APPLIED ORGANOMETALLIC CHEMISTRY
Appl. Organometal. Chem. 2004; **18**: 421–422

Crystallographic report

Diisopropylammonium [3-(2-thienyl)-2-sulfanylpropenoato]triphenylstannate

José S. Casas[1], Marıa D. Couce[2], Agustín Sánchez[1], José Sordo[1]*, José M. Varela[1] and Ezequiel M. Vázquez-López[2]

[1]Department of Inorganic Chemistry, University of Santiago de Compostela, 15782 Santiago de Compostela, Galicia, Spain
[2]Department of Inorganic Chemistry, University of Vigo, 36200 Vigo, Galicia, Spain

Received 7 April 2004; Revised 15 April 2004; Accepted 16 April 2004

The title compound comprises trigonal bipyramidal $SnPh_3$(tspa) anions and $^{i}Pr_2NH_2$ cations linked into centrosymmetric dimers by N–H···O hydrogen bonds. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; triphenyltin(IV); X-ray diffraction; organotin(IV) compounds

COMMENT

The structure comprises $SnPh_3$(tspa) anions and $^{i}Pr_2NH_2$ cations that form centrosymmetric dimers via hydrogen bonds; Fig. 1. In the anion the tin atom is coordinated to three phenyl carbon atoms and to the sulfur and one oxygen atom of a tspa dianion, defining a distorted trigonal bipyramidal *cis*-C_3SnOS kernel in which the O1 and C21 atoms occupy the apical positions. The apical Sn–C bond is longer than the equatorial Sn–C bonds, but all three are within the range 2.12–2.18 Å reported for other triphenyltin compounds.[1,2] The Sn–S and Sn–O bonds are longer than those previously found in other triphenyltinsulfanylpropenoates.[2]

EXPERIMENTAL

[$^{i}Pr_2NH_2$][$SnPh_3$(tspa)] was obtained by reaction of $SnPh_3OH$ and H_2tspa following a published method.[2] The solution of the crude product in dimethylsulfoxide that had been used for NMR spectroscopy afforded crystals suitable for X-ray diffractometry. Anal. Found: C, 58.3; H, 5.2; N, 2.2; S, 9.6. Calc. for $C_{31}H_{35}NO_2S_2Sn$: C, 58.5; H, 5.3; N, 2.2; S, 10.0%. Intensity data were collected at 293(2) K from a crystal of dimensions $0.10 \times 0.20 \times 0.35$ mm^3 in a Bruker Smart CCD 1000 K diffractometer. The thienyl group is disordered, S2 being found both cis and trans to S1 because of rotation about C3–C4; refinement of the alternative positions of S2 and C7 led to occupancy factors of 50%, which were fixed in the last cycle of the refinement. Crystallographic data: $C_{31}H_{35}NO_2S_2Sn$, $M = 636.41$, triclinic, $P\overline{1}$, $a = 10.3191(1)$, $b = 11.5299(1)$, $c = 13.2716(2)$ Å, $\alpha = 82.5370(11)$, $\beta = 88.7597(4)$, $\gamma = 83.10(5)^\circ$, $V = 1554.30(3)$ Å^3, $Z = 2$, 7352 unique reflections and 5389 with $I \geq 2\sigma(I)$, $R = 0.047$ (obs. data), $wR = 0.100$ (all data). Programs used: SHELXS-97, SHELXL-97, ORTEP. CCDC number: 235277.

Acknowledgements

We thank the Directorates General for Regional Policies (EU) and for Research (Spanish Ministry of Science and Technology) for financial support under ERDF programs (projects BQU2002-04524-CO2-01 and BQU2002-04524-CO2-02).

REFERENCES

1. James BD, Magee RJ, Patalinghug WC, Skelton BW, White AH. *J. Organometal. Chem.* 1994; **464**: 51.
2. Casas JS, Castiñeiras A, Couce MD, Jorge ML, Russo U, Sánchez A, Seoane R, Sordo J, Varela JM. *Appl. Organometal. Chem.* 2000; **14**: 421.

