BISMUTH

ANNUAL SURVEY COVERING THE YEAR 1981 *

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No reviews devoted solely to organobismuth compounds have appeared in 1981. The annual survey of organometallic compounds published by the Chemical Society [1] includes organobismuth compounds, and they are also briefly mentioned in the 1981 supplement to *Rodd's Chemistry of Carbon Compounds* [2].

Two bismuthines containing chiral bismuth atoms have been reported by Bras and coworkers [3]. The first compound, $4\text{-MeC}_6H_4Bi(Br)C_6H_4CH_2NMe_2\text{-}2$, was prepared by the reaction of *p*-tolyldibromobismuthine with 2-dimethylaminomethylphenyllithium. The PMR spectrum of this compound in $CDCl_3$ revealed that the benzylic protons gave two signals at $\delta = 3.55$ and $\delta = 3.87$. These protons are diastereotopic due to the chirality of the bismuth atom. The two methyl signals for the two methyl groups attached to nitrogen also occurred as doublets. This result was explained by assuming that the lone pair electrons on the nitrogen coordinated with the bismuth atom and hence that the nitrogen became a prochiral center. This was confirmed by the fact that the ^{13}C spectrum gave two signals for the two carbons attached to the nitrogen. Thus the bismuth atom is five coordinate with a lone pair occupying one of the five positions. The diastereotopic effect of the NMe_2 and NCH_2 protons was found to be solvent dependent, varying considerably in such solvents as $CDCl_3$, $(CD_3)_2CO$, C_6D_6, and $(CD_3)_2SO$. In the last solvent the signals for both the benzylic protons and the methyl protons attached to nitrogen become singlets. The authors explained this result by concluding that in the strongly coordinating solvent DMSO the nitrogen was no longer coordinated to bismuth so that both methyl groups on nitrogen were equivalent. In the absence of the coordinating nitrogen atom there was rapid inversion around the bismuth atom on the NMR time scale so that the methylene protons became equivalent. This result is rather surprising since chiral phosphines, arsines, and stibines do not rapidly invert.

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*Bismuth; Annual Survey covering the year 1980 see J. Organometal. Chem., 227 (1982) 57-60.

The second compound containing a chiral bismuth atom was $4\text{-MeC}_6H_4Bi(Br)C_6H_4CH_2OMe\text{-}2$, obtained from $4\text{-MeC}_6H_4BiBr_2$ and 2-BrMgC_6H_4OMe. This bismuthine also contained a diastereotopic bismuth atom and gave two signals for the methylene protons in $CDCl_3$ solution, but only one signal in $(CD_3)_2CO$ and in C_6D_6. This result was explained by assuming that the ether oxygen was more weakly coordinating than the nitrogen atom and hence that in C_6D_6 and in $(CD_3)_2CO$, the oxygen was not coordinated to the bismuth atom.

The reaction between triphenylbismuthine and the mercapto acids, mercaptoacetic, 3-mercaptopropionic, 2-mercaptopropionic, or 2-mercaptobenzoic, has been found to be dependent on both the solvent and on the concentrations of the reactants [4]. At room temperature in chloroform (chloroform and methanol in the case of 2-mercaptobenzoic acid) all three carbon-bismuth bonds were cleaved to give three moles of benzene and an inorganic bismuth compound:

$$Ph_3Bi + 2\ HSRCO_2H \longrightarrow 3\ PhH + Bi(SRCO_2)(SR(H)CO_2)$$

The same bismuth compounds were readily prepared from bismuth hydroxide and two molar equivalents of the same mercapto acids. The reaction between lower concentrations of 3-mercaptopropionic acid or 2-mercaptobenzoic acid and triphenylbismuthine in acetone led to the cleavage of only two carbon-bismuth bonds:

