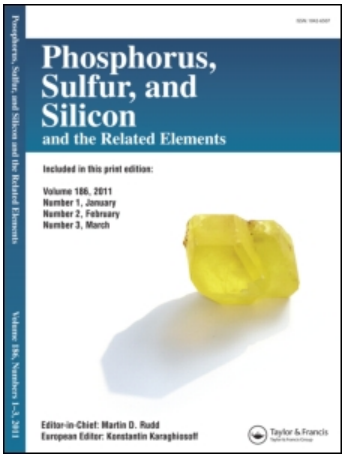
This article was downloaded by:
On: *29 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Phosphorus, Sulfur, and Silicon and the Related Elements
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

SYNTHESIS OF TWO TETRAPHENYLANTIMONY COMPLEXES OF PYRIDINE-*N*-OXIDES; CRYSTAL STRUCTURE OF TETRAPHENYLANTIMONY (2-MERCAPTOPYRIDINE-*N*-OXIDE)

Georgia M. Arvanitis[a]; Michael E. Berardini[a]; Thomas B. Acton[a]; Philip E. Dumas[a]
[a] Department of Chemistry, Trenton State College, Hillwood Lakes, NJ

To cite this Article Arvanitis, Georgia M. , Berardini, Michael E. , Acton, Thomas B. and Dumas, Philip E.(1993) 'SYNTHESIS OF TWO TETRAPHENYLANTIMONY COMPLEXES OF PYRIDINE-*N*-OXIDES; CRYSTAL STRUCTURE OF TETRAPHENYLANTIMONY (2-MERCAPTOPYRIDINE-*N*-OXIDE)', Phosphorus, Sulfur, and Silicon and the Related Elements, 82: 1, 127 — 135
To link to this Article: DOI: 10.1080/10426509308047416
URL: http://dx.doi.org/10.1080/10426509308047416

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

Phosphorus, Sulfur, and Silicon, 1993, Vol. 82, pp. 127–135
Reprints available directly from the publisher
Photocopying permitted by license only

© 1993 Gordon and Breach Science Publishers S.A.
Printed in the United States of America

SYNTHESIS OF TWO TETRAPHENYLANTIMONY COMPLEXES OF PYRIDINE-*N*-OXIDES; CRYSTAL STRUCTURE OF TETRAPHENYLANTIMONY (2-MERCAPTOPYRIDINE-*N*-OXIDE)

GEORGIA M. ARVANITIS,† MICHAEL E. BERARDINI, THOMAS B. ACTON and PHILIP E. DUMAS

Department of Chemistry, Trenton State College, Hillwood Lakes, NJ 08650

(Received February 23, 1993; in final form May 23, 1993)

Tetraphenylantimony(2-mercaptopyridine-*N*-oxide) and tetraphenylantimony(2-selenopyridine-*N*-oxide) were synthesized from tetraphenylantimonybromide and the appropriate pyridine-*N*-oxide derivative. X-ray crystal structure analysis shows that the former compound crystallizes in space group C2/c, with *a* 29.095(2), *b* 10.6965(8), *c* 18.134(1), *β* 94.154(5) and *Z* 8. The mercaptopyridine-*N*-oxide ligand binds via both its sulfur and oxygen atoms, leading to a distorted octahedral geometry about the antimony atom. Tetraphenylantimonypyridine-*N*-oxide derivatives of sulfur and selenium were found to possess antimalarial activity.

Key words: Antimony complexes; x-ray; selenium; sulfur; antimalarial activity; biological activity; IR.

Downloaded At: 12:52 29 January 2011

INTRODUCTION

2-Mercaptopyridine-*N*-oxide has been used as a ligand for a variety of metals and the resulting complexes display diverse structural features.[1,2] In addition such compounds have often been found to possess significant biological activity.[3,4] We were interested in using this potentially bidentate moiety as a ligand for metal complexes which themselves exhibit significant therapeutic properties. In the present investigation we have selected tetraphenylantimony(V) as a suitable species. The synthesis and characterization of tetraphenylantimony-2-mercaptopyridine-*N*-oxide, **1**, and tetraphenylantimony-2-selenopyridine-*N*-oxide, **2**, are presented along with the crystal structure of the former compound. A noteworthy feature of the structure is the bidentate nature of the mercaptopyridine-*N*-oxide ligand.

