

Two-step synthesis of triarylmets (As, Sb, Bi) starting from the metal oxides and 2,6-dimethoxybenzenethiol

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Abstract

Reported here is a new synthetic method of triarylmets MAr_3 ($\text{M} = \text{As, Sb, Bi}$). It is composed of two-step reactions starting from the metal oxides: (1) A reaction of the metal oxide with 2,6-dimethoxybenzenethiol ΦSH [$\Phi = 2,6\text{-(MeO)}_2\text{C}_6\text{H}_3$] in the presence of acid to give the thiolatometals $\text{M(S}\Phi)_3$, and (2) A reaction of $\text{M(S}\Phi)_3$ with organolithium reagent LiAr to give MAr_3 ($\text{Ar} = \text{Ph, 4-MeC}_6\text{H}_4, 4\text{-Me}_2\text{NC}_6\text{H}_4, \Phi$). Since ΦSH is odorless and crystalline, most of it can be recovered after the reactions without difficulty for repeated usages. The intermediates $\text{M(S}\Phi)_3$ are also crystalline and inert against hydrolysis. © 1997 Elsevier Science S.A.

Keywords: Arsenic; Antimony; Bismuth; 2,6-Dimethoxybenzenethiol; Thiolatometals; Aryllithium

1. Introduction

A variety of organometallic compounds of group 15 elements have been synthesized by reactions of organolithium reagents or Grignard reagents with the metal(III) chlorides. Unfortunately, these metal(III) chlorides are generally intractable materials due to the hygroscopic character. In addition, the reactions are often highly exothermic, and a mild and less hygroscopic starting reagent is desirable for successful experimentals. It has been reported that Grignard reagents react with these metal(III) oxides to give the triorganometals, but the reaction products are often contaminated with di- and monoorganometals [1–4]. Stamm and Breindel treated trialkylaluminums with these metal(III) oxides; the reactions were successful for arsine(III) oxide and stibine(III) oxide when excess trialkylaluminum was employed [5]. However, no application to triarylmets synthesis has been reported. Srivastava, Krannich, and Watkins reviewed with comments and criticisms upon a variety of synthetic methods for triorganoarsane, and they found a chlorodioxarsolane AsCl(OR)_2 to be a versatile compound as the starting material [6].

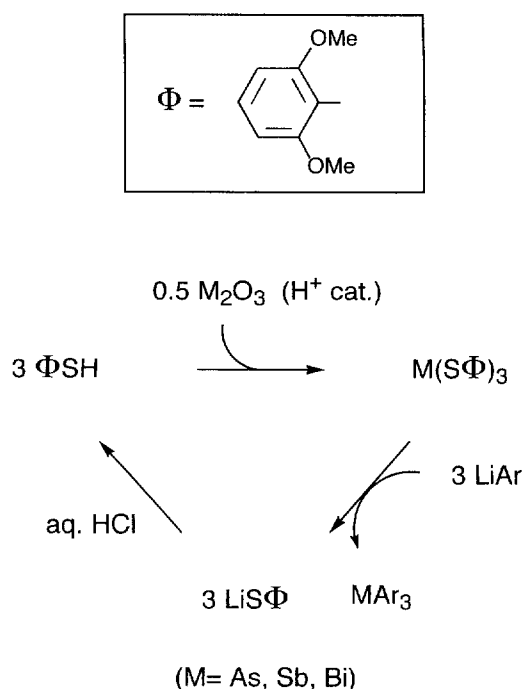
During the course of our works on 2,6-dimethoxyphenyl derivatives of typical elements, we realized that 2,6-dimethoxybenzenethiol, ΦSH [$\Phi = 2,6\text{-(MeO)}_2\text{C}_6\text{H}_3$], was quite a useful reagent due to its odorless and crystalline properties [7–9]. We present here a new synthetic method of triarylmets of group 15 elements (As, Sb, Bi) using the thiolatometals $\text{M(S}\Phi)_3$ (Scheme 1). These compounds can be obtained without difficulty by reaction of metal(III) oxide with ΦSH . They are crystalline, inert against hydrolysis, and soluble in some common organic solvents. In addition, most of ΦSH can be recovered for repeated usages after the reaction with aryllithium.

