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A Convenient Alternative to Prepare Phosphine Sulfides

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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 polysulfide 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 elements¹, constituents in herbicides formulations² as well as catalysts in several processes³. Of particular interest is Thiotepa (tris-N-aziridinylphosphine sulfide) due to its relevant antineoplastic activity⁴.

As a part of a program⁵ 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 polysulfide solution.

In the table are summarized the results of several experiments performed to obtain compounds 1-8 in the presence of a sodium polysulfide solution, using MeOH-Me₂CO 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 polysulfide solution serves as reagent-indicator since a pale yellow color is obtained at the end of the reaction.

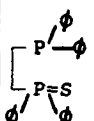
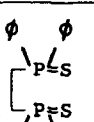
Experimental.

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 polysulfide 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-Me₂CO (1:9) were added by

TABLE
SULFIDES FROM PHOSPHINES AND SODIUM POLISULFIDE
SOLUTION

	Phosphine	Sulfide ^a	Yield (%) ^b	Time (min) ^c	m.p. (°C)
1	Triphenyl	$\phi_3\text{P}=\text{S}$	95	2	157-8
2	Tri- <i>o</i> -Tolyl	<i>o</i> -Me $\phi_3\text{P}=\text{S}$	90	2	157-9
3	Tri- <i>m</i> -Tolyl	<i>m</i> -Me $\phi_3\text{P}=\text{S}$	92	2	152-4
4	Tri- <i>p</i> -Tolyl	<i>p</i> -Me $\phi_3\text{P}=\text{S}$	90	3	185-6
5	Tri- <i>o</i> -Ani- syl	<i>o</i> -MeO $\phi_3\text{P}=\text{S}$	90	3	236-8
6	Tri- <i>p</i> -Ani- syl	<i>p</i> -MeO $\phi_3\text{P}=\text{S}$	92	5	108-9
7	bis(1,2-di- phenylphos- phine)-etha- ne		85	5	188-90
8	bis(1,2-di- phenylphos- phine)-etha- ne		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 DETERMINED 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.

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