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NOTE

Dipotassium Hydrogen Phosphate Powder Catalyzed Conversion of Dimethyl-2-(5-bromo-2-hydroxyphenyl)-3-(triphenylphosphoranylidene)butanedioate to Methyl-6-bromo-2-oxo-2*H*-chromene-4-carboxylate

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Dipotassium hydrogen phosphate powder was found to catalyze conversion of dimethyl 2-(5-bromo-2-hydroxyphenyl)-3-(triphenylphosphoranylidene)butanedioate to methyl 6-bromo-2-oxo-2*H*-chromene-4-carboxylate in solvent-free conditions at room temperature in fairly high yield.

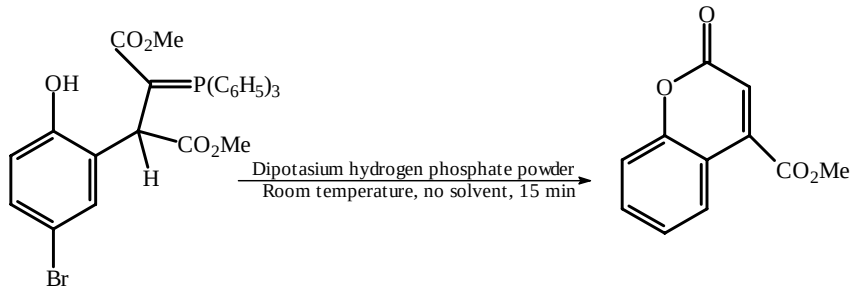
Key Words: Coumarin, 4-Bromophenol, Dipotasium hydrogen phosphate, Vinyltriphenylphosphonium salt, Dimethyl acetylenedicarboxylate.

Chromene skeleton compounds are well known natural products and many such compounds exhibited high levels of biological activity[1-7]. Earlier, a convenient, one-pot method is established for preparing stabilized phosphorus ylides utilizing *in situ* generation of the phosphonium salts[8]. Recently, the catalytic role of silica gel powder in the synthesis of coumarins in solvent-free conditions[9] have been reported[10]. In this paper, the catalytic role of dipotasium hydrogen phosphate in the conversion of dimethyl-2-(5-bromo-2-hydroxyphenyl)-3-(triphenylphosphoranylidene)butanedioate (**1**) to methyl-6-bromo-2-oxo-2*H*-chromene-4-carboxylate (**2**) in solvent-free conditions at room temperature in fairly high yield (**Scheme-1**).

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were recorded on a Shimadzu IR-460 spectrometer. ^{1}H and ^{13}C NMR spectra were measured with a Bruker DRX-500 Avance spectrometer at 500 and 125 MHz, respectively.

Procedure for the preparation of coumarin (2): Mixture of powdered ylide **1** (1 mmol) and dry dipotasium hydrogen phosphate (1.5 g) was stirred at room temperature for 15 min and then placed over a column of silica gel (10 g). The column chromatography was washed using ethyl acetate-light petroleum ether (1:10) as eluent. The solvent was removed under reduced pressure and product was obtained as white crystals (yield: 72 %). The characterization data of the compound **1** are reported previously[11].

Dipotasium hydrogen phosphate was found to catalyzed conversion of dimethyl-2-(5-bromo-2-hydroxyphenyl)-3-(triphenylphosphoranylidene)-butanedioate (**1**) to methyl-6-bromo-2-oxo-2*H*-chromene-4-carboxylate (**2**) in solvent-free conditions at room temperature in fairly high yield (**Scheme-1**). TLC indicated formation of coumarin **2**. The reaction was completed after 15 min. The reaction proceeds smoothly and cleanly under solvent-free conditions and no side reactions were observed.



Scheme-1

The structure of compound **2** was deduced from its UV, IR, ^{1}H NMR, ^{13}H NMR and Mass spectrometry. All of these data are given in our previous report[11].

In summary, the dipotasium hydrogen phosphate powder is found to catalyze conversion of dimethyl-2-(5-bromo-2-hydroxyphenyl)-3-(triphenylphosphoranylidene)butanedioate (**1**) to methyl-6-bromo-2-oxo-2*H*-chromene-4-carboxylate (**2**) in solvent-free conditions at room temperature in fairly high yield.

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