2. Experimental details

2.1. General

^1H NMR spectra were recorded for solutions in CDCl_3 using a JEOL model JNM-GX-270 spectrometer operating at 270 MHz. IR spectra were recorded for Nujol mull using a Shimadzu FTIR-4200 spectrophotometer. GC-MS spectra were recorded using a Shimadzu QP-5000 mass spectrometer.

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Scheme 1.

2.2. Preparation of 2,6-dimethoxybenzenethiol, Φ SH [Φ = 2,6-(MeO)₂C₆H₃]

To a mixture of 1,3-dimethoxybenzene (110 ml, 800 mmol) and 15% solution of butyllithium in hexane (500 ml, 800 mmol) was added at 0°C a catalytic amount of *N,N,N',N'*-tetramethylethylenediamine (1 ml). The mixture was stirred at room temperature for 20 min to give a white suspension, to which was added with stirring a solution of elemental sulfur (23.1 g, 720 mmol) dissolved in toluene (600 ml). The mixture was kept stirring at room temperature overnight, and then it was treated with water (1000 ml). The water layer was acidified with 1 M hydrochloric acid (800 ml) to give white precipitates of Φ SH in 86% yield after recrystallization from methanol; mp 80–81°C (reported 82–83°C [7]); IR (Nujol) 2570 cm⁻¹ ν (S–H); ¹H NMR (CDCl₃) δ = 7.03 (1H, t, J_H = 8 Hz, 4-H), 6.56 (2H, d, J_H = 8 Hz, 3,5-H), 4.03 (1H, s, SH), and 3.89 (6H, s, MeO); MS *m/z* (rel. intensity) 170 (100, M⁺).

2.3. Preparation of tris(2,6-dimethoxybenzenethiolato) metals

2.3.1. As(S Φ)₃

To a mixture of arsine(III) oxide (1.19 g, 6 mmol) and Φ SH (5.10 g, 30 mmol) in acetone (100 ml) was added aqueous 1 M nitric acid (30 ml). The mixture was heated at 50°C for 1 h to give a white suspension. Acetone (200 ml) was added, and the mixture was heated to dissolve most of the precipitates. It was filtered while hot, water (100 ml) was added to give a

light suspension, and it was cooled to –30°C to give colorless crystals of As(S Φ)₃ in 93% yield; mp 121–122°C. The analytical sample was obtained by recrystallization from acetone; mp 125°C; ¹H NMR (CDCl₃) δ = 7.12 (3H, t, J_H = 8 Hz, 4-H), 6.47 (6H, d, J_H = 8 Hz, 3,5-H), and 3.71 (18H, s, MeO). Found: C, 49.78; H, 4.74%. Calcd. for C₂₄H₂₇AsO₆S₃: C, 49.48; H, 4.67%.

This compound is also obtained by the reaction of arsine(III) chloride (0.5 mmol), Φ SH (1.5 mmol), and triethylamine (1.6 mmol) in ethanol (15 ml) in 74% yield after 0.5 h at room temperature.

The compound is soluble in acetonitrile, chloroform, dimethyl sulfoxide, nitromethane, tetrahydrofuran, hot acetone (recrystallizable), hot ethyl methyl ketone, and hot toluene (recrystallizable), but it is insoluble in diethyl ether, methanol, hexane, 2-propanol, and water.

2.3.2. Sb(S Φ)₃

To a mixture of stibine(III) oxide (1.46 g, 5 mmol) and Φ SH (33 mmol) in ethanol (100 ml) was added 6 M nitric acid (5.5 ml). The mixture was stirred at room temperature for 5 h to give a yellow suspension. The resultant precipitates were recrystallized from acetone to give light yellow crystals of Sb(S Φ)₃ in 63% yield; mp 154–155°C; ¹H NMR (CDCl₃) δ = 7.06 (3H, t, J_H = 8 Hz, 4-H), 6.49 (6H, d, J_H = 8 Hz, 3,5-H), and 3.76 (18H, s, MeO). Found: C, 46.05; H, 4.21%. Calcd. for C₂₄H₂₇O₆S₃Sb: C, 45.80; H, 4.32%.