$$Ph_3Bi + HSRCO_2H \longrightarrow PhBi(SRCO_2) + 2\ PhH$$

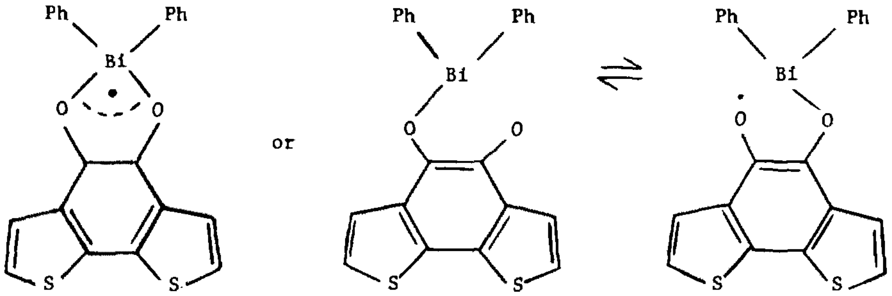
The reaction of all four mercapto acids and trimethylbismuthine in acetone solution also led to the cleavage of only two carbon-bismuth bonds:

$$Me_3Bi + HSRCO_2H \longrightarrow MeBi(SRCO_2) + 2\ MeH$$

This latter reaction appeared to be concentration independent.

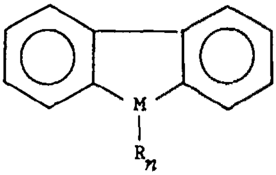
The structure of the bismuth-containing products was investigated by means of their IR and PMR spectra. It was determined that the two products $Bi(SRCO_2)(SR(H)CO_2)$, derived from mercaptoacetic and 3-mercaptopropionic acids, each contained a free carboxyl group but no free mercapto group, whereas the products obtained from 2-mercaptopropionic acid or 2-mercaptobenzoic acid each contained a free mercapto group but no free carboxyl group. All four of these products were insoluble in polar and non-polar organic solvents (except for pyridine) and were probably polymeric in the solid state. The organic compounds $PhBi(SRCO_2)$ and $MeBi(SRCO_2)$ were insoluble in organic solvents including pyridine and were also believed to be polymers. The exact structure of all of the polymeric products could not be determined on the basis of their PMR and IR spectra.

ESR spectroscopy has been used to study the paramagnetic adducts formed by the reaction of several inorganic Group VA halides ($AsCl_3$, $SbCl_3$, SbF_3, and $BiCl_3$) and a number of organoarsenic, -antimony, and -bismuth compounds with benzo[2,1-b;3,4-b']dithiophene-4,5-dione (TQ) [5]. The only organobismuth compound used was Ph_3Bi. The solvents were diglyme, tetraglyme, and molten biphenyl. The ESR spectra of the bismuth-containing adducts were more complex than those obtained from the arsenic or antimony compounds. Thus, with Ph_3Bi and TQ in diglyme only a single species was obtained as judged by the ESR spectrum. This was believed to be a radical of the type $[TQBiPh_2]^\cdot$ in which either the metal was four coordinate or there was a rapid dynamic equilibrium between two equivalent tautomers:



The spectrum obtained in molten biphenyl was more complex and contained at least two and probably three different bismuth-containing radicals.

Davydov and coworkers [6] have studied the singlet and triplet state energies, luminescence properties, and triplet-triplet absorption spectra of a series of fluorene-like molecules of the following type:

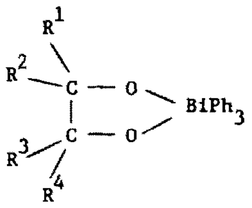


(where M was a Group IV element, C, Si, Ge, or Sn, a Group V element N, P, As, Sb, or Bi or a Group VI element, O, S or Se; R was an alkyl or aryl group; and n = 0-2)

In the one bismuth compound studied R = Ph, n = 1. The results of this study are also discussed in the Antimony section.

In a paper written for a special issue of *Tetrahedron* in honor of the Nobel prize-winning chemist, R.B. Woodward, Barton and coworkers [7] have summarized and expanded on their recent communications dealing with the use of organobismuth(V) reagents as selective oxidizing agents in organic chemistry. They first gave (in tabular form) the results of the oxidation of a variety of primary and secondary alcohols (many of which are complex natural products) with the organobismuth reagents μ-oxobis-(chlorotriphenylbismuth), $(Ph_3BiCl)_2O$, or triphenylbismuth carbonate, as well as the oxidation of several thiols to disulfides with Ph_3BiCO_3. The cleavage of several vicinal diols by both of these reagents was also listed. Experimental details of these reactions, not given in the earlier communications, were given in sufficient detail. Also described were oxidations effected by several other organobismuth(V) reagents, *e.g.* $Ph_3Bi(OAc)_2$, $Ph_3Bi(O_2CCF_3)_2$, $(p\text{-}MeC_6H_4)_3Bi(O_2CPh)_2$, and $(p\text{-}MeOC_6H_4)_3BiCO_3$. Methods used for preparing these and other organobismuth(V) compounds were given, and an improved synthesis of triphenylbismuth carbonate was described.

