A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

The procedures in *Organic Syntheses* are intended for use only by persons with proper training in experimental organic chemistry. All hazardous materials should be handled using the standard procedures for work with chemicals described in references such as "Prudent Practices in the Laboratory" (The National Academies Press, Washington, D.C., 2011; the full text can be accessed free of charge at http://www.nap.edu/catalog.php?record_id=12654). All chemical waste should be disposed of in accordance with local regulations. For general guidelines for the management of chemical waste, see Chapter 8 of Prudent Practices.

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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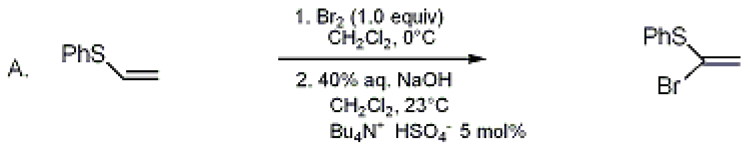
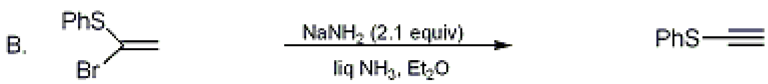
Organic Syntheses, Coll. Vol. 9, p.656 (1998); Vol. 72, p.252 (1995).

PHENYLTHIOACETYLENE

[Benzene, (ethynylthio)-]





Submitted by Plato A. Magriotis and John T. Brown[1].
Checked by Armin Walser, Carl Mason, and David L. Coffen.

1. Procedure

CAUTION! These operations involve reagents and solvents with potentially harmful vapors (Br_2, NH_3) and therefore should be conducted in an efficient hood. The use of disposable gloves is highly recommended.

A. *1-Bromovinyl phenyl sulfide*. A 1-L, round-bottomed flask is equipped with a 1.5-in egg-shaped magnetic stirring bar and a 100-mL, pressure-equalizing, addition funnel fitted with a Claisen adapter that contains a drying tube and stopper. The flask is charged with 40.8 g (0.30 mol) of phenyl vinyl sulfide (Note 1) and 250 mL of dichloromethane (Note 2). The resulting solution is cooled to 0°C and 49.6 g (0.31 mol) of bromine (Note 3) is added dropwise over approximately 1 hr through the funnel, until a bright red color persists. The intermediate product, phenylthio-1,2-dibromoethane (Note 4), is then treated at 0°C with 250 mL of aqueous 40% sodium hydroxide followed by 5.1 g (15 mmol, 5 mol%) of the phase-transfer catalyst tetrabutylammonium hydrogen sulfate. Vigorous stirring is continued at ambient temperature for 2–3 hr until TLC (10% benzene, 90% hexane) indicates that dehydrobromination is complete (Note 5). The organic layer is separated and the aqueous layer is extracted with two 200-mL portions of dichloromethane. The combined organic extracts are washed with a saturated solution of sodium bisulfite (250 mL), water (250 mL), and brine (250 mL) and dried over sodium sulfate. Excess dichloromethane is removed under reduced pressure on a rotary evaporator and the resulting dark brown oil is distilled under vacuum (1.5 mm) to provide 51.5–58.0 g (80–90%) of pure 1-bromovinyl phenyl sulfide as a pale yellow liquid, bp 76–78°C (Note 6) and (Note 7).

B. *Phenylthioacetylene*. An oven-dried, 2-L, three-necked, round-bottomed flask is equipped with a mechanical stirrer (Note 8), an acetone-dry ice condenser with a drying tube containing potassium hydroxide pellets, and a gas inlet. The flask is placed in an acetone-dry ice bath (−40°C, bath temperature) and 450 mL of anhydrous ammonia (Note 9) is condensed into the flask. Upon addition of a small piece of sodium metal (ca. 0.6 g) to the liquid ammonia the characteristic deep blue color develops. A catalytic amount of anhydrous ferric chloride (0.25 g, 1.5 mmol; 0.3 mol%) is added with continued stirring (Note 10) and the color of the reaction mixture turns gray. The remaining sodium metal (10.0 g, 0.46 g-atom total) is added in 0.6-g pieces over ca. 1 hr, since the blue color must be discharged before each new addition of sodium. A gray suspension of sodium amide is obtained upon completion of this addition. The temperature of the cooling bath is adjusted to −50°C and the gas inlet is replaced with a 250-mL, pressure-equalizing addition funnel containing 49.5 g (0.23 mol) of 1-bromovinyl phenyl sulfide in 100 mL of anhydrous ether (Note 11). This solution is added dropwise to the freshly generated sodium amide over 20 min, while the temperature of the acetone-dry ice bath is maintained at −50°C. Stirring is continued (Note 8) at this temperature for 0.5 hr, the brown-red reaction mixture is allowed to warm to reflux temperature (−33°C) during 1 hr, and then is recooled to −60°C (bath temperature). Solid ammonium chloride is added slowly (Note 12) to quench the sodium phenylthioacetylide, the cooling bath is removed, and the ammonia is allowed to evaporate. During evaporation, 400 mL of anhydrous ether (Note 13) is added dropwise through the addition funnel to replace ammonia. The resulting mixture is filtered at ambient temperature and reduced pressure through a coarse, Celite-packed, fritted-glass filter to remove the inorganic salts that are subsequently washed three times with 50 mL of anhydrous ether. The combined ethereal filtrate and washes are concentrated on a rotary evaporator and the dark brown residue (Note 14) is transferred to a 100-mL, round-bottomed flask fitted with a short-path distillation head. Pure product (Note 15) is distilled at 1.5 mm pressure (bp 48–50°C, (Note 16)) into an ice-cooled receiver. In this way, 21.6–24.7 g (70–80% yield) of phenylthioacetylene (Note 17) is obtained as a pale yellow liquid, which turns brown-red upon storage at −10°C (freezer) within a few hours (Note 18). Phenylthioacetylene stored under these conditions is stable for several months.

