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Michael Addition of Phthalimide and Saccharin to α,β-Unsaturated Esters Under Solvent-Free Conditions

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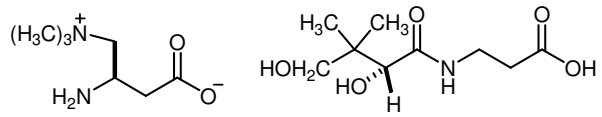
An efficient, clean and simple procedure for the Michael addition of phthalimide and saccharin to various α,β-unsaturated esters in the presence of tetrabutylammonium bromide (TBAB) and 1,4-diazabicyclo[2,2,2]octane (DABCO) under solvent-free conditions is described. By this method, the Michael adducts are obtained in good to excellent yields in short reaction times under thermal and microwave conditions.

Keywords: Michael addition, Phthalimide, Saccharin, α,β-Unsaturated esters, Solvent-free

INTRODUCTION

The Michael reaction of phthalimide and saccharin with α,β-unsaturated esters is of importance because this reaction provides a simple route towards the synthesis of *N*-alkyl derivatives of phthalimide and saccharin, which are biologically interesting compounds. Some *N*-alkyl phthalimides have been applied as antipsychotic [1] and anti-inflammatory agents [2], and as receptors [3]. Saccharin and its derivatives are important in medicinal chemistry [4]. Furthermore, saccharin can form metal complexes by its reaction with metal cations, such as Mg^{2+}, Ca^{2+}, Ba^{2+}, Sr^{2+}, and Cu^{2+} [5]. The conjugate addition of phthalimide and saccharin to α,β-unsaturated esters also affords protected β-amino acids, essential components in many bioactive compounds and drug scaffolds, such as β-peptides [6], emeriamine (a hypoglycemic and antiketogenic agent, Fig. 1) [7], vitamin B_3 (Fig. 1) [8], cryptophycin (an antitumor agent) [9] and TAN-1057 A (an antibiotic) [10].



Emeriamine Vitamin B_3

Fig. 1. The structures of emeriamine and vitamin B_3.

Solvent-free organic reactions have been applied as useful protocols in organic synthesis [11]. Solvent-free reactions under thermal or microwave conditions often lead to shorter reaction times, increased yields and easier workup, in addition to working well in green chemistry protocols, and enhancing the regio- and stereoselectivity of reactions [11].

Different methods have been established for the Michael addition of various nucleophiles to α,β-unsaturated compounds [12]; however, there are only a few reports regarding the aza-conjugate addition of imides. In aza-

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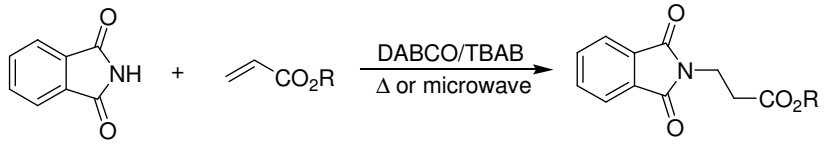
conjugate addition, the nucleophilic nitrogen is usually among the most powerful, such as the use of amines [13], although this can lead to side products, such as amides, *via* nucleophilic attack of amine to the carbonyl group of α,β-unsaturated esters [13a]. Furthermore, 1,2 and 1,4 condensations of β-amino residues with α,β-unsaturated esters cause polymerization or tar formation [13a]. Moreover, the reported procedures often require a large excess of reagents, such as triethylamine [13b]. Therefore, in this context, the use of imides instead of amines in the *N*-conjugate addition with α,β-unsaturated esters is more desirable, since the lower nucleophilicity of nitrogen reduces the amount side product formation.

In previous methods for the aza-Michael addition of imides or their salts to α,β-unsaturated compounds, reagents such as Na in absolute ethanol [14a], K_2CO_3 [14b] and $AlMe_2Cl$ [14c] have been used. However, these reactions have been performed in solution using long reaction times. To the best of our knowledge, the Michael addition of imides to α,β-unsaturated esters under solvent-free conditions is rare in the literature. Along with our previous works on other Michael additions [15], and in extension of our previous studies on the application of solvent-free conditions in organic synthesis [16], we now describe a solvent-free procedure for the Michael addition of phthalimide and saccharin to α,β-unsaturated esters in the presence of TBAB and DABCO under thermal and microwave irradiation conditions (Schemes 1 and 2).

EXPERIMENTAL

All chemicals were obtained from Merck or Fluka Chemical Companies. Some α,β-unsaturated esters were prepared from the corresponding acid chlorides by the reported method [17] and their structures were established using IR and ^{1}H NMR spectra. The progress of the reactions was followed by TLC using silica gel SIL G/UV 254 plates. IR spectra were obtained on a Shimadzu FTIR-8300 spectrophotometer. ^{1}H NMR (250 MHz) and ^{13}C NMR (62.5 MHz) were performed on a Bruker Avanced DPX-250 FT-NMR spectrometer (δ in ppm, *J* in Hz). Mass spectra were recorded on a Shimadzu GCMS-QP1000 EX spectrometer. Melting points were recorded on a Büchi B-545 apparatus in open capillary tubes and are uncorrected. A domestic microwave oven (MB 245, Butan Industrial Company, Iran) was used for microwave irradiation.



