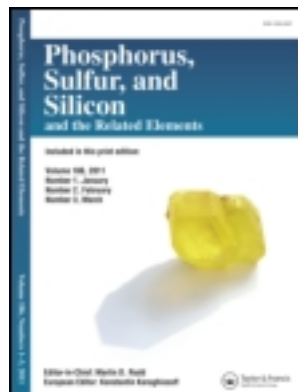


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### Reaction of a 2H-1,2,3-Diazaphosphole with Ethane-1,2-Dithiol

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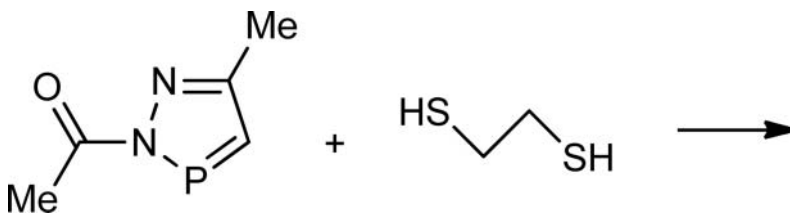
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## REACTION OF A 2*H*-1,2,3-DIAZAPHOSPHOLE WITH ETHANE-1,2-DITHIOL

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### GRAPHICAL ABSTRACT



**Abstract** Reaction of 2-acetyl-5-methyl-2*H*-1,2,3-diazaphosphole with ethane-1,2-dithiol at  $-30^{\circ}\text{C}$  leads to the formation of 2-acetyl-3-( $\beta$ -mercaptoethylthio)-5-methyl-1,2,3-diaza-phospholene. On heating, this product forms 2-( $\beta$ -mercaptoethylthio)-1,3,2-dithia-phospholane, 1,2-bis(1,3,2-dithiaphospholanyl)-dithioethane, and *N*-acetyl-*N'*-isopropylidene-hydrazine.

**Keywords** 2*H*-1,2,3-Diazaphosphole; ethanedithiol; 1,2,3-diazaphospholene; 1,3,2-dithia-phospholane

## INTRODUCTION

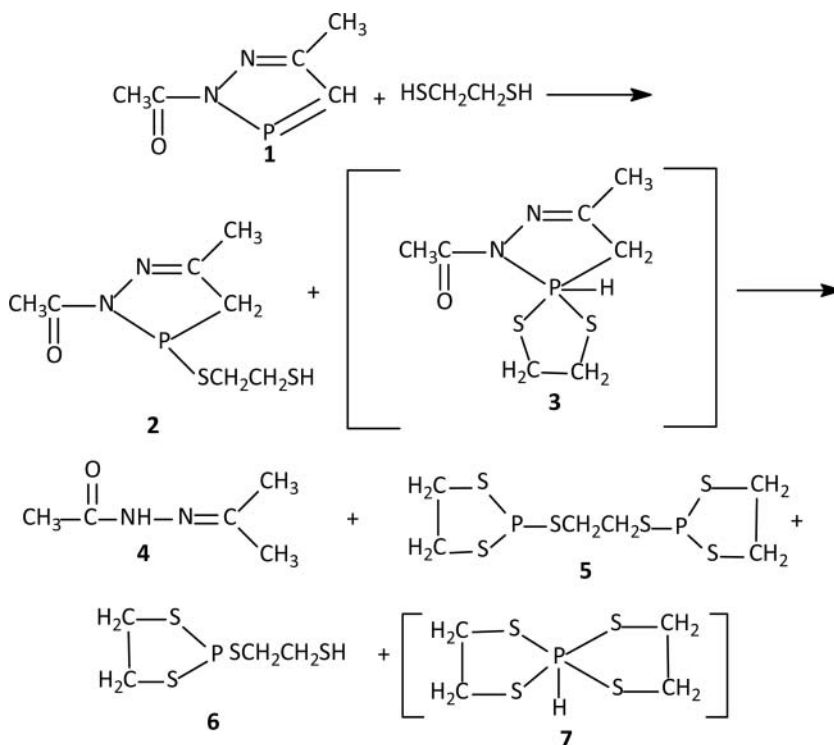
The interaction of heterocyclic two-coordinated phosphorus derivatives containing  $\lambda 3\sigma^2\text{-P} = \text{C}$  moieties with bifunctional nucleophilic reagents is only little investigated. Earlier, we reported that the reaction of 2-acetyl-5-methyl-2*H*-1,2,3-diazaphosphole with (*rac*)-butane-2,3-diol at temperatures below  $0^{\circ}\text{C}$  leads to the formation of a hydrospiroposphorane containing both a diazaphospholene and a dioxaphospholane ring and a  $\beta$ -hydroxy-alkoxy-1,2,3-diaza-phospholene. On heating, these products form hydrospirotetraoxaphosphorane, its tautomeric monocyclic  $\beta$ -hydroxyalkylphosphite, and *N*-acetyl-*N'*-isopropylidene hydrazine.<sup>1</sup>

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## RESULTS AND DISCUSSION

Herein, we report the results of our study of the reaction of 2-acetyl-5-methyl-2*H*-1,2,3-diazaphosphole (**1**) with ethane-1,2-dithiol. Monitoring the reaction of a mixture of these reactants in CH<sub>2</sub>Cl<sub>2</sub> via <sup>31</sup>P NMR spectroscopy at -30°C showed the appearance of a signal at 74 ppm, which indicated the formation of 2-acetyl-3-(β-mercaptoethylthio)-5-methyl-1,2,3-diazaphospholene (**2**) (Scheme 1). Gradually, an insoluble powder forms in the reaction mixture. The mass spectrometric analysis of the powder shows the presence of a few compounds. The main ion peaks [MH]<sup>+</sup> in the mass spectra (chemical ionization) of the powder were at 115, 217, 239, and 339 m/z. Consequently, the masses of the molecular ions of the compounds that are present in the reaction mixture were 114 (*N*-acetyl-*N'*-isopropylidene-hydrazine [**4**]),<sup>1</sup> 216, 238, and 338. Based on a consideration of the fragmentation pattern of these ions, it follows that the molecular ion peak at 216 m/z (C<sub>4</sub>H<sub>9</sub>PS<sub>4</sub>) corresponds to 2-(β-mercaptoethylthio)-1,3,2-dithiaphospholane (**6**). The molecular ion peak with a mass of 338 (C<sub>6</sub>H<sub>12</sub>P<sub>2</sub>S<sub>6</sub>) corresponds to the 1,2-bis(1,3,2-dithiophospholanyl)dithioethane (**5**) and the peak with a mass of 238 corresponds to diazaphospholene **2**.



Scheme 1

It was established (<sup>31</sup>P NMR) that the interaction of **1** with ethanedithiol under different reaction conditions does not proceed with formation of the hydrospirothiophosphoranes **3** or **7**. Instability of the thiophosphoranes **3** and **7** is confirmed by quantum-chemical calculations (PM3 and HF/6-31G(*d,p*)).

2-Acetyl-3-( $\beta$ -mercaptoethylthio)-5-methyl-1,2,3-diazaphospholene (**2**): yield 78%, m.p. 83–85°C.  $^{31}\text{P}$  NMR (300 MHz,  $\text{CH}_2\text{Cl}_2$ ):  $\delta = 74$  ppm. EA Calcd. for  $\text{C}_7\text{H}_{12}\text{N}_2\text{OPS}_2$ : C 35.74, H 5.11, N 11.91, S 27.23, P 13.19; found: C 35.79, H 5.61, N 12.02, S 27.11, P 13.19.

## REFERENCE

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