This compound is also obtained by the reaction of stibine(III) chloride (0.5 mmol), Φ SH (1.5 mmol), and triethylamine (1.6 mmol) in methanol (20 ml) in 84% yield after 0.5 h at room temperature.

The compound is soluble in chloroform, dimethyl sulfoxide, nitromethane, hot acetone (recrystallizable), hot acetonitrile, hot ethyl methyl ketone, hot tetrahydrofuran, and hot toluene (recrystallizable), but it is insoluble in diethyl ether, methanol, hexane, 2-propanol, and water.

2.3.3. Bi(S Φ)₃

To a mixture of bismuth(III) oxide (2.80 g, 6 mmol) and Φ SH (30 mmol) in acetone (100 ml) was added 1 M nitric acid (3 ml). The mixture was stirred at room temperature for 1 h to give a yellow suspension. The precipitates were recrystallized from acetone–water (9:1 ~ 8:2) to give orange crystals of Bi(S Φ)₃ in 69% yield; mp 166–168°C; ¹H NMR (CDCl₃) δ = 6.97 (3H, t, J_H = 8 Hz, 4-H), 6.50 (6H, d, J_H = 8 Hz, 3,5-H), and 3.79 (18H, s, MeO). Found: C, 40.53; H, 3.77%. Calcd. for C₂₄H₂₇BiO₆S₃: C, 40.23; H, 3.80%.

This compound is also obtained by the reaction of bismuth(III) chloride (0.5 mmol), Φ SH (1.5 mmol), and triethylamine (1.6 mmol) in methanol (10 ml) in 90% yield after 0.5 h at room temperature.

The compound is soluble in dimethyl sulfoxide, hot acetone (recrystallizable), hot acetonitrile (recrystalliz-

able), hot chloroform, hot ethyl methyl ketone, hot nitromethane (recrystallizable), hot tetrahydrofuran (recrystallizable), but it is insoluble in diethyl ether, hexane, methanol, 2-propanol, toluene, and water.

2.4. Reactions of thiolatometals with aryllithium

2.4.1. $M(S\phi)_3$ with $LiPh$

A solution of phenyllithium was prepared from bromobenzene (3.6 ml, 36 mmol) and a 15% solution of butyllithium in hexane (21 ml, 33 mmol) in dry diethyl ether (100 ml) stirring at room temperature for 2 h under argon. Crystals of $As(S\phi)_3$ (5.82 g, 10 mmol) was added, and the mixture was stirred at room temperature for 6 h to give a white suspension. The excess phenyllithium was quenched with methanol (5 ml), and the mixture was filtered to give the solid A and the filtrate. The solid A was washed with methanol (5 ml), and the methanol was added to the filtrate. The filtrate was concentrated to ca. one fifth under reduced pressure to give colorless crystals of $AsPh_3$ (1.57 g, 5.1 mmol, 51% yield); mp 60°C (reported 60–61°C [6]); 1H NMR ($CDCl_3$) δ = 7.32 (5H, s, Ph); MS m/z (rel. intensity) 306 (40, M^+), 152 (100, $AsPh^+$). The solid A was extracted with water (300 ml) to leave unreacted $As(S\phi)_3$ (0.160 g, 0.27 mmol, 3% recovery). The aqueous layer was acidified with 1 M hydrochloric acid (33 ml) to give crystals of ΦSH (22 mmol, 74% yield).

