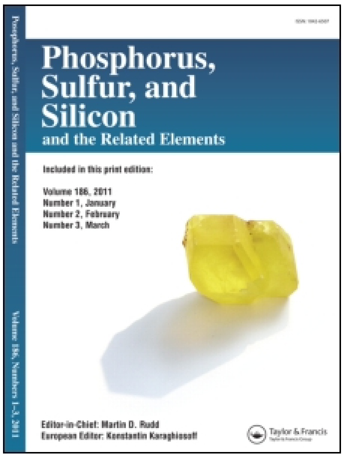

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# On the Reaction of Triaryl Trithioarsenites, $(ArS)_3As$, with Iodine

Maria D. Markaki[a]; Panayiotis V. Ioannou[a]
[a] Department of Chemistry, University of Patras, Patras, Greece

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# On the Reaction of Triaryl Trithioarsenites, $(ArS)_3As$, with Iodine

**Maria D. Markaki**
**Panayiotis V. Ioannou**
Department of Chemistry, University of Patras, Patras, Greece

*Iodine in dry nonprotic solvents oxidizes triaryl trithioarsenites, $(ArS)_3As$, to As(III) iodide, $AsI_3$, and disulfides, ArSSAr. The reaction most likely involves arylsulfenyl iodide, ArSI, as an intermediate. With triphenyl and tris(4-chlorophenyl) trithioarsenites, $AsI_3$ is prepared in very good yields. When the disulfide, which is produced, has MeCONH— or —NH—$CMe_2$—OH groups, then it acts as a Lewis base towards $AsI_3$ forming complexes with stoichiometry $2AsI_3 \cdot 3ArSSAr$. Probable coordination modes of the $AsI_3$ are discussed.*

**Keywords** Arsenic triiodide; arsenic triiodide as Lewis acid; diphenyl aryldithioarsonites; disulfides; iodine; triaryl trithioarsenites

## INTRODUCTION

We have recently been studying the effect of the oxidizing element octasulfur on dithioarsonites [$L\text{-}As(SPh)_2$, L = Ar or R][1] and on triaryl[2] and trialkyl[3] trithioarsenites [$(ArS)_3As$ and $(RS)_3As$], spurred by the (erroneous) report[4] that $(p\text{-}MePhS)_3As$ is oxidized by octasulfur in refluxing carbon disulfide to the ester $(p\text{-}MePhS)_3As{=}S$. We have found that the previously mentioned As(III) esters are not oxidized to the As(V) ones probably because the As(III) is not nucleophilic towards octasulfur.

Next, we studied the air oxidation of triaryl[5] and trialkyl[3] trithioarsenites, and we found that electron-withdrawing groups at the *para* position of the phenyl group tend to favor the oxidative decomposition compared to those having electron-donating groups, according to Eq. (1):

$$2(ArS)_3As + 3/2O_2 \rightarrow As_2O_3 + 3ArSSAr \qquad (1)$$

Aliphatic trithioarsenites bearing a terminal —COOH were decomposed by air (Eq. 1), while those bearing a $—COO^-$ group were

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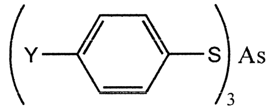
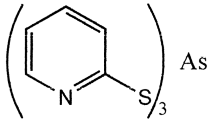
Address correspondence to Panayiotis V. Ioannou, University of Patras, Department of Chemistry, Patras, Greece. E-mail: ioannou@chemistry.upatras.gr

resistant to air oxidation. The esters As(glutathione)$_3$, As(cysteine)$_3$, and As(penicillamine)$_3$ were found to be air oxidized at a slower rate than their parent thiols. These and other[6] oxidative decomposition results have been rationalized by assuming dioxygen binding to As(III). The adduct then decomposes to the observed products.

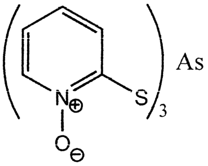
Stronger oxidizing elements other than octasulfur and dioxygen are the halogens. The action of iodine, dissolved in ethanol, on the ester **1** with an unspecified stoichiometry was reported[7] to give diphenyl disulfide, but the fate of the arsenic was not determined.