2. Notes

1. Phenyl vinyl sulfide is prepared from thiophenol and 1,2-dibromoethane according to the procedure described by Paquette and Carr; see: *Org. Synth., Coll. Vol. VII* **1990**, 453. An alternative preparation may be found on p. 662. An earlier, three-step synthesis employing 2-chloroethanol instead of 1,2-dibromoethane is available.[2]
2. Dichloromethane (A.C.S. certified) was obtained from Fisher Scientific Company and used as received.
3. Bromine (A.C.S. certified) was purchased from Aldrich Chemical Company Inc., used as received, and measured with a 50-mL graduated cylinder in the hood.
4. At this point crude phenylthio-1,2-dibromoethane can be isolated by separation of the organic phase, extraction of the aqueous layer with dichloromethane, washing of the combined extracts with saturated sodium bisulfite solution, drying ($MgSO_4$), and concentration (95% crude yield): ^1H NMR (270 MHz, $CDCl_3$) δ: 3.75 (dd, 1 H, J = 11.0, 8.6), 3.94 (dd, 1 H, J = 11.0, 5.5), 5.39 (dd, 1 H, J = 8.6, 5.5), 7.20–7.70 (m, 5 H). This dibromide is relatively unstable giving rise to a streak on silica gel TLC (10% benzene, 90% hexanes; R_f of the streak front is ca. 0.35, anisaldehyde detection). It has been reported that a 2:1–3:1 ratio of cis- and trans-2-bromovinyl phenyl sulfide is obtained upon distillation of the above dibromide.[2]
5. Neither a streak nor any significant by-product is detected (UV and anisaldehyde) by TLC analysis (Merck 0.25-mm thickness silica gel plates with 254 nm UV indicator).
6. Assay of this material by GC/MS (HP 5970 Mass Selective Detector equipped with a 50-m HP-1 capillary column) shows it to be ca. 96% pure (R_t = 3.6 min; 80–280°C, 20°C/min). The spectral and analytical properties are as follows: ^1H NMR (270 MHz, $CDCl_3$) δ: 5.83 (d, 1 H, J = 2.2), 5.93 (d, 1 H, J = 2.2), 7.35–7.55 (m, 5 H); MS m/e (relative intensity) 216 (M^+, 17), 214 (16), 135 (100), 109 (15). Anal. Calcd for C_8H_7BrS: C, 44.67; H, 3.28; S, 14.91. Found: C, 44.30; H, 3.46; S, 15.23.
7. The literature boiling point is reported as 70–73°C (2 mm). 1,8-Diazabicyclo[5.4.0]undec-7-ene (DBU) in ether has been employed to effect this elimination.[3] However, the procedure reported here is more amenable to large scale preparation because of its lower cost.
8. The stirring blade should be glass because sodium in ammonia solution attacks Teflon.
9. Commercial anhydrous ammonia is employed without further drying.
10. Stirring is maintained at a rate such that splattering of the reaction mixture on the upper parts of the flask's side-wall is minimized (see also ref 4, Chapter I, pp 1–4).
11. Ether is distilled from sodium-benzophenone ketyl under argon just prior to use.
12. A total of 35 g of solid ammonium chloride is added in portions of ca. 1 g with a spatula.
13. Anhydrous ether (A.C.S. certified) was obtained from Fisher Scientific Company and used as received.
14. TLC analysis (10% benzene, 90% hexanes) of the crude reaction mixture (95% yield) indicates complete conversion of vinyl bromide (R_f = 0.5) to phenylthioacetylene (R_f = 0.6). A minor product, which can be purified by flash column chromatography and identified as cis-1,2-bis(phenylthio) ethylene,[4 5 6 7 8 9] is also detected (R_f = 0.3) in variable amounts (5–15% yield) depending on the run.
15. Assay of this material by GC/MS shows it to be >98% pure (R_t = 3.0 min; 80–280°C, 15°C/min).

