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Phosphorus, Sulfur, and Silicon and the Related Elements

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Phosphonomethylation of Dinitroaniline-Substituted Alkylamines

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PHOSPHONOMETHYLATION OF DINITROANILINE-SUBSTITUTED ALKYLAMINES

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Abstract It was found that aminoalkyl derivatives of 2,4-dinitroaniline can be phosphonomethylated at the terminal aliphatic amino group using a known method, whereas the aromatic part of the molecule and bound nitrogen atom remain unaffected.

Keywords *a*-Aminophosphonates; phosphonomethylation

INTRODUCTION

N-Alkylated 2,4-dinitroanilines show a strong inhibition of plant growth and may be used as contact herbicides.¹ 2,4-Dinitrophenylpiperazine derivatives are similarly active.² The purpose of our study was the synthesis and evaluation of properties of analogous compounds containing hydrophilic phosphonic groups in their structure.

Phosphonomethylation of alkylamines or ammonia by a mixture of formaldehyde and phosphoric acid in strong acidic medium is a well known and widely used reaction.³ At the same time, we have not found examples of the use of this reaction for the synthesis of aminophosphonates containing N-phenyl group in their structure. It is the result of the undesired interaction of formaldehyde with the anilines in the reaction condition that results in the formation of byproducts of polymeric character.

RESULTS AND DISCUSSION

The starting N-aminoalkyl derivatives of 2,4-dinitroaniline were synthesized in a reaction of 2,4-dinitrochlorobenzene with an excess of a polyamine.

When refluxed in hydrochloric acid with phosphoric acid and formaldehyde, the 2,4-dinitrophenyl substituted amino group and the aromatic part of the molecule was not modified in the title reaction, while the primary or secondary alkyl amine converted to the corresponding bis (compound 1 and 3) or mono methylenephosphonate (compound 2), respectively (Scheme 1). The products crystallized from the reaction mixture after partial evaporation of hydrochloric acid and dilution the mixture with water.

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Scheme 1 The products of phosphonomethylation of selected N-aminoalkyl derivatives of 2,4-dinitroaniline and the obtained yields.

The products are bright yellow solids that decompose above 200°C, and are insoluble in water and in most organic solvents. They can be recrystallized from DMSO or acetic acid. They form salts with alkali and alkylamines, and are well soluble in water and DMSO, respectively.

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