**1** Y = H
**2** Y = Cl
**3** Y = $NO_2$
**4** Y = $NHCOCH_3$
**5** Y = $NH_2$



**6**



**7**

**FORMULAS 1–7**

In this article, we report our results on the action of iodine and, in one case, bromine on the trithioarsenites **1–7**. We found that the reaction of iodine with the esters **1–7** is simpler in nonprotic solvents; arsenic triiodide is produced that either precipitates out or forms Lewis adducts when the produced disulfide has certain nitrogen and/or oxygen donor atoms.

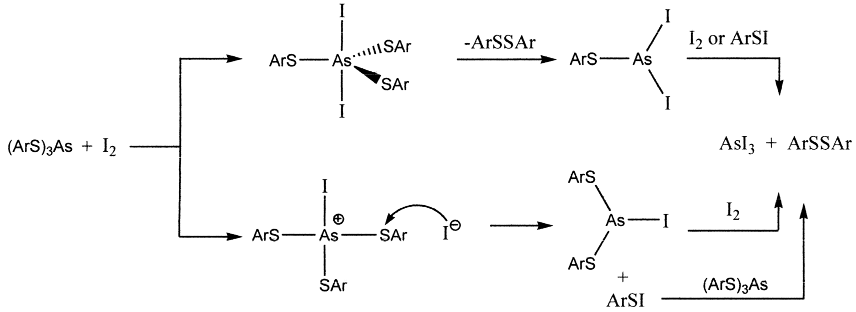
## RESULTS AND DISCUSSION

The reaction of **1** with iodine in methanol under various stoichiometries gave a complex mixture of products, among them diphenyl disulfide, as revealed by TLC analyses. However, in dry nonprotic solvents, e.g., chloroform, acetone, ether, **1** reacted cleanly with iodine giving, by TLC, only two compounds with the stoichiometry of Eq. (2):

$$2(PhS)_3As + 3I_2 \rightarrow 2AsI_3 + 3PhSSPh \qquad (2)$$

The best solvent for the isolation of $AsI_3$ was ether, in which the triiodide is sparingly soluble (1:500).[8] The yields, 85–90%, compare favorably with those reported for its preparation in aqueous media[8a,9] using $As_2O_3$/HCl/KI. Because the ester **1** is easily prepared and is stable to storage,[10] the reaction (2) can be useful for preparations of milligram amounts of arsenic triiodide. The ester **2** gave $AsI_3$ (75–80%), while from the reaction of **3** with iodine, pure bis(4-nitrophenyl) disulfide, was isolated.

The most likely mechanism for the reaction (2) is shown in Scheme 1. It is not easy to say which of the two intermediates, the tetracoordinated $(ArS)_3AsI^+I^-$ or the pentacoordinated $(ArS)_3As(I_2)$, is preferred.

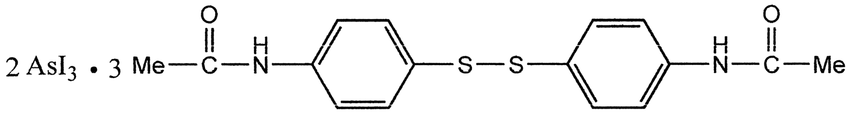


**SCHEME 1**

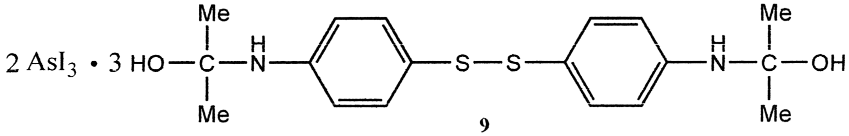
The mechanism suggests that arylsulfenyl iodide ArSI is an intermediate, and we sought a preparation of phenylsulfenyl iodide to test this hypothesis. However, arylsulfenyl iodides are only accessible in special instances,[11] and the use of phenylsulfenyl iodide in the literature[12] is questionable. We therefore prepared phenylsulfenyl bromide,[13] contaminated by diphenyl disulfide, by reacting diphenyl disulfide with bromine,[14] and reacted it with **1** with a 3:1 stoichiometry. The reaction was very fast, and **1** was not detected by TLC. Therefore, there are strong indications that phenylsulfenyl iodide is an intermediate in the reaction (2), and its reactivity explains the complex mixture of products obtained when the reaction (2) was done in methanol.