RESULTS AND DISCUSSION

The reaction of a solution of tetraphenylantimony bromide with the pyridine-*N*-oxide derivative in a 1:1 molar ratio in the presence of triethylamine leads to a high yield of the corresponding tetraphenylantimony derivative. Both compounds are very pale yellow solids with sharp melting points and are soluble in common organic solvents.

The infrared spectra of complexes **1** and **2** are detailed in the experimental section, however, several of their important features are discussed here. Mercaptopyridine-*N*-oxide itself shows an extremely broad band in the 2700–2600 cm^{-1} region due to S—H stretching. The absence of this band suggests the formation of

an antimony sulfur bond. A strong band at 1263 cm^{-1} characterizes the NO group which shifts to lower frequency (1197 cm^{-1}) upon coordination to the metal center. Such behavior has been noted for other pyridine-*N*-oxide-metal complexes.[5] Similarly, the selenium analogue does not show a band at 2440 cm^{-1} for an Se—H bond and its N—O stretch has shifted to 1196 cm^{-1}. Therefore, in both derivatives, the solid state structure contains a bidentate pyridine-*N*-oxide moiety.

While the ^{13}C NMR spectrum of **1** contains signals for only one type of phenyl group, the ^{1}H NMR spectrum shows evidence of the stereochemical nonrigidity of the complex in solution. The ortho and para resonances of the phenyl groups of **1** appear as a multiplet of sharp peaks at 298 K; these signals begin to broaden at 276 K and continue to do so down to 223 K. No limiting spectrum was obtained upon cooling. These phenyl signals coalesce when the sample is brought back to room temperature. Heating to 323 K produces no changes in the spectrum. The remaining protons are uneffected by the temperature change. The NMR suggests equilibration of the phenyl ligands may result from either pseudorotation of a pentacoordinate species in solution or a trigonal twist of an octahedron. Both types of fluxional processes have been observed for Sb(V) complexes.[6,7]

Analysis of the crystal structure of **1** shows that in the solid state the mercaptopyridine-*N*-oxide ligand is bidentate, coordinating to the antimony by both its sulfur and oxygen atoms. The molecular structure of **1** and the atomic numbering scheme are shown in Figure 1. Important bond distances and angles are given in Table I.

The coordination sphere of the antimony can be described as distorted octahedral. The Sb—O bond of 2.231 Å is only slightly longer than what has been reported for such compounds as $Ph_3Sb(O_2CCH_3)_2$ [8] and falls within the extremes of 1.935

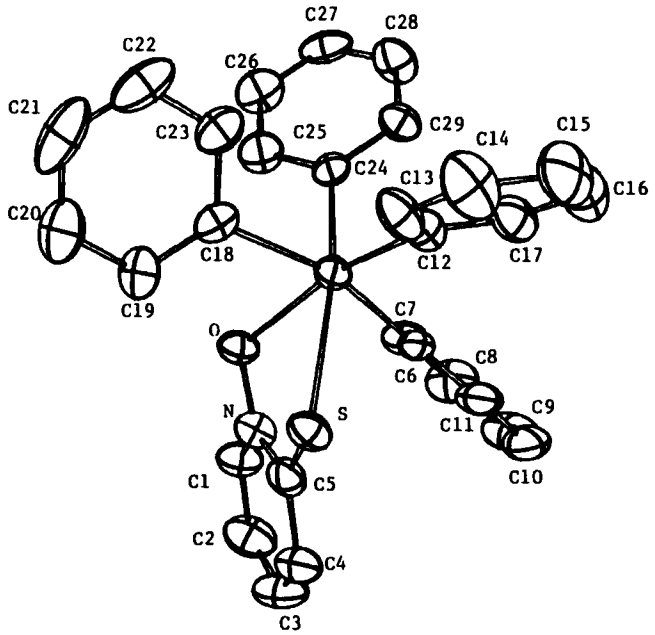


FIGURE 1 Structure and labeling scheme for complex **1** (30% probability ellipsoids).