In essentially an analogous manner as above, a treatment of phenyllithium solution with $Sb(S\phi)_3$ at room temperature for 16 h resulted to give colorless crystals of $SbPh_3$ in 57% yield; mp 51–53°C (reported, 50°C [10]); 1H NMR ($CDCl_3$) δ = 7.45–7.42 (2H, m, 2,6-H) and 7.33–7.31 (3H, m, 3,4,5-H); MS m/z (rel. intensity) 354 (6, M^+), 196 (100, $SbPh^+$). From the aqueous layer was obtained ΦSH in 90% yield.

In essentially an analogous manner as above, a treatment of phenyllithium solution with $Bi(S\phi)_3$ at room temperature overnight resulted to give colorless crystals of $BiPh_3$ in 71% yield; mp 77–78°C (reported 78°C [6]); 1H NMR ($CDCl_3$) δ = 7.74 (2H, dd, J_H = 8 and 2 Hz, 2,6-H) and 7.41–7.31 (3H, m, 3,4,5-H); MS m/z (rel. intensity) 286 (57, $BiPh^+$), 209 (100, Bi^+). From the aqueous layer was obtained ΦSH in 77% yield.

2.4.2. $M(S\phi)_3$ with $Li(4-MeC_6H_4)$

A solution of 4-tolylithium was prepared as mentioned above from 4-bromotoluene (1.54 g, 9 mmol) and a 15% solution of butyllithium in hexane (5 ml, 8 mmol) in dry diethyl ether (20 ml). It was treated with $Sb(S\phi)_3$ (2 mmol) at room temperature for 6 h as above to give colorless crystals of $Sb(4-MeC_6H_4)_3$ (0.381 g, 0.96 mmol, 48% yield); mp 120–123°C after recrystallization from acetone (reported 125–126°C [10]); 1H NMR($CDCl_3$) δ = 7.32 (6H, d, J_H = 8 Hz, 2,6-H), 7.12 (6H, d, J_H = 8 Hz, 3,5-H), and 2.33 (9H, s, Me); MS m/z (rel. intensity) 394 (16, M^+), 212

(100, $4-MeC_6H_4Sb^+$). From the aqueous filtrate was obtained ΦSH in 89% yield.

In essentially an analogous manner as above, a treatment of 4-tolylithium solution with $As(S\phi)_3$ resulted to give colorless crystals of $As(4-MeC_6H_4)_3$ in 60% yield; mp 145–146°C (reported 146 [6], 148–149°C [11]); 1H NMR ($CDCl_3$) δ = 7.22 and 7.12 (12H, A_2B_2 pattern, J_H = 8 Hz, 2,3,5,6-H), and 2.33 (9H, s, Me); MS m/z (rel. intensity) 348 (20, M^+), 166 (100, $4-MeC_6H_4As^+$). From the aqueous layer was obtained ΦSH in 97% yield.

In essentially an analogous manner as above, a treatment of 4-tolylithium solution with $Bi(S\phi)_3$ resulted to give colorless crystals of $Bi(4-MeC_6H_4)_3$ in 53% yield; mp 117–119°C (reported 119–120°C [12]); 1H NMR ($CDCl_3$) δ = 7.62 (6H, d, J_H = 8 Hz, 2,6-H), 7.18 (6H, d, J_H = 8 Hz, 3,5-H), and 2.31 (9H, s, Me); MS m/z (rel. intensity) 394 (16, M^+), 212 (100, $4-MeC_6H_4Bi^+$). From the aqueous layer was obtained ΦSH in 57% yield.

2.4.3. $M(S\phi)_3$ with $Li(4-Me_2NC_6H_4)$

A solution of 4-dimethylaminophenyllithium was prepared as mentioned above from 4-bromo-*N,N*-dimethylaniline (1.8 g, 9 mmol) and a 15% solution of butyllithium in hexane (5 ml, 8 mmol) in dry diethyl ether (10 ml). It was diluted with toluene (10 ml) and was treated with $Bi(S\phi)_3$ (2 mmol) at room temperature for 24 h as above to give a 9:1 mixture of $Bi(4-Me_2NC_6H_4)_3$ and $Bi(S\phi)_3$ (0.621 g). The filtrate was concentrated to ca. one fourth under reduced pressure, and it was cooled at $-30^\circ C$ to give yellow crystals of $Bi(4-Me_2NC_6H_4)_3$ (0.69 mmol); mp 212–213°C after recrystallization from acetone; 1H NMR ($CDCl_3$) δ = 7.58 (6H, d, J_H = 8 Hz, 2,6-H), 6.73 (6H, d, J_H = 8 Hz, 3,5-H), and 2.91 (18H, s, Me). From the aqueous layer was obtained ΦSH in 75% yield.

