

AN IMPROVED SYNTHESIS OF 1-AMINOPHOSPHONIC ACIDS

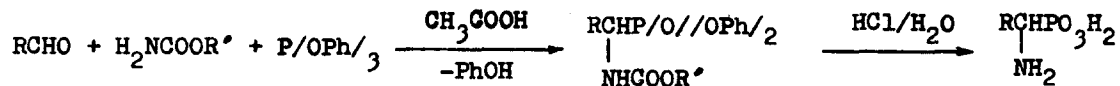
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Aminophosphonates are most often obtained by reaction of amines, aldehydes or ketones and dialkyl phosphites¹⁻⁴. It is also known that urea or thiourea react readily with aldehydes and triphenyl phosphite to form the corresponding ureido or thioureidophosphonates⁵.

In this communication we report a new synthesis of aminophosphonic acids involving the condensation alkylcarbamates with aldehydes and triphenyl phosphite. Acid hydrolysis of the crude condensation products affords fair yields of aminophosphonic acids.



This method seems to be the simplest procedure for synthesis of 1-aminophosphonic acids.

GENERAL PROCEDURE

A solution of an alkylcarbamate /0,05 mol/, triphenylphosphite /0,05 mol/ and an aldehyde /0,07 mol/ in 10 ml of glacial acetic acid is refluxed for 1 h. Then the mixture is treated with 50 ml of conc. hydrochloric acid and hydrolyzed under reflux for 6 h. The cooled solution is then washed with 20 ml benzene /to remove phenol/ and the aqueous solution is evaporated in vacuo to dryness. The residue is dissolved in 50 ml of methanol and treated with propene oxide until pH 6 is reached. The precipitated aminophosphonic acid is recrystallized from ethanol/water. Results are summarised in the table.

Table. 1-Aminophosphonic Acids RCHPO_3H_2
 NH_2

R	Yield %/		M.p.	Lit. m.p.
	$\overset{\text{R}}{\text{R}}=\text{C}_2\text{H}_5$	$\overset{\text{R}}{\text{R}}=\text{CH}_2\text{C}_6\text{H}_5$		
CH_3	42	54	270-2°	272-4° ³
C_2H_5	36	59	262-3°	264-6° ³
n- C_3H_7	38	51	273-4	262-4° ³
i- C_3H_7	39	55	270-2	274° ⁶
C_6H_5	35	43	281-2°	280-2° ³
p- $\text{CH}_3\text{C}_6\text{H}_5$	32	43	278-9°	276° ⁷

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