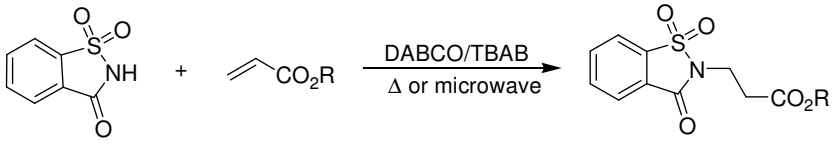
R = alkyl, aryl

1a-i

Δ 88-94%

MW 91-96%

Scheme 1



R = alkyl

2a-e

Δ 42-45%

MW 28-31%

Scheme 2

General Procedure for Michael Addition of Imides to α,β-Unsaturated Esters Under Solvent-Free Conditions

Thermal procedure. α,β-Unsaturated ester (15 mmol) was added to a 25 ml round-bottomed flask containing a well-ground mixture of imide (10 mmol), TBAB (1.61 g, 5 mmol) and DABCO (1.12 g, 10 mmol) connected to a reflux condenser. The resulting mixture was thoroughly stirred with a glass rod and then magnetically stirred in an oil-bath (100 °C) for the appropriate time (see Table 4). The reaction mixture was cooled to room temperature. Then chloroform (250 ml) was added to the mixture and the resulting chloroform solution was washed with water (2 × 200 ml) and dried over anhydrous Na_2SO_4. The chloroform was evaporated and the crude product was purified by column chromatography on silica gel eluted with EtOAc/*n*-hexane (1/5).

Microwave procedure. α,β-Unsaturated ester (15 mmol) was added to a test tube containing a well-ground mixture consisting of imide (10 mmol), TBAB (1.61 g, 5 mmol) and DABCO (1.12 g, 10 mmol) and mixed thoroughly with a glass rod. The resulting mixture was irradiated in a microwave oven at 300 W for the appropriate reaction time (see Table 4). The reaction mixture was then cooled to room temperature and dissolved in chloroform (250 ml). The chloroform solution was washed with water (2 × 200 ml) and dried over anhydrous Na_2SO_4. The solvent was evaporated and the crude product was purified by column chromatography on silica gel with EtOAc/*n*-hexane (1/5).

Physical and Spectroscopic Data of Isolated Products

(1a). Colorless solid; m.p.: 60-61 °C; IR (KBr): 3051, 2968, 1774, 1716 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.11 (t, *J* = 7.1 Hz, 3H, CH_3), 2.57 (t, *J* = 7.2 Hz, 2H, O=CCH_2), 3.86 (t, *J* = 7.2 Hz, 2H, NCH_2), 4.06 (q, *J* = 7.1 Hz, 2H, OCH_2), 7.57-7.70 (complex, 4H); ^{13}C NMR ($CDCl_3$): δ 13.8, 32.7, 33.5, 64.3, 122.9, 131.7, 133.8, 167.5, 170.6; MS m/z (%): 247 (M^+, 20.1).

(1b). Colorless solid; m.p.: 51-52 °C; IR (KBr): 3069, 2960, 1774, 1716 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.90 (t, *J* = 6.8 Hz, 3H, CH_3), 1.28 (m, 2H, CH_3CH_2), 1.59 (m, 2H, $CH_3CH_2CH_2$), 2.58 (t, *J* = 7.2 Hz, 2H, O=CCH_2), 3.91 (t, *J* = 7.2 Hz, 2H, NCH_2), 4.08 (t, *J* = 6.9 Hz, 2H, OCH_2), 7.64-7.76 (complex, 4H); ^{13}C NMR ($CDCl_3$): δ 13.3, 18.5, 30.2, 32.5, 33.4, 64.1, 122.8, 131.7, 133.6, 167.3, 170.3; MS m/z (%): 275 (M^+, 15.5).

(1c). Colorless solid; m.p.: 40-41 °C; IR (KBr): 3055, 2970, 1774, 1716 cm^{-1}; ^1H NMR ($CDCl_3$): δ 0.89 (t, *J* = 6.7 Hz, 3H, CH_3), 1.29-1.36 (complex, 6H), 1.60 (m, 2H, $CH_3(CH_2)_3CH_2$), 2.59 (t, *J* = 7.0 Hz, 2H, O=CCH_2), 3.95 (t, *J* = 7.0 Hz, 2H, NCH_2), 4.09 (t, *J* = 6.9 Hz, 2H, OCH_2), 7.68-7.83 (complex, 4H); ^{13}C NMR ($CDCl_3$): δ 13.7, 22.1, 25.4, 28.1, 30.2, 32.4. 33.4, 64.3, 122.7, 132.1, 133.6, 167.2, 170.2; MS m/z (%): 303 (M^+, 27.2).