The oxidation of the trithioarsenite **1** with bromine gave diphenyl disulfide and $AsBr_3$, which could not be separated. Overall, iodine and bromine can therefore oxidize trithioarsenites to $AsX_3$ plus disulfides, and the use of the solid iodine is more convenient for preparative purposes.

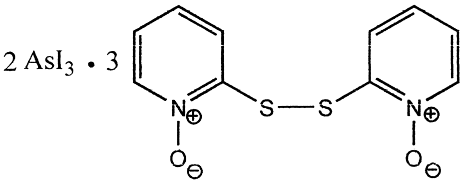
Iodine should oxidize esters **4–7** to $AsI_3$ and disulfide. The gum produced from **6** could not be crystallized, while the powdery solids isolated from the reaction of **4**, **5**, and **7** with iodine were the Lewis adducts **8–10**, in which each $AsI_3$ acts as a Lewis acid formally coordinated to three donor atoms. Because $AsI_3$ is not coordinated to the —S—S— of diphenyl disulfide, it follows that it must be coordinated to N and/or O atoms in **8–10**, and it is not extracted by carbon disulfide in which it is soluble (1:18).[8]



**8**



**9**



**10**

**FORMULAS 8–10**

The first adduct of $AsI_3$, acting as a Lewis acid, $AsI_3 \cdot 3PhNH_2$, was prepared by H. Schiff in 1863 and other earlier examples[8a] are $AsI_3 \cdot xpy$ ($x = 2, 3$), $AsI_3 \cdot xNH_3$ ($x = 3, 4, 12$), and $AsI_3 \cdot Me_2(Ph)As$. More recent work on the Lewis acid behavior of $AsI_3$ includes the reaction with Lewis bases having N,[15–17] P,[16,18] and As[16,18] as well as O,[17,19,20] S,[21] and Se[21] donor atoms. In some instances,[18,19,21] the determination of the structure of the complexes by X-ray crystallography was possible.

Bis(4-acetamidophenyl) disulfide in the solid state must have intermolecular hydrogen bonding, forming transoid-transoid "polymer"[22] because strong (at 3294 $cm^{-1}$) and weaker (at 3171, 3108 $cm^{-1}$) bands for N—H stretching and very strong amide I (at 1662 $cm^{-1}$) and amide II (at 1540 $cm^{-1}$) bands are seen in its solid phase IR spectrum. The IR spectrum of the black **8** is quite similar to that of the disulfide except that the bands are at 3304, 1654, and 1534 $cm^{-1}$, respectively. These data indicate that the "polymeric" structure of the bis(4-acetamidophenyl) disulfide is retained in **8**, having a C=O···H—N hydrogen-bonding network, which is slightly perturbed by $AsI_3$. From the composition of **8** and its IR spectrum, the $AsI_3$ molecule seems to be bonded to the lone pairs of three nitrogen atoms. Then, each arsenic will have a $N_3I_3$ environment. However, the brown 1:1 complexes of $AsI_3$ with acetamide and with *N*, *N*-dimethylacetamide have, by IR, arsenic coordinated to carbonyl oxygen.[20]

