Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

A Convenient Alternative to Prepare Phosphine Sulfides

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Published online: 24 Sep 2006.

To cite this article: R. Miranda , I. Salas , J. Mondragón & L. Velasco (1992) A Convenient Alternative to Prepare Phosphine Sulfides, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 22:7, 1077-1080, DOI: 10.1080/00397919208019299

To link to this article: http://dx.doi.org/10.1080/00397919208019299

SYNTHETIC COMMUNICATIONS, 22(7), 1077-1080 (1992)

A CONVENIENT ALTERNATIVE TO PREPARE PHOSPHINE SULFIDES

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Abstract: An efficient procedure to obtain phosphine sulfides by means of their corresponding phosphines in presence of sodium polisulfide solution, is described. Reaction yields are high and the work-up is very simple.

It has been reported that phosphine sulfides like their corresponding phosphines and phosphine oxides are very important as ligands in complexes of transitional elements[1], constituents in herbicides formulations[2] as well as catalysts in several processes[3]. Of particular interest is Thiotepa (*tris* -N-aziridinylphosphine sulfide) due to its relevant antineoplasic activity[4].

As a part of a program[5] we are interested in triphenylphosphine sulfides as ligands to prepare metal complexes

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in order to synthesize new conducting polymers by an electrochemical procedure. In the course of obtaining the target molecules(1-8) we found that these compounds may be obtained using a sodium polisulfide solution.

In the table are summarized the results of several experiments performed to obtain compounds 1-8 in the presence of a sodium polisulfide solution, using MeOH-Me_2CO as solvent. It shows that the corresponding sulfides are obtained in excellent yields.

In general, the pure compound was obtained almost immediately, and isolated by direct crystallization from the reaction mixture; besides the work-up procedure is very simple.

It is also worth mentioning that the sodium polisulfide solution serves as reagent-indicator since a pale yellow color is obtained at the end of the reaction.

Experimental.

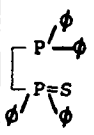
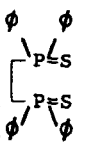
The chemicals obtained were characterized by common spectroscopic methods. ^1H NMR spectra were recorded on a Varian FT-80 spectrometer. EIMS spectra were obtained on a Hewlett-Packard 5985-B GC/MS spectrometer. Sodium polisulfide solution was obtained by a previously reported method.[6]

Sulfide derivatives:

In a typical example to 1.31g (5 mmol) of triphenylphosphine in 25 ml of MeOH-Me_2CO (1:9) were added by

TABLE

SULFIDES FROM PHOSPHINES AND SODIUM POLISULFIDE SOLUTION

	Phosphine	Sulfide[a]	Yield[b] (%)	Time[c] (min)	m.p. (°C)
1	Triphenyl	$\phi_3P{=}S$	95	2	157-8
2	Tri-*o*-Tolyl	*o*-Me$\phi_3P{=}S$	90	2	157-9
3	Tri-*m*-Tolyl	*m*-Me$\phi_3P{=}S$	92	2	152-4
4	Tri-*p*-Tolyl	*p*-Me$\phi_3P{=}S$	90	3	185-6
5	Tri-*o*-Anisyl	*o*-MeO$\phi_3P{=}S$	90	3	236-8
6	Tri-*p*-Anisyl	*p*-MeO$\phi_3P{=}S$	92	5	108-9
7	*bis*(1,2-diphenylphosphine)-ethane		85	5	188-90
8	*bis*(1,2-diphenylphosphine)-ethane		85	5	227-9

a.-ALL THE REACTIONS WERE CARRIED OUT AT ROOM TEMPERATURE. b.-YIELDS ARE OF ISOLATED PURE COMPOUNDS.
c.- SINCE THE REACTION TIMES ARE VERY SHORT, THE END-POINTS ARE DETERMINATED BY MONITORING THEM WITH THE REAGENT-INDICATOR.

dropping under magnetic stirring the sodium polisulfide solution at r.t. The reaction was monitored by tlc (*n*-hexane/EtOAc 8:2) and by means of the color permanence of the reagent-indicator. The triphenylphosphine sulfide crystallize from the reaction mixture. Yield 95% of pure compound.

ACKNOWLEDGEMENTS:
R. Miranda gratefully acknowledges DGAPA UNAM - IN301791 by which this work was supported.

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(Accepted in USA 1 November, 1991)