) |
| Sn1—C1 | 2.122 (4) | | |
| O1—Sn1—C1 | 100.4 (1) | O3$^i$—Sn1—C13 | 84.5 (1) |
| O1—Sn1—C7 | 87.9 (1) | C1—Sn1—C13 | 121.4 (2) |
| O1—Sn1—O3$^i$ | 174.1 (1) | C1—Sn1—C7 | 117.1 (2) |
| O3$^i$—Sn1—C1 | 82.0 (1) | C13—Sn1—C7 | 118.5 (2) |
| O3$^i$—Sn1—C7 | 86.2 (1) | | |

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

Each H atom was refined with an isotropic displacement parameter equal to 1.5 times the $U_{eq}$ of the parent C atom.

Data collection: *CAD*-4 *VAX/PC* (Enraf–Nonius, 1988). Cell refinement: *CAD*-4 *VAX/PC*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PLUTON* (Spek, 1994), *ZORTEP* (Zsolnai & Pritzkow, 1994). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1075). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

---

### References

Clayton, J. P., Luk, K. & Rogers, N. H. (1979). *J. Chem. Soc. Perkin Trans.* 1, pp. 308-313.

Enraf-Nonius (1988). *CAD-4 VAX/PC Fortran System. Operator's Guide to the Enraf-Nonius CAD-4 Diffractometer Hardware, its Software and the Operating System.* Enraf-Nonius, Delft, The Netherlands.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Ng, S. W., Chen, W. & Kumar Das, V. G. (1988). *J. Organomet. Chem.* **364**, 59–64.

Ng, S. W. & Kumar Das, V. G. (1994). *J. Chem. Crystallogr.* **24**, 337–340.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351-359.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for Refinement of Crystal Structures.* University of Göttingen, Germany.

Spek, A. L. (1994). *PLUTON. Molecular Graphics Program.* University of Utrecht, The Netherlands.

Tiekink, E. R. T. (1991). *Appl. Organomet. Chem.* **5**, 1-23.

Tiekink, E. R. T. (1994). *Trends Organomet. Chem.* **1**, 71-116.

Zsolnai, L. & Pritzkow, H. (1994). *ZORTEP. Interactive Graphics Program.* University of Heidelberg, Germany.

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# *mer*-[2′-(2-Benzoyl-1-methylethylidene)-1-naphthohydrazido(2–)](methanol)-*cis*-dioxomolybdenum(VI)

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## Abstract

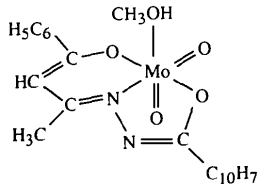
In the molecule of the title compound, $[MoO_2(L)(CH_3OH)]$ [$L$ is the dianion of 2′-(2-benzoyl-1-methylethylidene)-1-naphthohydrazide, $C_{21}H_{16}N_2O_2$], the Mo atom displays distorted octahedral coordination geometry, being coordinated by two mutually *cis* oxo ligands, the methanol O atom and the tridentate ligand $L$ bonded through the azomethine N atom, the enolic O atom and the oxazine O atom (equatorially disposed). The packing in the crystal structure involves a hydrogen bond from the methanol OH as donor to an oxo ligand as acceptor.

## Comment

Owing to the diverse properties of carboxylic acid hydrazides and hydrazones and their wide application in many fields (*e.g.* medicine and analytical chemistry), particularly for the preparation of new heterogeneous catalysts for oxido-reduction processes (Troepol'skaya & Munin, 1977; Kogan, Zelentsov, Larin & Lukov, 1990), increased attention has been devoted to the study of their complexing affinities towards different transition metals. The complexation of such a ligand with molybdenum as a bioelement is of great interest, since molybdenum is a component of several redox enzymes and is essential for the biological binding of molecular nitrogen (Cotton & Wilkinson, 1988; Kamenar, 1981).

Special significance is attached to $Mo^{VI}O_2$ complexes because the active site of the molybdenum coordination sphere is considered to be that *trans* to the oxo O atom. The *trans* ligand is replaced by the substrate molecule, thus making possible the basic principal function of the enzyme.

The title compound, $[MoO_2(L)(CH_3OH)]$ [$L$ is the dianion of 2′-(2-benzoyl-1-methylethylidene)-1-naphthohydrazide], (I), is one of several complexes that we have synthesized (Leovac, Ivanović, Andjelković & Mitrovski, 1995). In order to confirm our proposal of the structure, and to determine the molecular dimensions in detail, we have undertaken the crystal structure analysis of (I). The structure consists of monomeric molecules; a perspective view showing the atomic numbering scheme is given in Fig. 1.



(I)

The Mo atom is coordinated by a pair of mutually *cis* oxo ligands and the tridentate equatorially disposed $L$ ligand bonded *via* the azomethine N atom, the enolic O atom (O2) and the $\alpha$-oxyazine atom (O1). Distorted octahedral coordination is completed by the O atom of the methanol. The enolic O atom is *trans* to the $\alpha$-oxyazine O atom, and the O4 oxo ligand is *trans* to the azomethine N atom. The interatomic distances Mo—O4 and Mo—O5 of 1.714 (2) and 1.689 (3) Å,

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