In essentially an analogous manner as above, a treatment of 4-dimethylaminophenyllithium with $As(S\phi)_3$ at room temperature for 6 h resulted to give colorless crystals of $As(4-Me_2NC_6H_4)_3$ in 73% yield; mp 220–224°C after recrystallization from acetone (reported 240–242°C [13]); 1H NMR ($CDCl_3$) δ = 7.21 (6H, d, J_H = 9 Hz, 2,6-H), 6.68 (6H, d, J_H = 9 Hz, 3,5-H), and 2.92 (18H, s, Me). From the aqueous layer was obtained ΦSH in 77% yield.

In essentially an analogous manner as above, a treatment of 4-dimethylaminophenyllithium with $Sb(S\phi)_3$ at room temperature for 12 h resulted to give colorless crystals of $Sb(4-Me_2NC_6H_4)_3$ in 59% yield; mp 205–210°C after recrystallization from acetone–methanol (9:1); 1H NMR ($CDCl_3$) δ = 7.31 (6H, d, J_H = 9 Hz, 2,6-H), 6.69 (6H, d, J_H = 9 Hz, 3,5-H), and 2.92 (18H, s, Me). Found: C, 59.56; H, 6.56; N, 8.50%. Calcd. for $C_{24}H_{30}N_3Sb$: C, 59.77; H, 6.27; N, 8.71%. From the aqueous layer was obtained ΦSH in 80% yield.

2.4.4. $\text{As}(\text{S}\Phi)_3$ with $\text{Li}\Phi$

A suspension of $\text{Li}\Phi$ was prepared as mentioned above using 1,3-dimethoxybenzene (1 ml, 8 mmol), 15% hexane solution of butyllithium (4.4 ml, 7 mmol), and N,N,N',N' -tetramethylethylenediamine (0.1 ml) in diethyl ether (20 ml). Crystals of $\text{As}(\text{S}\Phi)_3$ (2 mmol) was added at 0°C , and the mixture was stirred at room temperature for 24 h to give a yellow suspension. The excess $\text{Li}\Phi$ was quenched with methanol (1 ml), and the resultant solid was washed with water (50 ml) to leave crystals of $\text{As}\Phi_3$ (0.629 g, 65% yield) [14]. The organic filtrate was washed with water (10 ml). The aqueous layers were combined, and it was acidified with 6 M hydrochloric acid (5 ml) to give crystals of ΦSH (4.8 mmol, 80% yield).

2.4.5. $\text{As}(\text{S}\Phi)_3$ with MgBrPh

A solution of phenylmagnesium bromide was prepared from bromobenzene (0.9 ml, 9 mmol) and magnesium (0.194 g, 8 mmol) in dry tetrahydrofuran (15 ml) under argon. Crystals of $\text{As}(\text{S}\Phi)_3$ (2 mmol) was added at 0°C , and the mixture was stirred at room temperature for 7 h. Hexane (30 ml) was added, and the mixture was treated with 1 M hydrochloric acid (10 ml) to give crystals of ΦSH (3.1 mmol, 52% yield). The filtrate was concentrated under reduced pressure to ca. one third and was kept at -30°C for 3 days to give crystals of AsPh_3 in 69% yield.