The solid state IR spectrum of **9** is quite simple, does not resemble that of bis(4-aminophenyl) disulfide,[23] and does not contain decomposition products that were found with $AsI_3 \cdot 3PhNH_2$, which in ether gave $PhNH_2 \cdot HI$ and I-As(NHPh)$_2$.[15] While from the mass balance and percent analysis six acetone molecules must be present, in the IR spectrum we find a very weak carbonyl peak at 1704 cm$^{-1}$. This fact, coupled with the absence of —NH$_2$ vibrations at 3418, 3335, and 3212 cm$^{-1}$ of the disulfide[23] and the presence of two very strong but not too broad bands at 3444 and 2876 and a much weaker peak at 2575 cm$^{-1}$, indicates that the amino group of the disulfide has been added to the carbonyl group of acetone to give a geminal aminoalcohol (N-substituted hemiaminal), which did not dehydrate to a Schiff base most likely because the electron pair on nitrogen is not free to initiate the dehydration. Therefore, the $AsI_3$ must be coordinated to the nitrogen and, from the composition, each arsenic must have a $N_3I_3$ environment. The band, then, at 3444 cm$^{-1}$, must be due to intermolecularly hydrogen bonded —OH groups, and the band at 2876 cm$^{-1}$ must be due to a stretching vibration of an elongated and, thus, weaker N—H bond. The elongation is caused by the "donation" of the nitrogen electron pair to the arsenic.

The solid state IR spectrum of the black **10** differs from that of 2,2′-dithiobis(pyridine *N*-oxide).[23] Of the C=C vibrations at 1463 and 1422 cm$^{-1}$ of the free ligand, the one at 1422 cm$^{-1}$ was less intense in **10**. The N—O stretching vibrations at 1249 and 1230 cm$^{-1}$ of the free ligand were less intense in **10**, and a new vibration at 1182 cm$^{-1}$ appeared that is attributed to N—OAs stretching. The N—O bending vibration moved from 838 to 832 cm$^{-1}$ in **10**. The coordination of related ligands showed that, e.g., when pyridine *N*-oxide binds to platinum(II), the N—O stretching is shifted from 1242 to 1235 cm$^{-1}$.[24] When 2-mercaptopyridine *N*-oxide, which exists mainly as the tautomeric 1-hydroxypyrid-2-thione with $\beta_{CH}$ at 1263 cm$^{-1}$ and N—O stretching at 1112 and 1081 cm$^{-1}$,[25] is coordinated in a bidentate mode to tetraphenylantimony(V), the N—O stretching appears at 1197 cm$^{-1}$.[26] The stretching vibrations of the N—O of the free ligand, 2,2′-bipyridine *N*,*N*′-dioxide (BipyO$_2$), at 1262 and 1255 appeared at 1265 and 1210 cm$^{-1}$ in the reddish-brown complex $AsI_3 \cdot 2BipyO_2$, while the N—O bending vibrations of the free ligand at 852 and 842 cm$^{-1}$ appeared at 842 cm$^{-1}$ in the complex.[17] From the composition of **10** and its IR spectrum, a bidentate coordination of the ligand to give, for example, a 9-membered ring and an $O_3I_3As^{3-}$ core with a high charge on arsenic is not probable. We propose that 2,2′-dithiobis(pyridine *N*-oxide) most probably acts, through its —O$^-$, as a monodentate ligand, thus lowering the charge on the arsenic atom compared to the bidentate coordination, and, moreover, the positive charge on nitrogen, N$^+$—O—As$^-$, can be dissipated through the —S—S— bridge to the other pyridine *N*-oxide ring via their canonical structures.[27] Whether **10** is a single compound is an open question in the absence of crystallographic data.

Arsenic triodide is quite stable in water; in fact, it can be recovered unchanged from the solution[9] and has been used in medicine for the treatment of tuberculosis in pigs, of anaemia from hereditary syphilis, of arthritis and certain dermatitises, but it is toxic.[8a] Given the effectiveness of arsenic(III) oxide ($As_2O_3$, arsenox$^R$) in treating acute promyelocytic leukemia in man,[28] arsenic triiodide and its complexes, like **8–10**, should be examined in place of arsenic(III) oxide. Based on the behavior of $(ArS)_3As$ and $I_2$, the reaction of iodine with thioesters of arsonous and arsinous acids, L-As$(SPh)_2$ and $L_2$As-SPh, should give L-As$I_2$ and $L_2$As-I (L = R or Ar) and PhSSPh. From these iodides, a wide range of organoarsenic compounds can be prepared. Such studies are underway.

