

This research was supported by grants from the National Natural Science Foundation of China and the Natural Science Foundation of Fujian Province.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1312). Services for accessing these data are described at the back of the journal.

## References

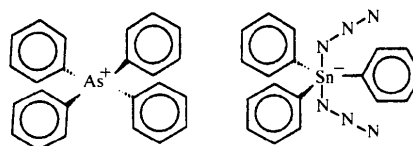
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around the metal ion, with the azide moieties in axial positions having equal bond distances [Sn—N 2.278 (5) and 2.281 (6) Å] but different bond angles [Sn—N—N 117.4 (4) and 125.2 (5)°] to the tin. The cation has its usual tetrahedral geometry.

## Comment

Previously, we synthesized and characterized by Mössbauer and vibrational spectroscopy tetraphenylarsonium and tetramethylammonium salts of the complex anions [Ph<sub>3</sub>Sn(N<sub>3</sub>)<sub>2</sub>]<sup>−</sup>, [Ph<sub>3</sub>Sn(N<sub>3</sub>)(NCS)]<sup>−</sup>, [Me<sub>2</sub>Sn(N<sub>3</sub>)<sub>4</sub>]<sup>2−</sup> and [Ph<sub>2</sub>Sn(N<sub>3</sub>)<sub>2</sub>(NCS)<sub>2</sub>]<sup>2−</sup> (Barbieri *et al.*, 1975). Trigonal-bipyramidal structures have been suggested for Ph<sub>3</sub>Sn<sup>IV</sup> with apical pseudohalide ligands, while for the R<sub>2</sub>Sn<sup>IV</sup> derivatives, *trans* octahedral geometries have been assumed (R = Me, Ph).

The X-ray structure determination of [(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>P]<sub>2</sub>[Sn(N<sub>3</sub>)<sub>4</sub>Me<sub>2</sub>] (Halfpenny, 1995) confirmed the Mössbauer results for the octahedral geometry, while the trigonal-bipyramidal structure with the azide groups in axial positions has been found in the X-ray structures of piperidinium and quinuclidinium [Ph<sub>3</sub>Sn(N<sub>3</sub>)<sub>2</sub>] (Wharf *et al.*, 1997). In the latter, the azide groups are not equivalent, with different Sn—N bond distances due to hydrogen-bond interactions with the cations. These results indicated that a compound without protonated cations should provide interesting information. Accordingly, we now describe the X-ray structure of the title compound, (I).



(I)

*Acta Cryst.* (1999). **C55**, 1664–1665

## Tetraphenylarsonium diazidotriphenylstannate

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(Received 9 June 1999; accepted 30 June 1999)

## Abstract

The triphenyltin–azide anion of the title tetraphenylarsonium salt, [As(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>][Sn(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>(N<sub>3</sub>)<sub>2</sub>], shows approximately trigonal-bipyramidal coordination geometry

The anion has the expected trigonal-bipyramidal coordination geometry around the Sn atom, with the azide ligands in axial positions and equivalent in their Sn—N and N—N bond lengths (Fig. 1 and Table 1). In fact, the Sn—N bond distances are equal and their values are intermediate between those found for asymmetric bonding in the protonated derivatives, which vary from 2.216 (3) to 2.396 (2) Å in the piperidinium salt and from 2.226 (4) to 2.371 (4) Å in the quinuclidinium salt (Wharf *et al.*, 1997). The azide ligands are coordinated with the difference in Sn—N—N angles probably resulting from packing requirements rather than from different resonance forms of the azide ligands because N<sub>α</sub>—N<sub>β</sub> and N<sub>β</sub>—N<sub>γ</sub> are the same in the two azide ligands. The present structure has no significant non-bonded contacts, the shortest is an intramolecular contact of 2.45 Å between N1 and H—C14 of the coordinated phenyl ligand, while the shortest interionic contact is N6<sup>⋯</sup>H40—C40

of 2.59 Å [symmetry code: (i)  $-x, 1-y, -z$ ]. The dihedral angles between the SnC<sub>3</sub> moiety and the phenyl rings of the anion are different [SnC<sub>3</sub>/C1–C6 61.1 (2), SnC<sub>3</sub>/C7–C12 55.2 (2) and SnC<sub>3</sub>/C13–C18 71.2 (2)°] with respect to the geometries reported with the protonated cations [range 54.56 (17)–81.56 (13)°], showing once again the importance of the cation–anion interactions in the [Ph<sub>2</sub>Sn(N<sub>3</sub>)] anion geometry. The reciprocal orientations of the two azide ligands can be defined by the dihedral angle between the Sn/N1/N2/N3 and Sn/N4/N5/N6 planes of 85.2 (6)°, with an approximately linear N<sub>α</sub>—Sn—N<sub>α</sub> arrangement.