16. Four literature boiling points are reported: 78–79°C (7 mm),[5] 86–88°C (14 mm),[2] 61–62°C (5 mm),[2] and 48°C (0.8 mm).[10]
17. Spectral and analytical properties for phenylthioacetylene are as follows: IR (neat) cm^{-1}: 3285, 2040, 1585; ^{1}H NMR (270 MHz, $CDCl_3$) δ: 3.26 (s, 1 H), 7.20–7.50 (m, 5 H), ; ^{13}C NMR (67.8 MHz, $CDCl_3$) δ: 70.0, 87.0, 126.5, 126.7, 129.2, 131.4; MS m/e (relative intensity) 134 (M^+, 100), 90 (24), 89 (26), 51 (44). Anal. Calcd for C_8H_6S: C, 71.60; H, 4.51; S, 23.89. Found: C, 71.77; H, 4.63; S, 24.31.
18. It has been reported that methyl phenyl sulfide acts as a stabilizer; phenylthioacetylene did not tend to darken in its presence.[5]

Waste Disposal Information

All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

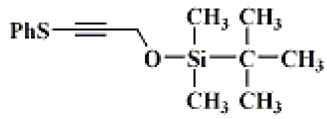
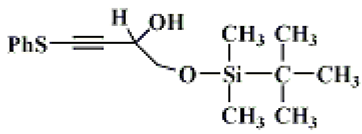
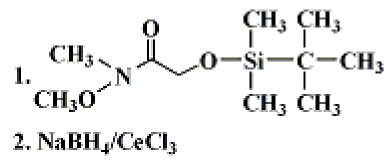
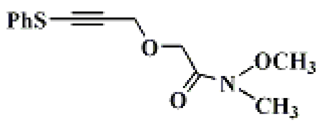
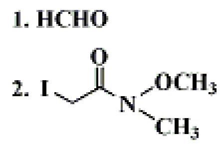
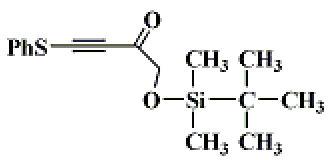
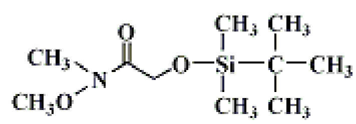
Phenylthioacetylene has been prepared by elimination of thiophenol and dehydrobromination of cis-1,2-bis(phenylthio)ethylene[4,5,6,7,8,9] and cis-1-bromo-2-phenylthioethylene,[2,11] [12] respectively. The latter was obtained by addition of thiophenol to propiolic acid in ethanol and subsequent one-pot bromine addition, decarboxylative dehalogenation, and careful distillation to remove the trans isomer.[2,11,12] On the other hand, cis-1,2-bis(phenylthio)ethylene was prepared by double addition of thiophenol to cis-1,2-dichloroethylene.[4,5,6,7] Although these procedures can provide useful amounts of phenylthioacetylene, they were found to be somewhat less satisfactory in our hands as far as operation and/or overall yields are concerned. Furthermore, we have encountered problems with regard to the reproducibility of one-pot dehydrobrominations of phenylthio-1,2-dibromoethane.[10] [13] [14] [15] However, the stepwise execution of the double dehydrobromination, as described in the modified procedure reported here, provides preparatively useful quantities of phenylthioacetylene in a practical manner.

A reported procedure based on lithium diisopropylamide induced double elimination of ethanol from bromoacetaldehyde diethyl acetal also was not very effective for the large scale preparation of phenylthioacetylene.[16] Another more recent synthesis of the title compound relies on the reaction of dimethyl(chloroethynyl)carbinol with an alkali metal phenylthiolate, followed by elimination of acetone under basic conditions.[17] Finally, synthesis of phenylthioacetylene has been achieved by the reaction of lithium trimethylsilyl acetylide with either phenylsulfinyl chloride[18] or the relatively expensive (>$10/1 g, Fluka Chemical Corp.) phenyl benzenethiosulfonate[15,19] and subsequent desilylation.