3. Results and discussion

3.1. Reactions of metal oxides with 2,6-dimethoxybenzenethiol

It is only recently found that arsine(III) oxide reacts with a variety of thiols to give the trithiolatoarsane [15]. We also found that arsine(III) oxide reacted quite easily with ΦSH in acetone in the presence of aqueous nitric acid to give $\text{As}(\text{S}\Phi)_3$. Stibine(III) oxide also reacted with ΦSH in ethanol to give $\text{Sb}(\text{S}\Phi)_3$. Bismuth(III) oxide was the most reactive, and it reacted in acetone in the presence of only a catalytic amount of aqueous nitric acid to give $\text{Bi}(\text{S}\Phi)_3$. No special care was necessary during the reactions, since these products are crystalline, absolutely odorless, and inert against hydrolysis under normal conditions. However, when aqueous hydrochloric acid was used in place of nitric acid, mixtures of $\text{M}(\text{S}\Phi)_3$ and possibly $\text{MCl}(\text{S}\Phi)_2$ were often obtained for $\text{M} = \text{Sb}$ and Bi . Naturally, these thiolatometals $\text{M}(\text{S}\Phi)_3$ could be obtained also by reactions of metal trihalide with ΦSH in alcohols in the presence of base such as triethylamine.

All of the thiolatometals $\text{M}(\text{S}\Phi)_3$ are highly crystalline with melting points higher than 120°C after recrystallization from aqueous acetone or alcohols. They are soluble in some organic solvents such as ni-

tromethane, acetonitrile, chloroform, acetone, and toluene, but the solubility decreases drastically in the order of $\text{M} = \text{As} > \text{Sb} > \text{Bi}$. Thus, while $\text{As}(\text{S}\Phi)_3$ is very soluble in nitromethane, acetonitrile, and chloroform at room temperature, $\text{Bi}(\text{S}\Phi)_3$ is soluble in these solvents only on heating. On prolonged heating the solutions in nitromethane or in acetonitrile, both $\text{Sb}(\text{S}\Phi)_3$ and $\text{Bi}(\text{S}\Phi)_3$ decomposed to give turbid suspensions.

It is worth noting here that tin(IV) oxide did not react with ΦSH under analogous conditions, although tin(II) oxide reacted easily to give $\text{SnCl}_2(\text{S}\Phi)_2$, $\text{SnCl}(\text{S}\Phi)_3$, and/or $\text{Sn}(\text{S}\Phi)_4$ depending on the conditions.

3.2. Reactions of tris(2,6-dimethoxybenzenethiolato)-metals with aryllithiums

The thiolatoarsane $\text{As}(\text{S}\Phi)_3$ reacted with a slight excess of phenyllithium in diethyl ether (10 mmol/100 ml scale) in 6 h at room temperature to give precipitates of $\text{LiS}\Phi$. From the filtrate on concentration, crystals of triphenylarsane were obtained in 51% yield. The precipitates of $\text{LiS}\Phi$ were often contaminated with a small amount of unreacted $\text{As}(\text{S}\Phi)_3$. Since $\text{LiS}\Phi$ is very soluble in water, unreacted $\text{As}(\text{S}\Phi)_3$ can be separated easily by extraction with water to leave $\text{As}(\text{S}\Phi)_3$ (for an example, in 3% recovery). On acidification of the aqueous layer, crystals of ΦSH were obtained in 74% yield.

The reaction of $\text{Sb}(\text{S}\Phi)_3$ with phenyllithium seems to be slower, but they reacted in 16 h to give triphenylstibane, ΦSH , and $\text{Sb}(\text{S}\Phi)_3$ in 57, 90, and 10% yields, respectively. The reaction of $\text{Bi}(\text{S}\Phi)_3$ with phenyllithium is much slower, and it is necessary to use a less amount of solvent and to extend the reaction period. Under such conditions, triphenylbismuthane and ΦSH were obtained in 71 and 77% yields, respectively, and $\text{Bi}(\text{S}\Phi)_3$ was recovered in 18% yield.

