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ON THE PREPARATION AND PROPERTIES OF A THIODI-PHOSPHORUS (IV)-ACID BETAINE

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On heating phosphorus(V) sulfide in anhydrous pyridine—the pentathiodiphosphorus(V) acid-P,P'-bis(pyrididium betaine), $(C_8H_8N)_2P_2S_8$ (1), is formed (1,2). In 1 the P atoms are linked via an S atom. The nucleophilic degradation of the sulfid P₄S₇ by pyridine yields the new tetrathiodiphosphorus(IV) acid-P,P'-bis(pyrididium betaine), $(C_8H_8N)_2P_2S_4$ (2) which contains a direct P-P bond. 2 is already formed at room temperature on shaking of P₄S₇ with anhydrous pyridine for several days, resulting as a microcrystalline yellow solid in almost 50% yield. As has been proven by means of PNMR spectroscopy, P₄S₃ is a further product of this reaction.

On reaction of the sulfide P_4S_9 with anhydrous pyridine an equimolar mixture of $\underline{1}$ and $\underline{2}$ results:

 $P_4S_9 + 4 C_9H_8N \longrightarrow (C_9H_9N)_2P_2S_9 + (C_9H_9N)_2P_2S_4$ Due to different solubilities in pyridine, the dimeric betaines may be easily separated from eachother.

 $\underline{2}$ is significantly more stable then $\underline{1}$. On heating of $\underline{2}$ loss of pyridine sets in only above 140°C. The oxidation of $\underline{2}$ with H_2O_2 yield hypophosphate, $M_4^1(P_2O_4)$. On heating in strongly basic solution the corresponding tetrathiohypophosphate is formed in high yield:

 $(C_8H_8N)_2P_2S_4 + 4 M^1OH \longrightarrow M^1_4(P_2O_2S_4) + 2 C_8H_8N + 2 H_2O$

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