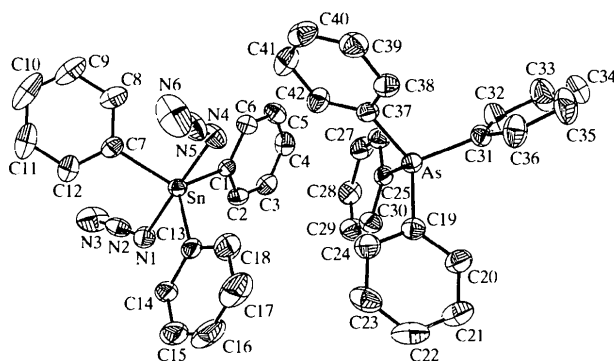


Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 40% probability level.

## Experimental

The title compound was prepared as described in the literature (Barbieri *et al.*, 1975) and crystallized from ethanol/ether.

### Crystal data

[As(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>][Sn-  
(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>(N<sub>3</sub>)<sub>2</sub>]  
*M<sub>r</sub>* = 817.37  
Monoclinic  
*P*2<sub>1</sub>/*n*  
*a* = 14.577 (3) Å  
*b* = 10.875 (2) Å  
*c* = 23.985 (5) Å  
*β* = 90.40 (5)°  
*V* = 3802.1 (13) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.428 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

### Data collection

Philips PW1100 four-circle diffractometer  
Profile-fitted *θ*/*2θ* scans (Lehmann & Larsen, 1974)

Mo *Kα* radiation  
*λ* = 0.71073 Å  
Cell parameters from 30 reflections  
*θ* = 9–12°  
*μ* = 1.573 mm<sup>-1</sup>  
*T* = 293 (2) K  
Prism  
0.40 × 0.28 × 0.26 mm  
Colourless

5761 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.052  
*θ<sub>max</sub>* = 25.01°  
*h* = -17 → 17

Absorption correction:  
*ψ* scan (North *et al.*, 1968)  
*T<sub>min</sub>* = 0.605, *T<sub>max</sub>* = 0.664  
6810 measured reflections  
6645 independent reflections

### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.050  
*wR*(*F*<sup>2</sup>) = 0.128  
*S* = 1.290  
6645 reflections  
451 parameters  
H atoms constrained  
*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0272*P*)<sup>2</sup> + 10.9518*P*]  
where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3

*k* = 0 → 12  
*l* = 0 → 28  
3 standard reflections  
frequency: 200 min  
intensity decay: none

(Δ/σ)<sub>max</sub> = 0.004  
Δρ<sub>max</sub> = 0.701 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.637 e Å<sup>-3</sup>  
Extinction correction: none  
Scattering factors from  
*International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Sn—N1	2.281 (6)	As—C31	1.904 (5)
Sn—N4	2.278 (5)	As—C37	1.915 (5)
Sn—C1	2.142 (5)	N1—N2	1.176 (8)
Sn—C7	2.149 (6)	N2—N3	1.146 (9)
Sn—C13	2.151 (6)	N4—N5	1.172 (8)
As—C19	1.906 (6)	N5—N6	1.131 (9)
As—C25	1.904 (5)		
N1—Sn—N4	178.6 (2)	C7—Sn—C13	121.7 (2)
C1—Sn—N1	90.8 (2)	C13—Sn—N4	90.9 (2)
C7—Sn—N1	91.2 (2)	C13—Sn—N1	87.8 (2)
C1—Sn—N4	89.4 (2)	N2—N1—Sn	125.2 (5)
C7—Sn—N4	90.0 (2)	N5—N4—Sn	117.4 (4)
C1—Sn—C7	117.4 (2)	N1—N2—N3	176.4 (7)
C1—Sn—C13	120.9 (2)	N4—N5—N6	177.7 (8)

Data collection: *FEBO* (Belletti, 1993). Cell refinement: *FEBO*. Data reduction: *FEBO*. Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *PARST94* (Nardelli, 1995).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1430). Services for accessing these data are described at the back of the journal.

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