A mechanism for the oxidation reactions which involved two competing reaction pathways was outlined (this mechanism had also been suggested in an earlier paper). One pathway involved the formation of Ar_3Bi; the other pathway gave Ar_2BiY (from Ar_3BiY_2). This dual pathway mechanism was necessitated by the fact that less than one mole of Ar_3Bi was formed for each mole of oxidation product (ketone or aldehyde). By contrast the cleavage of vicinal glycols by Ph_3BiCO_3 or $(Ph_3BiCl)_2O$ invariably gave a quantitative yield of Ph_3Bi. It was also mentioned that the cleavage of *cis*-1,2-cyclohexanediol by Ph_3BiCO_3 was considerably faster than cleavage of the *trans-isomer*. (Only cleavage of the *cis*-isomer was mentioned in earlier papers.) These two observations suggested to the authors that the glycol cleavage reaction involved the formation of a cyclic intermediate:



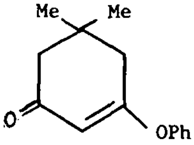
Breakdown of this intermediate then resulted in the exclusive formation of triphenylbismuthine. The authors predicted that it should be possible to effect a catalytic cycle involving the cleavage of vicinal glycols by a triarylbismuthine and excess of an oxidizing agent. Accordingly, the cleavage of *meso*-hydrobenzoin was attempted with amounts of triphenylbismuthine ranging from 1.0 to 0.05 equivalents. Hydrogen peroxide (3 equivalents in the presence of sodium bicarbonate) or *tert*-butyl hydroperoxide (3 equivalents) were the oxidizing agents. The yields of benzaldehyde under these reaction conditions ranged from 98% to 70%. The 98% yield was effected with H_2O_2 and $NaHCO_3$ and one equivalent of Ph_3Bi in one hour; the 70% yield used the name reagents with 0.05 equivalents of Ph_3Bi but required 16 hours. The authors stated that they had so far been unable to extend this cleavage reaction to other glycols.

In a second paper Barton and coworkers [8] reported that the cleavage of vicinal glycols by triphenylbismuthine and an oxidizing agent could be readily achieved when the oxidizing agent was either *N*-bromosuccinimide or *N*-bromoacetamide. Bromine could also be used, but was not as successful as the other two oxidants. The presence of sodium carbonate and a small amount of water was also necessary for the reaction to proceed. The yields of aldehyde or ketone were comparable or superior to those obtained when lead tetraacetate or sodium periodate was used for cleaving the glycol. The amount of triphenylbismuthine used in the reactions varied between 0.01 and 0.1 equivalent. Although longer reaction periods apparently were necessary with the smaller amount of reagent, the yields were essentially the same for all of the above amounts of triphenylbismuthine. Thus, for the cleavage of *meso*-hydrobenzoin with 0.10, 0.05, and 0.01 equivalent of triphenylbismuthine and reaction times of 5, 10, and 20 minutes, the yields of benzaldehyde were 89%, 91%, and 85%, respectively.

The reaction is of particular value in the cleavage of vicinal glycols obtained from natural products. Thus, the cleavage of 1,2:5,6-di-O-isopropylidene-D-mannitol was achieved, without racemization, in 72% yield with 0.1 equivalent of triphenylbismuthine; the reaction time was 30 minutes. Experimental details of this reaction were described. The cleavage of both *cis*- and *trans*-decalin-9,10-diol was achieved at about the same rates with the same amounts of triphenylbismuthine. The authors commented that this result suggested that cleavage of the diol did not involve formation of a cyclic intermediate. A mechanism for the cleavage reaction was suggested that involved the formation of an intermediate $Ph_3Bi(OH)Br$ and subsequent attack by carbonate ion to form Ph_3BiO or $[Ph_3Bi(OH)OCO_2]K$. Either of these latter compounds could then act as the active oxidant.