## EXPERIMENTAL

The aromatic trithioarsenites have been prepared by literature methods as follows: **1**,[10] **3**, **5**, and **6**,[2] **2**, **4**, and **7**.[5] Silica gel 60 H (Merck) was used for Thin Layer Chromatography (TLC). AR grade solvents have been dried over $A_4$ molecular sieves and de-aerated by boiling, flushing with nitrogen, stoppering, and cooling to r.t. TLC was run on microslides using the appropriate disulfides[2,5,10] as standards. The visualization was effected by iodine vapors followed by spraying with 35% sulfuric acid and charring. Compounds containing an ArS— group initially gave colored spots and on charring left a faint black spot. For melting points, an Electrothermal (model 9100) apparatus was used, and IR spectra were obtained on a Perkin-Elmer (model 16PC FT-IR) spectrometer. Elemental analyses were obtained through the Centre of Instrumental Analyses, University of Patras, Patras, Greece.

## The Reaction of the Trithioarsenites 1–7 With Iodine

### *Triphenyl Trithioarsenite, 1*

To a solution of **1** (201 mg, 0.5 mmol) in ether (6 mL), iodine (191 mg, 0.75 mmol) was added and stirred at r.t. for 1 h. Centrifugation and washing with ether (2 × 2 mL) gave arsenic triiodide (204 mg, 90%) as an orange solid having m.p. 141°C (lit.[8a] 138.6, 140.7, 146°C). IR (KBr): 3431 s, broad, 1636 w, broad, 595 m, broad. Evaporation of the dark red supernatant gave a light orange solid (169 mg), which was diphenyl disulfide (by TLC and IR) contaminated by arsenic triiodide.

### *Tris(4-Chlorophenyl) Trithioarsenite, 2*

The reaction of **2** (101 mg, 0.2 mmol) with iodine (73 mg, 0.3 mmol) in ether (1 mL), as previously mentioned, gave arsenic triiodide (72 mg, 78%), m.p. 141–142°C.

### *Tris(4-Nitrophenyl) Trithioarsenite, 3*

To a suspension of **3** (0.25 mmol) in dichloromethane (3 mL), iodine (0.375 mmol) was added. At once, the ester **3** dissolved giving a dark brown solution from which an orange solid precipitated. Evaporation, drying in vacuum, addition of carbon disulfide (3 mL), centrifugation, and washing with carbon disulfide (1 × 2 mL) left the disulfide (99 mg, 86%), m.p. 183–184°C (lit.[29] 173–181°C; lit.[30] 191°C). Its IR (KBr) was the same as reported.[23] From the supernatant, arsenic triiodide (120 mg) contaminated by the disulfide (by TLC and IR) was obtained, m.p. 140°C.

### *Tris(4-Acetamidophenyl) Trithioarsenite, 4*

The ester **4** (0.147 mmol) and iodine (0.221 mol) in acetone (2 mL) after 1 h of stirring and evaporation gave a cherry-red gum. The addition of carbon disulfide (3 mL) and boiling transformed the gum to a black solid. Centrifugation and washing with carbon disulfide (2 × 2 mL) left **8** as a black powder (121 mg, 88%). It is soluble in acetone (yellow solution) and in methanol (orange solution), sparingly soluble in chloroform, and insoluble in ether and carbon disulfide. M.p. 148–149°C. Calculated for $C_{48}H_{48}N_6O_6S_6I_6As_2$ ($M_r$ 1908.58): C, 30.20; H, 2.53; N, 4.40; S, 10.08%. Found. C, 30.41; H, 2.48; N, 4.33; S, 10.55%. IR (KBr): 3303, s, sharp, 3112 m, 1654 vs, 1594 s, 1534 vs, 1490 ms, 1396 ms, 1320 m, 1117 w, 1078 w, 1045 w, 1006 m, 816 s, 672 mw, 604 mw.