Downloaded At: 12:52 29 January 2011

TABLE I

Selected interatomic distances (Å) and angles (°) for 1 with e.s.d.'s in parentheses.

Sb	- S	2.716(3)	
Sb	- O	2.231(8)	
Sb	- C6	2.170(3)	
Sb	- C12	2.143(3)	
Sb	- C18	2.149(3)	
Sb	- C24	2.153(3)	
S	- C5	1.696(4)	
O	- N	1.35(1)	
N	- C1	1.33(2)	
N	- C5	1.37(1)	
C1	- C2	1.34(2)	
C2	- C3	1.38(2)	
C3	- C4	1.33(2)	
C4	- C5	1.40(1)	
all phenyl ring distances 1.395			
C12-	Sb - C24	103.7(1)	
C12-	Sb - C18	95.9(1)	
C6	- Sb - C18	163.5(1)	
C6	- Sb - C12	98.0(1)	
O	- Sb - C18	81.1(2)	
O	- Sb - C6	82.9(2)	
S	- Sb - C24	166.7(1)	
S	- Sb - C12	89.5(1)	
S	- Sb - O	71.7(2)	
Sb	- S - C5	92.4(2)	
Sb	- O - N	115.0(6)	
O	- N - C5	120.2(8)	
N	- C5 - C4	113.4(7)	
S	- C5 - C4	126.1(6)	
S	- C5 - N	120.4(5)	
Sb	- C6 - C11	120.9(2)	
Sb	- C12- C17	119.9(2)	
Sb	- C18- C23	114.3(2)	
Sb	- C24- C29	121.4(2)	
C1	- N - C5	125 (1)	
N	- C1 - C2	121 (1)	
C2	- C3 - C4	121 (1)	
C3	- C4 - C5	122 (1)	

Downloaded At: 12:52 29 January 2011

Å to 2.506 Å for antimony(V) oxygen bonds.[9,10] The S—Sb—O angle measuring 71.7° and the angle which is opposite it, C(24)—Sb—C(12), deviate by far the most from 90°. This is not surprising, since the S—Sb—O unit is part of a five-membered ring. In fact, the value is between that of the four-membered chelate of phenylbis[2-pyridinethiolato(1-)]antimony(III)[11] and that of the six-membered system in (acetylacetonato)dichlorodiphenylantimony(V).[12] Furthermore, the Sb—C bond lengths are in the range of 2.14–2.18 Å, as one would expect for an octahedral geometry,[13] rather than the shorter Sb—C bonds of equatorial phenyl groups in a trigonal bipyramid.[14]

The N—O bond is essentially the same length as that of 2-[[(hydroxymethoxy)methyl]thio]pyridine-*N*-oxide,[15] about 1.35 Å. The remaining interatomic distances for the pyridine moiety are also as expected. This ligand is planar, with a maximum deviation of 0.001 Å from the plane; the antimony atom is 1.25 Å from the mean plane defined by S, C5, N and O.

Intermolecular distances are greater than 3.3 Å, not considering those involving hydrogen atoms.

Attempts to crystallize **2** led to its decomposition, with bis-2-selenopyridine-*N*-oxide as the only selenium containing product observed.[16] Similar reactions are common for tetraorganoantimony(V) mercaptides[17]; we consider these results of interest and have begun to investigate further.

The antiparasitic nature of antimony compounds is well established[18,19] and they have been used in the past against numerous protozoan parasites, including those responsible for trypanosomiasis and schistosomiasis. Pentavalent antimonials remain the primary chemotherapeutic agents for the treatment of human leishmaniasis today.[20,21] Compounds **1** and **2** were tested for antimalarial activity after the method of Desjardins[22] and were found to inhibit the growth of both the W-2 and D-6 clones of *Plasmodium falciparum*. The results, along with standards used for comparison are reported in Table II. Both compounds are about as active as chloroquine, the most widely used antimalarial drug[23] and are two to three times as active as antimony compounds previously studied in our laboratory.[24] The significant activity of these complexes and others have encouraged us to continue our efforts in developing compounds of this type and in understanding the nature of their antiparasitic activity. We are currently screening these and other antimony compounds against a variety of related parasites.