A large variety of phenylthio-substituted alkynes can be conveniently prepared from phenylthioacetylene as the nucleophilic component (Table I).[20] [21] [22] [23] This type of construction is more flexible than the one based on nucleophilic substitution of a terminal alkali metal acetylide on phenyl benzenethiosulfonate, phenyl sulfinyl chloride, and/or diphenyl disulfide.[24,25] Regio- and stereoselective syntheses of functionalized vinyl sulfides[26] [27] [28] [29] [30] are accomplished by Pd(0)-catalyzed hydrostannation,[22] hydroboration,[31] treatment with low-valent tantalum,[32] and stannylcupration[15,33] of 1-phenylthio-1-alkynes. In turn, vinyl sulfides are very useful intermediates in organic synthesis not only as carbonyl-masking moieties,[34] [35] [36] [37] [38] but also in a variety of other transformations,[39] [40] [41] [42] [43] including the Ni(0)-catalyzed cross-coupling reactions with alkyl, aryl, and alkenyl Grignard reagents.[44] [45] [46] [47] The important role of the phenylthio group and its higher oxidation states in activating and directing olefins in cycloaddition reactions[48] [49] [50] has been reviewed.[48]

TABLE
PREPARATION OF PHENYLTHIO ALKYNES FROM PHENYTHIOACETYLENE1

Entry	Phenylthioalkyne[a]	Electrophile[b]	% Yield[c]
1.		1. HCHO	82

		2. t-BuMe$_2$SiCl	
2.		HCHO	86
3.		MeI	89
4.		 2. NaBH$_4$/CeCl$_3$	70
5.		1. HCHO 	78
6.		ClCO$_2$Et	85
7.			75
8.		PhI	73
9.		BuI	80
10.		Me$_3$SiCl	90

[a]All Phenylthio alkyne products exibited spectral properties (^{1}H NMR, IR, and GC/MS) in accord with the assigned structures. [b]The electrophile employed in entries 4 and 7 was prepared from glycolic acid by reaction of its bis(*tert*-butyldimethylsilyl) derivative with oxalyl chloride followed by N,O-dimethylhyroxylamine hydrochloride.[51] Entry 8 involved Pd(O)- and Cu(I)-catalyzed coupling. [c]Isolated yield after preparative TLC, silica flash chromatography, or short-path distillation.

Finally, useful stereoselectivities have been recorded for the heteroconjugate addition of organometallic reagents to 1-silyl substituted vinyl sulfones.[52] [53] The synthesis of such sulfones can be achieved starting from phenylthioacetylene.[15,19,54] The synthesis of the dicobalt hexacarbonyl complex[55] and the polymerization of phenylthioacetylene[56] have been described.

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Appendix
Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

hexanes

brine

HCHO

sodium-benzophenone ketyl

N,O-dimethylhyroxylamine hydrochloride

t-$BuMe_2SiCl$

MeI

$ClCO_2Et$

PhI

BuI

Me_3SiCl

ethanol (64-17-5)

ammonia (7664-41-7)

Benzene (71-43-2)

ether (60-29-7)

ammonium chloride (12125-02-9)

sodium hydroxide (1310-73-2)

bromine (7726-95-6)

sodium sulfate (7757-82-6)

sodium bisulfite (7631-90-5)

acetone (67-64-1)

potassium hydroxide (1310-58-3)

sodium (13966-32-0)

1,2-dibromoethane (106-93-4)

ferric chloride (7705-08-0)

2-chloroethanol (107-07-3)

dichloromethane (75-09-2)

Thiophenol (108-98-5)

tantalum (7440-25-7)

sodium amide (7782-92-5)

hexane (110-54-3)

argon (7440-37-1)

diphenyl disulfide (882-33-7)

phenylsulfinyl chloride,
phenyl sulfinyl chloride (4972-29-6)

methyl phenyl sulfide (100-68-5)

lithium diisopropylamide (4111-54-0)

anisaldehyde (123-11-5)

bromoacetaldehyde diethyl acetal (2032-35-1)

propiolic acid (471-25-0)

tetrabutylammonium hydrogen sulfate (32503-27-8)

Phenyl vinyl sulfide (1822-73-7)

1-Bromovinyl phenyl sulfide (80485-53-6) cis- and trans-2-bromovinyl phenyl sulfide

1,8-diazabicyclo[5.4.0]undec-7-ene (6674-22-2)

Phenylthioacetylene,
Benzene, (ethynylthio)- (6228-98-4)

phenylthio-1,2-dibromoethane

sodium phenylthioacetylide

cis-1,2-bis(phenylthio)ethylene

cis-1,2-dichloroethylene (156-59-2)

dimethyl(chloroethynyl)carbinol

lithium trimethylsilyl acetylide

phenyl benzenethiosulfonate

cis-1-bromo-2-phenylthioethylene