### *Tris(4-Aminophenyl) Trithioarsenite, 5*

The addition of iodine (0.375 mmol) to a solution of **5** (0.25 mmol) in acetone (3 mL) immediately precipitated a brown-orange gum. After 3 h of stirring at r.t., evaporation and drying in a vacuum gave a sticky, colored foam. It was covered with ether and stirred at r.t. overnight. Centrifugation and washing with ether (1 × 2 mL) gave **9** as a light yellow-brown solid (246 mg, 98%) insoluble in carbon disulfide and ether, sparingly soluble in chloroform (yellow), and soluble in methanol (yellow). M.p.: 73°C shrinks, 92°C turns red, 99–101°C melts to a red oil. Calculated for $C_{54}H_{72}N_6O_6S_6I_6As_2$ ($M_r$ 2004.79): C, 32.35; H, 3.62; N, 4.19; S, 9.60%. Found: C, 31.97; H, 3.15; N, 4.69; S, 9.55%. IR (KBr): 3444 vs, slightly broad, 2876 vs, slightly broad, 2575 m, 1704 vw, 1618 s, 1565 m, 1488 vs, 1406 w, 1374 w, 1313 w, 1206 w, 1122 w, 1017 w, 808 s, 612 w, 495 w.

### *Tris(2-Pyridyl) Trithioarsenite, 6*

The reaction of **6** (0.25 mmol) suspended in dichloromethane (3 mL) with iodine (0.375 mmol) gave a cherry-red oil that could not be induced to crystallize and was not studied further.

### *Tris(2-pyridyl N-Oxide) Trithioarsenite, 7*

To a suspension of **7** (0.82 mmol) in acetone (5 mL), iodine (1.23 mol) was added. At once, the ester dissolved and a new solid formed. After 2 h of stirring, evaporation and drying in vacuum gave a black crystalline solid. The addition of carbon disulfide (3 mL), centrifugation, and washing with carbon disulfide (1 × 2 mL) gave **10** as a black-brown powder (677 mg, 99%). It is soluble in methanol (yellow) and acetone (orange), sparingly soluble in chloroform, and insoluble in ether. M.p. 105–108°C. Calculated for $C_{30}H_{24}N_6O_6S_6I_6As_2$ ($M_r$ 1668.21): C, 21.69; H, 1.45; N, 5.04; S, 11.53%; Found: C, 21.48; H, 1.02; N, 5.36; S, 11.52%. IR (KBr): 3444 m, slightly broad, 3090 m, 1590 w, 1554 m, 1460 vs, 1418 s, 1248 m, 1230 sh, 1182 m, 1142 m, 1084 w, 1039 w, 828 m, 758 s, 704 m, 578 s.

## The Reaction of Triphenyl Trithioarsenite, 1, With Bromine

The reaction of **1** (0.315 mmol) and bromine (0.473 mmol) in ether (6 mL) gave a yellow solution from which the arsenic tribromide and diphenyl disulfide could not be separated due to similar solubilities.[8a] Hydrolysis of the mixture in acetone gave $As_2O_3$ (16 mg, 50% recovery) and impure diphenyl disulfide (114 mg), which was recrystallized from petroleum ether, m.p. 59–60°C (lit.[31] 61–62°C).

## The Reaction of 1 With Phenylsulfenyl Bromide

To a solution of bromine (103 mg, 0.625 mmol) in dichloromethane, diphenyl disulfide (136 mg, 0.625 mmol) was added, and the red solution was left at r.t. for 2 h. TLC (petroleum ether) showed the presence of disulfide ($R_f$ 0.46) and a spot as $R_f$ 0.0 (due to the hydrolysis of PhSBr on the plate). The evaporation left an oil (202 mg, expected 236 mg PhSBr). To the oil in dichloromethane (3 mL), solid **1** (0.35 mmol) was added. At once, the solution decolorized to a nearly colorless solution. TLC (petroleum ether) showed only the presence of disulfide and the absence of **1** ($R_f$ 0.10).

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