TABLE II

IC_{50} (ng/mL) against clones of *Plasmodium falciparum*

compound	W-2	D-6
1	13.0	8.39
2	13.8	9.05
chloroquine	18.22	3.24
mefloquine	0.024	2.31
tetracycline	264.3	185.39

Downloaded At: 12:52 29 January 2011

EXPERIMENTAL

Synthesis

Tetraphenylantimony bromide (Alfa) and 2-mercaptopyridine-*N*-oxide (Aldrich) were used without further purification. 2-Selenopyridine-*N*-oxide was prepared by the published procedure.[25] NMR spectra were obtained in $CDCl_3$ and are reported in ppm relative to TMS. IR spectra were obtained in KBr. Infrared spectra were obtained on a Perkin Elmer 1720-x Infrared Fourier Transform Spectrometer fitted with a diffuse reflection accessory. ^1H and ^{13}C NMR spectra were obtained on a Varian XL 300 spectrometer equipped with a 5 mm dedicated proton probe.

(2-mercaptopyridine-N-oxide)tetraphenylantimony. To a solution of 0.510 g tetraphenylantimony bromide in 20 mL of dry ethanol was added 0.120 g 2-mercaptopyridine-*N*-oxide followed by 0.50 mL of triethylamine. The reaction mixture was refluxed for 1 hour, during which time a white precipitate appeared. After cooling on an ice bath and filtering, the solvent was removed under reduced pressure. The product was crystallized from hot toluene. Recrystallization from chloroform/hexane led to 0.510 g (92% yield) pale yellow plates (mp = 199–201°C). X-ray quality crystals were obtained from slowly cooling a $CHCl_3$/ethanol solution.

Analysis calculated for **1**: C, 62.61; H, 4.35; N, 2.52. Found: C, 62.59; H, 4.47; N, 2.37.

^1H NMR: d, 7.940 (J = 6.47 Hz), 1H; d, 7.578 (J = 7.19 Hz), 8H; m, 7.233, 13H; t, 6.908 (J = 6.47 Hz), 1H; t, 6.641 (J = 6.47 Hz), 1H. ^{13}C NMR: 162.5, 146.7 (broad), 138.2, 135.0, 130.8, 129.2, 127.8, 127.0, 116.6.

IR: 3050m, 1602m, 1571w, 1544m, 1476m, 1458s, 1429s, 1302w, 1197m, 1144m, 1020m, 910w, 828m, 762m, 732s, 695s, 586m, 559m, 461s cm^{-1}.

(2-selenopyridine-N-oxide)tetraphenylantimony. To a solution of 0.511 g tetraphenylantimony bromide in 20 mL of dry ethanol under N_2 was added 0.170 g of 2-selenopyridine-*N*-oxide followed by 0.5 mL of triethylamine. A white precipitate formed rapidly and the solution was stirred for an hour at 40°C. After cooling on an icebath and filtering, the solvent was removed under reduced pressure. The solid obtained was recrystallized from toluene and hexane to give 0.485 g (80% yield) of a pale yellow solid. (mp = 181–183°C).

Analysis calculated for **2**: C, 57.74; H, 4.01; N, 2.32. Found: C, 57.05; H, 4.11; N, 2.16.

^1H NMR: d, 7.998 (J = 7.5 Hz), 1H; d, 7.592 (J = 6.6 Hz), 8H; d, 7.416 (J = 7.5 Hz), 1H; m, 7.279, 12H; t, 6.790 (J = 7.5 Hz), 1H; t, 6.690 (J = 7.5 Hz), 1H.

^{13}C NMR: 159.6, 146.9, 139.0, 134.9, 134.2, 128.4, 127.8, 127.0, 118.4.

IR: 3050m, 1598m, 1570w, 1544m, 1476m, 1457s, 1429s, 1301w, 1196m, 1128m, 1066m, 910w, 825m, 763m, 732s, 694s, 578m, 550m, 451s cm^{-1}.