In analogous manners, $\text{M}(\text{S}\Phi)_3$ ($\text{M} = \text{As}, \text{Sb}, \text{Bi}$) reacted with 4-tolylolithium, 4-dimethylaminophenyllithium, and 2,6-dimethoxyphenyllithium to give the corresponding triarylmetals and ΦSH both in moderate yields. For example, reactions of 4-tolylolithium resulted to give tri-4-tolylmetals in 60 (As), 48 (Sb), and 53 (Bi)% yields, and crystals of ΦSH were obtained in 97, 89, and 57% yields, respectively. Since the solubility of the product also differs each other, we had difficulties in finding the best condition for the separation of these products. One of the origins of poorer yield of tri-4-tolylmetal than that of ΦSH must be due to the high solubility. Reactions of 4-dimethylaminophenyllithium with $\text{M}(\text{S}\Phi)_3$ often gave precipitates of a mixture containing the triarylmetal, $\text{LiS}\Phi$, and a small amount of unreacted $\text{M}(\text{S}\Phi)_3$. After separation of $\text{LiS}\Phi$ by extraction of precipitates with water, the triarylmetal could be separated from $\text{M}(\text{S}\Phi)_3$ by extraction with acetone; $\text{M}(\text{S}\Phi)_3$ are poorly soluble in this solvent. Additional crystals of triarylmetal, of course, were obtained from

the ethereal layer. The combined yields of tris(4-dimethylaminophenyl)metals were 73 (As), 59 (Sb), and 79 (Bi)%, and crystals of Φ SH were obtained in 77, 81, and 75% yields, respectively. Using 2,6-dimethoxyphenyllithium, tris(2,6-dimethoxyphenyl)arsane ($\text{As}\Phi_3$) was obtained in 65% yield, and Φ SH was obtained in 80% yield.

The use of an acid should be avoided to neutralize the reaction mixture of $\text{Bi}(\text{S}\Phi)_3$ and aryllithium, since the produced triarylbiuthane is generally labile toward acid.

Phenylmagnesium bromide also reacted with $\text{As}(\text{S}\Phi)_3$ in tetrahydrofuran to give triphenylarsane and Φ SH in 69 and 52% yields, respectively. In diethyl ether, the reaction was too slow probably due to the poor solubility of $\text{As}(\text{S}\Phi)_3$.

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References

- [1] K. Matsumiya, M. Nakai, Mem. Coll. Sci. Kyoto Imp. Univ. 8A (1925) 309.
- [2] K. Matsumiya, M. Nakai, Chem. Abstr. 19 (1925) 3086.
- [3] E. Gryszkiewicz-Trochimowski, Roczniki Chim. 8 (1928) 250.
- [4] E. Gryszkiewicz-Trochimowski, Chem. Abstr. 22 (1928) 4523.
- [5] W. Stamm, A. Breindel, Angew. Chem. Int. Ed. Engl. 3 (1964) 66.
- [6] D.K. Srivastava, L.K. Krannich, C.L. Watkins, Inorg. Chem. 29 (1990) 3502.
- [7] M. Trost, L.N. Jungheim, J. Am. Chem. Soc. 102 (1980) 7910.
- [8] P. Jacob III, A.T. Shulgin, Synth. Commun. 11 (1981) 957.
- [9] M. Wada, K. Tenma, K. Kajihara, K. Hirata, T. Erabi, Chem. Express 4 (1989) 109.
- [10] G.S. Hiers, Org. Syn. Coll. I (1941) 550.
- [11] B.A. McCortney, B.M. Jacobson, M. Vreeke, E.S. Lewis, J. Am. Chem. Soc. 112 (1990) 3554.
- [12] D.H.R. Barton, N. Ozbalik, M. Ramesh, Tetrahedron 44 (1988) 5661.
- [13] G. Tomaschewski, J. Prakt. Chem. 33 (1966) 168.
- [14] M. Wada, S. Miyake, S. Hayashi, H. Ohba, S. Nobuki, S. Hayase, T. Erabi, J. Organometal. Chem. 507 (1996) 53.
- [15] S.V. Serves, Y.C. Charalambidis, D.N. Sotiropoulos, P.V. Ioannou, Phosphorus Sulfur Silicon 105 (1995) 109.