---

*Correspondence to: José Sordo, Department of Inorganic Chemistry, Faculty of Pharmacy, University of Santiago de Compostela, 15782 Santiago de Compostela, Spain.
E-mail: qijsordo@usc.es
Contract/grant sponsor: Directorates General for Regional Policies (EU) and for Research (Spanish Ministry of Science and Technology); Contract/grant numbers: BQU2002-04524-CO2-01; BQU2002-04524-CO2-02.

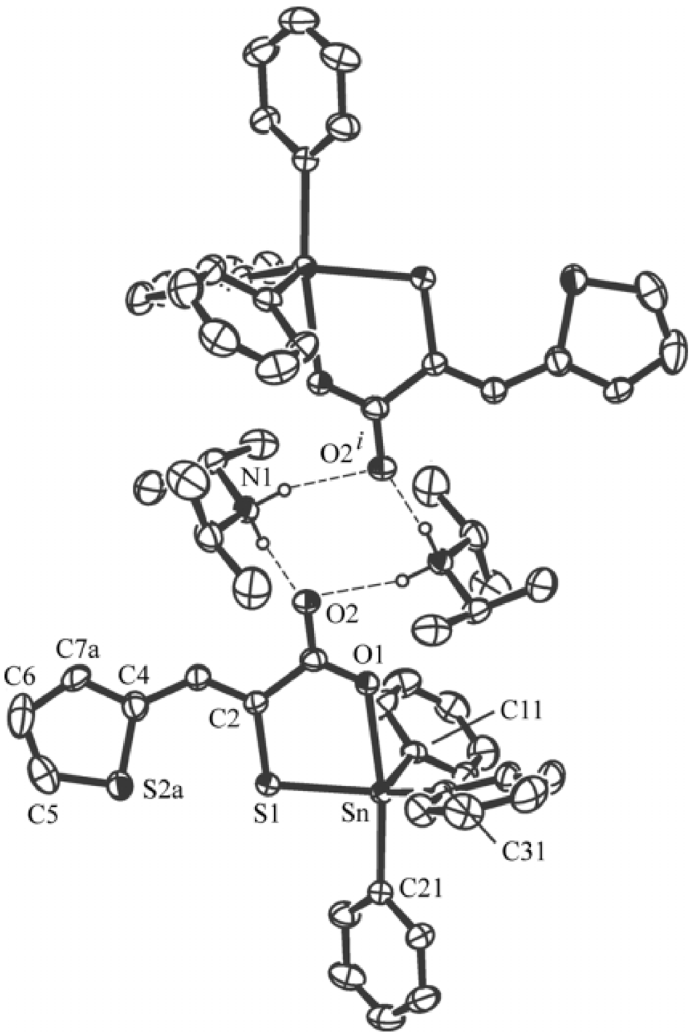

Figure 1. ORTEP plot showing the molecular structure of the hydrogen-bonded dimer of $[{}^{i}Pr_2NH_2][SnPh_3(tspa)]$; most hydrogen atoms have been omitted for clarity. Selected bond lengths (Å) and angles (°): Sn–S1 2.4532(10), Sn–O1 2.390(2), Sn–C11 2.158(3), Sn–C21 2.194(4), Sn–C31 2.149(4); S1–Sn–O1 76.36(7), S1–Sn–C11 115.82(11), S1–Sn–C21 95.23(10), S1–Sn–C31 116.95(10), O1–Sn–C11 82.36(11), O1–Sn–C21 171.59(11), O1–Sn–C31 83.22(11), C11–Sn–C21 101.43(13), C21–Sn–C31 101.11(14), C11–Sn–C31 119.52(14). N1–H1···O2: 0.93(4), 1.87(4), 2.794(4) Å, 171(4)°; N1–H10···O2^i: 0.94(4), 2.08(4), 2.974(4) Å, 160(3)°. Symmetry operation *i*: $-x$, $-y$, $2-z$.