Crystal structure of **1**

Crystal data. $C_{29}H_{24}NOSSb \cdot C_2H_5OH$, M = 602.40, monoclinic, a 29.095(2), b 10.6965(8), c 18.134(1), β 94.154(5), V = 5629 Å^3, Z = 8, D_c 1.42 gcm^{-3}, $F(000)$ 2448, space group C2/c suggested by systematic absences,[26] MoKα radiation, λ 0.7107 Å, μ(MoKα) 10.5 cm^{-1}, crystal size 0.39 × 0.21 × 0.08 mm.

Structure determination. Data were collected on an Enraf-Nonius Cad4 diffractometer for 4129 unique reflections of which 2733 with $F > 4\sigma(F)$ were observed. Corrections were applied to the data for Lorentz and polarization effects. An empirical absorption correction was also applied. Neutral atom scattering factors and anomalous dispersion corrections were used.[27] Structure solution was carried out by Patterson methods using SHELXS86.[28] After determination of the coordinates of the antimony atom, the nonhydrogen atoms were located on difference maps and a solvent molecule was found to be disordered along a two fold axis.[29] The structure was refined in SHELX76[30]; the full matrix least-squares refinement converged at R = 0.113 with isotropic and R = 0.065 and R_w = 0.081 with anisotropic thermal parameters[31]; the hydrogen atoms were placed at their calculated positions, but not refined. Maximum electron density in final difference map was a peak of approximately 1 eÅ^{-3}. Calculations performed on a micro-Vax-3900 computer. Refined atomic coordinates and equivalent isotropic thermal parameters are given in Table III. Additional material, including H-atom coordinates, anisotropic thermal parameters and structure factor listings, are available from the authors or the Cambridge Crystallographic Data Centre.

Downloaded At: 12:52 29 January 2011

TABLE III

Structure determination summary

Crystal Data	
Empirical Formula:	$C_{29}H_{24}NOSSb$
Formula Weight:	556.32 g mol^{-1}
Color/Habit:	yellow/lathe
Crystal Dimensions (mm):	0.39, 0.08, 0.21, along a, b, c
Crystal System:	monoclinic
Space Group:	C2/c
Unit Cell Dimensions:	29.095 Å, 10.696 Å, 18.133 Å 90.00 °, 94.15 °, 90.00 °
Unit Cell Volume:	5629 $Å^3$
Z:	8
Calculated Density:	1.41 Mg m-3
Absorption Coefficient:	10.5 cm-1
F000:	2448
Data Collection	
Diffractometer System Used:	Enraf-Nonius CAD4
Radiation:	Mo Kα (λ = 0.7107)
Temperature:	15 °C
Monochromator:	oriented graphite (0,0,2)
2 Theta Range:	2.0 to 50.0
Scan Type:	omega/2-theta
Reflections Collected:	5354
Data Reduction	
Data Reduction Program:	MolEN (Enraf-Nonius)
Absorption Correction Method:	empirical
Independent (Unique) Reflections:	4134 [R(ave) = 0.01]
Solution and Refinement	
Computer Programs Used:	SHELXS,SHELX-76,ORTEP
Solution Method:	Patterson Methods
Refinement Method:	Full-Matrix, Least-Squares
Hydrogen Atoms Treatment:	Riding x,y,z,U(iso)
Overall Scale, U (est.):	1.174, 0.057
Quantity Minimized:	Sig[w(fobs-Fcalc)2]
Weighing Scheme:	w = [sig2(F) + 0.0009*F2]$^{-1}$
Observed Reflections:	2926 [F > 4 sig(F)]
Parameters Refined:	226
Data-to-Parameter Ratio:	12.2:1
Final Parameter Shift/Error:	< 0.002
R indices (observed data):	R(F) = 0.064, wR(F) = 0.081
Goodness-of-Fit:	GOF = 1.765

Downloaded At: 12:52 29 January 2011

TABLE IV

Atomic fractional coordinates ($\times 10^4$; $\times 10^5$ for Sb) and equivalent isotropic displacement parameters (Å^2 $\times$ 10^3; $\times 10^4$ for Sb) with e.s.d.'s given in parentheses for complex **1**.