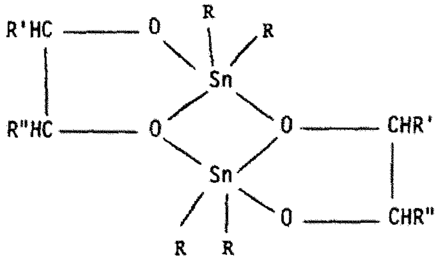
In earlier publications by Barton and coworkers on the use of organobismuth(V) compounds as reagents in organic synthesis, it was noted that these compounds could serve as either oxidizing agents or as phenylating agents. Among the reactions noted previously was the formation of *p*-nitrophenyl phenyl ether from *p*-nitrophenol and pentaphenylbismuth [9]. The mechanism suggested for this reaction involved the formation of an intermediate containing the $-O-BiPh_4$ moiety. This intermediate subsequently underwent preferential reductive elimination and formation of an aryl-oxygen bond. In the present paper Barton and coworkers [10] have suggested that use of an organobismuth(V) reagent containing a powerful electron-attracting group might facilitate ether formation. Accordingly, they prepared tetraphenylbismuth trifluoroacetate from pentaphenylbismuth and trifluoroacetic acid. And, indeed, this reagent proved to give satisfactory yields of unsymmetrical ethers when allowed to react with phenols. The phenols used and the yields of ethers obtained were: 2-naphthol (77%), 2,6-dimethylphenol (58%), 2,3,5,6-tetramethylphenol (57%), estrol (68%), and *p*-nitrophenol (51%). A slight modification of the method used with *p*-nitrophenol increased the yield of the ether to 70%.

The reaction of tetraphenylbismuth trifluoroacetate with readily enolizable compounds also led to the formation of ethers. Thus, dimedone gave the following ether in 55% yield:



2-Carboethoxycyclohexanone gave a 36% yield of the ether together with a 7% yield of 2-carboethoxy-2-phenylcyclohexanone. When tetraphenylbismuth acetate was used (rather than the trifluoroacetate), only C-phenylation was observed. Unsymmetrical diaryl sulfides were obtained from aryl thiols and tetraphenylbismuth trifluoroacetate. The yields of the compounds PhSAr were as follows: Ar = Ph, 70%; Ar = $o\text{-}C_6H_4Me$, 78%; Ar = $p\text{-}C_6H_4Me$, 70%. These yields were larger than those obtained by the reaction of pentaphenylbismuth with the same thiols [9]. *p*-Toluenesulfinic acid underwent reaction with either pentaphenylbismuth or with tetraphenylbismuth trifluoroacetate to give *p*-tolyl phenyl sulfone in 87% and 76% yields, respectively. The same product was obtained in much smaller yield (27%) from sodium *p*-toluenesulfinate and tetraphenylbismuth carbonate in aqueous medium.

A well known reaction in organic chemistry is the cleavage of vicinal diols with strong oxidizing agents such as $Pb(OAc)_4$ or $NaIO_4$. David and Thiéffry [11] have now reported the cleavage of tin derivatives of vicinal diols, dibutylstannylenes, by a variety of oxidizing agents, including triphenylbismuth diacetate. The dibutylstannylenes exist as dimers with the following structure:



(where R = Bu)

In the dibutylstannylene used in this study, R' = $PhCH_2CH_2$ and R" = $PhCH_2$. The products of the reaction were the aldehydes $PhCH_2CHO$ and $PhCH_2CH_2CHO$.

Since it had previously been shown [12] that triphenylbismuth carbonate, in the presence of a strong base, effected cleavage of vicinal diols, the authors attempted to cleave the diol *erythro*-$PhCH_2CH_2CHOHCHOHCH_2Ph$ with triphenylbismuth diacetate by refluxing the reactants in CH_2Cl_2 solution without the addition of base. Instead of cleaving the C-C bond the reaction produced two compounds which, after separation by column chromatography, were characterized by IR, PMR, and mass spectrometry as the two ethers $PhCH_2CH(OPh)CHOHCH_2CH_2Ph$ and $PhCH_2CHOHCH(OPh)CH_2CH_2Ph$. In a second experiment *trans*-1,2-cyclohexanediol, refluxed for four hours with triphenylbismuth diacetate, gave an 88% yield of *trans*-2-phenoxycyclohexanol. This reaction, the phenylation of one hydroxyl group of a vicinal diol, appeared to be specific for diols, since cyclohexanol was found not to react with triphenylbismuth diacetate when the two were refluxed for seven hours in methylene dichloride.

