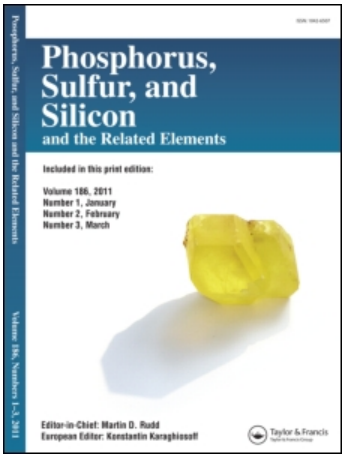

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MICROWAVE-ASSISTED N-ALKYLATION OF POTASSIUM PHTHALIMIDE AND POTASSIUM SUCCINIMIDE ONTO SILICA GEL IN DRY MEDIA

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Synthesis of N-alkyl phthalimides and N-alkyl succinimides through alkylation of potassium phthalimide and potassium succinimide in dry media catalyzed by phase-transfer catalyst under microwave irradiation will be reported. The reactions were with fairly high yield.

Keywords: Microwave; potassium phthalimide; potassium succinimide; TBAB

INTRODUCTION

In the past few years there has been interest in the use of microwave heating in organic synthesis.[1] The use of such nonconventional reaction conditions reveals features such as the following: a short reaction time compared to conventional heating, ease of workup after the reaction, reduction in the usual thermal degradation, better selectivity,[2] and reaction under dry conditions (i.e., in the absence of a solvent on a solid support with or without catalysts), all of which were originally developed in the late 1980s.[3] Synthesis without solvents under microwave irradiation offers several advantages.[4] The absence of solvent reduces the risk of explosions when the reaction takes place in a closed vessel in an oven.

Moreover, aprotic dipolar solvents with high boiling points are expensive and difficult to remove from the reaction mixtures. During microwave induction of reactions under dry conditions, the reactants adsorbed on the surface of alumina, silica gel, clay, and others absorb

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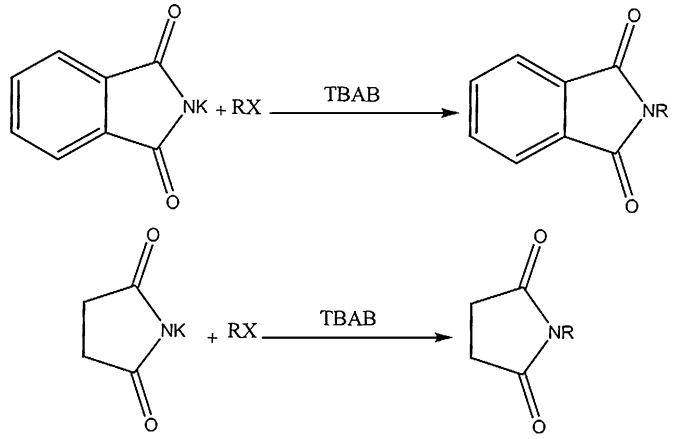
the microwaves, whereas the support does not, nor does it restrict the transmission of microwaves. Consequently, such supported reagents efficiently induce reactions under safe and simple conditions with domestic microwave ovens instead of specialized expensive commercial microwave systems.

The Gabriel synthesis for converting halides to primary amines is based on the reaction of halide with potassium phthalimide. The reaction is usually rather slow but can be conventionally speeded by the use of an aprotic solvent such as DMF.

In this article we report the remarkably fast synthesis method of N-alkyl phthalimide and N-alkyl succinimide via alkylation of potassium phthalimide and potassium succinimide in dry media under microwave irradiation (Scheme 1).



SCHEME 1

RESULTS AND DISCUSSION

The reactions were carried out by simply mixing of potassium phthalimide or potassium succinimide with an excess of alkyl halide, a catalytic amount of tetrabutyl ammonium bromide (TBAB), and silica gel powder in CH_2Cl_2 with stirring. The solvent was evaporated and then the mixture irradiated in an open vessel in a domestic microwave oven for the indicated time in Table I.

Superheating of liquids is common under microwave irradiation, thus the strategy of reaction is to keep the reaction temperature substantially below the boiling point of each compound used for the

TABLE I Phthalimide and Succinimide Derivatives Synthesis by TBAB Catalyst and Alkyl Bromide Under Microwave Irradiation

				m.p. (°C) or b.p. (°C)/mm Hg	
Product	RX	Time (min)	Yield (%)	Found (Bielstin)	Reported
N-benzyl phthalimide	Benzyl bromide	3	98	116	114
N-propargyl phthalimide	Propargyl bromide	4	91	66	67
N-allyl phthalimide	Allyl bromide	4	89	71	70
N-ethyl phthalimide	Ethyl bromide	4	76	79	77
N-methyl phthalimide	Methyl bromide	4	68	134	135
N-benzyl succinimide	Benzyl bromide	3	88	103	103
N-methyl succinimide	Methyl bromide	4	53	71	64.5
N-propargyl succinimide	Propargyl bromide	4	75	240	237/760 mm Hg
N-allyl succinimide	Allyl bromide	4	72	243	241/760 mm Hg
N-ethyl succinimide	Ethyl bromide	4	60	236	232/760 mm Hg

reaction. One of the best solutions is to repeat an experiment several times, increasing the power slowly so that vapors do not escape.

EXPERIMENTAL

Melting points were measured with an electrothermal 9100 apparatus and are uncorrected. Infrared (IR) spectra were recorded on a Philips PU 9800 fourier transform infrared (FTIR) instrument. ^{1}H and ^{13}C NMR spectra were recorded on a JEOL EX-90A spectrometer at 90 and 22.6 MHz. All products are known compounds; their physical and spectroscopic data were compared with those of authentic samples and were found to be identical. Mass spectra were recorded on a Finnigan-MaH8430 mass spectrometer operating at an ionization potential 70 eV.

GENERAL PROCEDURE

Potassium phthalimide (20 mmol) was powdered in a mortar and thoroughly mixed with TBAB (0.5 mmol) and silica gel powder (1 g). Alkyl halide was added dropwise, and then 5 ml of CH_2Cl_2 was added. The mixture was stirred, the solvent evaporated, and then the mixture placed in a microwave oven and irradiated for the time indicated in Table I. The product was extracted by methylene chloride, the solvent removed, and the crude product recrystallized from ethanol to afford the pure product.

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