Atom	x/a	y/b	z/c	U(eq)[a]
Sb	14682(3)	13827(7)	55615(4)	490(2)
S	1360(1)	3071(3)	6654(2)	65(1)
O	1893(3)	3062(7)	5347(4)	59(2)
N	1694(3)	4167(10)	5484(5)	63(3)
C1	1758(5)	5074(13)	5002(7)	77(4)
C2	1591(6)	6230(14)	5093(9)	101(5)
C3	1354(6)	6453(15)	5660(10)	109(6)
C4	1264(5)	5510(14)	6163(8)	92(4)
C5	1435(1)	4297(3)	6082(2)	66(3)
C6[b]	932(1)	2445(3)	4949(2)	62(3)
C7	1001(1)	2834(3)	4231(2)	82(4)
C8	668(1)	3552(3)	3836(2)	120(6)
C9	264(1)	3882(3)	4160(2)	137(7)
C10	195(1)	3494(3)	4877(2)	100(5)
C11	529(1)	2775(3)	5272(2)	79(4)
C12[b]	1033(1)	103(3)	6096(2)	60(3)
C13	1169(1)	-357(3)	6798(2)	79(4)
C14	879(1)	-1161(3)	7154(2)	101(5)
C15	454(1)	-1504(3)	6809(2)	121(6)
C16	318(1)	-1043(3)	6107(2)	109(5)
C17	608(1)	-240(3)	5751(2)	77(4)
C18[b]	2097(1)	841(3)	6173(2)	60(3)
C19	2410(1)	1651(3)	6546(2)	74(4)
C20	2806(1)	1177(3)	6925(2)	101(5)
C21	2890(1)	-108(3)	6932(2)	122(7)
C22	2577(1)	-918(3)	6559(2)	110(5)
C23	2180(1)	-443(3)	6180(2)	79(4)
C24[b]	1635(1)	398(3)	4580(2)	53(3)
C25	2065(1)	597(3)	4309(2)	80(3)
C26	2189(1)	-54(3)	3687(2)	93(5)

Downloaded At: 12:52 29 January 2011

TABLE IV (*Continued*)

Atom	x/a	y/b	z/c	U(eq)[a]
C27	1882(1)	-902(3)	3335(2)	88(4)
C28	1451(1)	-1100(3)	3606(2)	87(4)
C29	1328(1)	-450(3)	4229(2)	71(3)
O1a[c]	5000	1042(15)	2500	99(5)
C2a	4713(5)	1938(15)	2030(7)	179(7)
C3a	4903(6)	3296(16)	2233(10)	121(6)
C1b	5000	3526(15)	2500	135(7)
C2b	4757(5)	3324(15)	1836(8)	130(7)
C3b	4611(6)	2023(16)	1641(9)	97(5)

[a] U(eq) is the mean of the principal axes of the thermal ellipsoid.

[b] Phenyl rings C6-C11, C12-C17, C18-C23, C24-C29 refined as rigid groups.

[c] U_{iso} is given for ethanol.

Downloaded At: 12:52 29 January 2011

ACKNOWLEDGEMENTS

This work has been supported by a Schering-Plough Grant of Research Corporation (C-2891) and an American Cyanamid Corporation Grant-in-Aid. We thank Professor Helen M. Berman in whose laboratory the x-ray data were collected. We also thank Joseph Furch for assistance with NMR spectra. Biological assays were carried out at the Walter Reed Army Institute of Research. The authors wish to express indebtedness to Dr. Richard Stockel for many helpful discussions.