The above reaction (phenylation of one hydroxyl group of a vicinal diol by triphenylbismuth diacetate) was later extended to a large series of diols [13]. It was found that when the hydroxyl groups were on primary or secondary carbon atoms of vicinal diols, excellent yields of the hydroxy ethers were obtained. Thus ethylene glycol, 2,3-butanediol, and 4,5-octanediol gave 85%, 86%, and 91% of the corresponding hydroxy ethers, respectively. *meso*-1,2-Diphenylethylene glycol gave only a 37% yield of the corresponding hydroxy ether. The yields of ether with *cis*- and *trans*-cyclopentane-1,2-diols, and with *cis*- and *trans*-cyclohexane-1,2-diols were 41%, 51%, 87%, and 88%, respectively. When the hydroxyl group was on a tertiary carbon atom the yields were greatly reduced.

Pinacol gave only a 15% yield of the ether after refluxing for 24 hours. Tetraphenylethylene glycol failed to react with triphenylbismuth diacetate. Furthermore, when the hydroxyl groups were on both secondary and tertiary carbon atoms, only the hydroxyl group on the secondary carbon atom underwent phenylation. Phenylation of one hydroxyl group of a glycol was not, however, confined to vicinal diols. Thus propane-1,3-diol, butane-1, 4-diol, pentane-1,5-diol, and hexane-1,6-diol all gave the corresponding hydroxy ethers. (*Z*)-2-Butene-1,4-diol also gave the hydroxy ether. In a few cases some glycol cleavage was observed in addition to monophenylation. 1,2-Diphenylpropane-1,2-diol gave no phenyl ether under the general reaction conditions used, but when one equivalent of acetic acid was added to the reaction mixture the yield of cleavage products was reduced to 26%.

The use of organobismuth(V) compounds as oxidizing agents for primary or secondary alcohols has also been the subject of a patent by Barton and Motherwell [14]. The organobismuth compounds described in the patent were of the type R_3BiY_2 or $(R_3BiY)_2O$, where Y was an ionizable group. The alcohols mentioned in the patent were principally from natural products, *i.e.* steroidal, terpene, or sugar alcohols. Thus cholestanol was oxidized to chlolestanone in 75% yield by $(Ph_3BiCl)_2O$ in the presence of K_2CO_3.

Oxidation of primary and secondary alcohols, ROH, where R = Et, Bu, $PhCH_2$, Me_2CH, or EtCHMe, to the corresponding aldehydes or ketones by an organobismuth(V) reagent, $Ph_3Bi(OAc)_2$, has also been described by Russian authors [15]. The reactions were carried out in sealed, evacuated ampules at 80-100°C. In addition to the aldehydes and ketones, other products of the reaction were esters of the type ROAc, benzene, and compounds of the type $Ph_xBi(OAc)_y$.

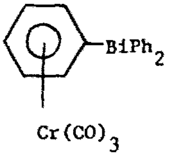
The preparation of a series of carbonylvanadium, carbonylmanganese, and carbonylmolybdenum complexes formed by the photoinduced reaction of the compounds $[Et_4N][V(CO)_6]$, η^5-$C_5H_5V(CO)_4$, η^5-$C_5H_5Mn(CO)_3$, or η^5-$C_5H_5Mo(CO)_3Me$, with the ligands *o*-$C_6H_4(EPh_2)(E'Ph_2)$ (where E and E' were group VA elements, P, As, Sb, or Bi), has been described [16]. One bismuth-containing ligand was reported, namely, *o*-$C_6H_4PPh_2(BiPh_2)$. It was prepared by means of the following reactions:

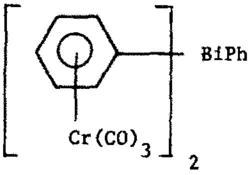
$$o\text{-}C_6H_4(Br)PPh_2 \;+\; 2\,Li \longrightarrow o\text{-}C_6H_4(Li)PPh_2 \;+\; LiBr$$

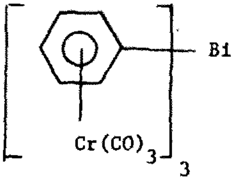
$$o\text{-}C_6H_4(Li)PPh_2 \;+\; Ph_2BiCl \longrightarrow o\text{-}C_6H_4PPh_2(BiPh_2) + LiCl$$

The product was recrystallized from 1-butanol. The mass spectrum of the compound exhibited the molecular ion. Five products were prepared from this bismuth-containing ligand L, *viz.* $[Et_4N][V(CO)_5L]$, *cis*-$[Et_4N][V(CO)_4L]$, η^5-$C_5H_5V(CO)_3L$, η^5-$C_5H_5Mn(CO)_2L$, and η^5-$C_5H_5Mo(CO)Me(L)$. None of these complexes were obtained in pure form, however. In contrast, most of the complexes obtained from the ligands o-$C_6H_4(EPh_2)(E'Ph_2)$, where E and E' were P, As, or Sb, were obtained in pure crystalline form and were characterized by elemental analyses.

The reaction between triamminechromium tricarbonyl and triphenylbismuthine or triphenylstibine have been investigated by Alad'in and coworkers [17]. In the case of triphenylbismuthine, the reaction was carried out by heating the chromium compound with three equivalents of Ph_3Bi in an inert atmosphere in dioxane solution. Three compounds were obtained which were readily separated by their differential solubility in different organic solvents. The three compounds were π-complexes with the following structures:

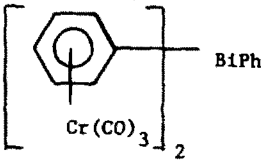






The above three compounds were obtained in 13%, 43%, and 31% yields, respectively. The PMR spectra of the first two compounds gave signals for protons of both the π-bonded phenyl groups and the phenyl groups bonded only to the bismuth atoms. The PMR spectrum of the third compound showed only one type of proton. The IR spectra of the compounds were also in accord with the assigned structures. The three bismuth compounds were yellow crystalline solids which were relatively air stable but extremely light sensitive. They slowly oxidized in the air, and decomposed rapidly in solution. The thermal behavior of the tricoordinated compound was studied by differential thermal analysis which showed two modes of decomposition involving both cleavage of the Ph-Cr bond and cleavage of the C-Bi bond with formation of metallic bismuth.

The crystal and molecular structure of one of the above compounds,



has been determined by X-ray diffraction [18].

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The NQR spectrum of triphenylbismuthine has been determined by Weaver and Robinson [19] at 77K ± 0.5 K. Since ^{209}Bi has a spin of 9/2, the spectrum consisted of four lines with frequencies of 30.625, 56.441, 85.435, and 119.019 MHz. The frequencies were temperature dependent and were about 3% higher than values obtained for triphenylbismuthine at room temperature. The line width of the lowest frequency was 36 kHz; the widths of the other lines were 12 kHz. Values of the coupling constant e^2qQ and the anisotropy factor η were 684,570 ± 10 kHz and 0.0903 ± 0.006, respectively.

The complexing of molecular oxygen with compounds of the type Ph_3E, where E was N, P, As, Sb, and Bi, has been studied by electronic spectroscopy [20]. It was found that the coordination was promoted by an increase in the π-electron density of the phenyl rings. As a result, triphenylamine had a high donor ability; the donor ability of triphenylphosphine was much lower. The π-donor abilities of triphenylarsine, -stibine, and -bismuthine were approximately equal and were less than that of triphenylphosphine.

The β-cleavage of C_6T_6 led to the cation $C_6T_5^+$ which reacted with Ph_3E compounds (where E was a group VA element) to give compounds of the type $[C_6T_5EPh_3]BF_4$ [21]. The yield where E was Bi was 10.8%.

Triphenylbismuthine (as well as the triphenyl derivatives of N, P, As, and Sb) underwent one-electron oxidation by radical cations of the type $(4\text{-}RC_6H_4)_3N^{+\cdot}$ (where R was MeO, Me, Ph, or Br) [22]. Both ESR and electronic spectroscopy were used in this investigation. The reactions obeyed a second order rate law; the rate was strongly dependent on the nature of R and on the Group V element. Triphenylbismuthine was the least effective one-electron reducing agent among the Group V triphenyl compounds. This paper is discussed in greater detail in the Antimony section.

In a paper devoted principally to cyclic voltammetry redox potentials of tertiary phosphines, triphenylbismuthine (as well as triphenylarsine and triphenylstibine) was mentioned [23]. The bismuthine exhibited an irreversible redox potential of + 1.21 V (vs. the SCE).

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