REFERENCES

1. A. Hodge, K. Nordquest and E. L. Blinn, *Inorg. Chim. Acta*, **3,** 491 (1972).
2. B. Song, Z. Lu, D. Niu, Y. Cao and C. Zeng, *Chin. Chem. Lett.*, **1,** 117 (1990).
3. B. Xu, G. Han, L. Meng, L. Chen, Z. Dai and Z. Ma, *Yaoxue Tongbao*, **17,** 302 (1982).
4. P. Kovicac, M. A. Kassel, J. R. Ames, B. A. Feinberg and W. Sliwa, *J. Biopharm. Sci.*, **1,** 331 (1990).
5. S. I. Shupack and M. Orchin, *J. Am. Chem. Soc.*, **85,** 902 (1963).
6. V. K. Jain, J. Mason and R. C. Mehrotra, *J. Organomet. Chem.*, **309,** 45 (1986).
7. V. K. Jain, R. Bohra and R. C. Mehrotra, Structure and Bonding in Organic Derivatives of Antimony(V), *Struct. Bonding*, **52,** 147 (1982).
8. D. B. Sowerby, *J. Chem. Res. (S)*, 80 (1979).
9. G. Ferguson, C. Glidewell, B. Kaitner, D. Lloyd and C. Metcalfe, *Acta Cryst.*, **C43,** 824 (1987).
10. R. Ruether, F. Huber and H. Preut, *J. Organomet. Chem.*, **295,** 21 (1985).
11. H. Preut, F. Huber and K.-H. Hengstmann, *Acta Cryst.*, **C44,** 468 (1988).
12. J. Kroon, J. B. Hulscher and A. F. Peerdeman, *J. Organomet. Chem.*, **37,** 297 (1972).
13. N. Kanehisa, K. Onuma, S. Uda, K. Hirabayashi, Y. Kai, N. Yasuoka and N. Kasai, *Bull. Chem. Soc. Jpn.*, **51,** 2222 (1978).
14. S. P. Bone and D. B. Sowerby, *J. Chem. Res. (S)*, 82 (1979).
15. R. D. Haugwitz, B. Toeplitz and J. Z. Gougoutas, *Cryst. Struct. Comm.*, **9,** 937 (1980).
16. G. M. Arvanitis, M. E. Berardini and D. Allardice, manuscript in preparation.
17. J. L. Wardell and D. W. Grant, *J. Organomet. Chem.*, **198,** 121 (1980).
18. J. P. Kreier, Ed., Parasitic Protozoa, Volumes I–IV, Academic Press, New York (1977).
19. D. H. Molyneux and R. W. Ashford, in Trypanosoma and Leishmania, Taylor and Francis, pp. 183–249 (1983).

20. J. Maurice, Ed., *Tropical Disease Research News. WHO*, **34,** 1 (1990).
21. J. Maurice, Ed., *Tropical Disease Research News. WHO*, **36,** 1 (1991).
22. R. E. Desjardins, C. J. Canfield, J. D. Haynes and J. D. Chulay, *Antimicrob. Agents Chemother.*, **16,** 1710 (1979).
23. J. Maurice and A. M. Pearce, Tropical Disease Research: A Global Partnership, World Health Organization, Geneva, pp. 24–31, (1987).
24. P. E. Dumas, R. Stockel and G. M. Arvanitis, *Proceedings of the Fourth International Symposium on Uses of Selenium and Tellurium*, Banff, Alberta, Canada, 456 (1989).
25. H. G. Mautner, S. H. Chu and C. M. Lee, *J. Org. Chem.*, **27,** 3671 (1962).
26. The space group assignment was also supported by the statistical distribution of $|E^2 - 1|$. Solution of the structure in the space group Cc did not provide a better model for the data.
27. International Tables for X-Ray Crystallography, Kynoch Press, Birmingham, Vol 4. 1974.
28. G. M. Sheldrick, SHELXS86. A Program for the Solution of Crystal Structures, Univ. of Gottingen, Germany, 1986.
29. The disordered ethanol was best refined with isotropic thermal parameters for two positions each with an occupancy factor of 0.5. The interatomic distances and angles for the solvent were normal and there are no close contacts involving ethanol molecules.
30. G. M. Sheldrick, SHELX76. A Program for Crystal Structure Determination, Univ. of Cambridge, England, 1976.
31. Attempts to model the phenyl ring carbon atoms C8–C10 and C20–C22 by partial occupancy of disordered groups did not provide significantly improved R indices.

Downloaded At: 12:52 29 January 2011