(1d). Colorless solid; m.p.: 57-59 °C; IR (KBr): 3061, 2958, 1774, 1716 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.59 (t, *J* = 7.1 Hz, 2H, O=CCH_2), 3.86 (t, *J* = 7.1 Hz, 2H, NCH_2), 4.94 (s, 2H, OCH_2), 7.08-7.17 (complex, 5H), 7.52 (m, 2H), 7.63 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 32.9, 33.7, 66.6, 122.6, 128.2, 128.5, 129.4, 131.9, 133.9, 135.6, 167.8, 170.6; MS m/z (%): 309 (M^+, 19.4).

(1e). Colorless solid; m.p.: 53-55 °C; IR (KBr): 3052, 2933, 1774, 1716 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.69 (t, *J* = 7.2 Hz, 2H, O=CCH_2), 2.87 (t, *J* = 7.1 Hz, 2H, ArCH_2), 3.93 (t, *J* = 7.2 Hz, 2H, NCH_2), 4.26 (t, *J* = 7.1 Hz, 2H, OCH_2), 7.13-7.26 (complex, 5H), 7.64 (m, 2H), 7.77 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 32.8, 33.6, 34.9, 65.2, 123.1, 126.4, 128.3, 128.9, 132.7, 133.9, 137.6, 167.8, 170.7; MS m/z (%): 323 (M^+, 18.5).

(1f). Pale yellow oil; IR (neat): 3061, 2954, 1774, 1716, 1497 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.68 (t, *J* = 7.1 Hz, 2H, O=CCH_2), 3.92 (t, *J* = 7.1 Hz, 2H, NCH_2), 4.64 (m, 2H, OCH_2), 6.12 (m, 1H, ArCH=CH), 6.49 (d, *J* = 15.8 Hz, 1H, ArCH), 7.17-7.26 (complex, 5H), 7.57 (m, 2H), 7.72 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 32.9, 33.7, 65.4, 122.8, 123.2, 126.6, 128.0, 128.5, 131.9, 133.9, 134.4, 136.1, 167.9, 170.6; MS m/z (%): 335 (M^+, 13.4).

(1g). Pale yellow oil; IR (neat): 3480, 3049, 2948, 1770, 1715 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.63 (t, *J* = 7.0 Hz, 2H, O=CCH_2), 3.67 (s, 3H, CH_3), 3.80-390 (complex, 5H), 4.11-4.19 (complex, 3H), 6.73-6.80 (complex, 4H), 7.54 (m, 2H), 7.68 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 32.8, 33.6, 55.7, 65.7, 67.9, 70.5, 111.9, 114.2, 120.9, 121.8, 123.2, 131.7, 134.0, 147.8, 149.3, 168.0, 170.8; MS m/z (%): 399 (M^+, 20.3).

(1h). Colorless solid; m.p.: 70-72 °C; IR (KBr): 3076, 2970, 1775, 1713 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.74 (t, *J* = 7.1 Hz, 2H, O=CCH_2), 3.95 (t, *J* = 7.1 Hz, 2H, NCH_2), 6.98 (d,

J = 8.0 Hz, 2H), 7.09 (d, *J* = 6.9, 1H), 7.24 (dd, *J* = 6.9, 8.0 Hz, 2H), 7.61-7.73 (complex, 4H); ^{13}C NMR ($CDCl_3$): δ 32.9, 33.5, 121.4, 122.9, 125.7, 129.2, 131.6, 133.9, 150.3, 167.5, 170.3; MS m/z (%): 295 (M^+, 1.9).

(1i). Pale yellow solid; m.p.: 88-90 °C; IR (KBr): 3065, 2958, 1775, 1716 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.69 (t, *J* = 7.2 Hz, 2H, O=C*CH*$_2$), 3.92 (t, *J* = 7.2 Hz, 2H, N*CH*$_2$), 7.01-7.08 (complex, 3H), 7.21-7.31 (complex, 3H), 7.55-7.64 (complex, 3H), 7.76 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 32.0, 33.8, 108.4, 116.8, 121.6, 122.2, 122.6, 125.4, 126.7, 127.8, 128.7, 129.9, 133.0, 133.6, 152.6, 167.4, 170.6; MS m/z (%): 345 (M^+, 2.4).

(2a). Yellow oil; IR (neat): 3039, 2938, 1733, 1455, 1333 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.84 (t, *J* = 7.4 Hz, 3H, *CH*$_3$), 1.25 (m, 2H, CH_3*CH*$_2$), 1.51 (m, 2H, CH_3CH_2*CH*$_2$), 2.80 (t, *J* = 6.8 Hz, 2H, O=C*CH*$_2$), 4.00-4.08 (complex, 4H, N*CH*$_2$ and O*CH*$_2$), 7.81-7.97(complex, 4H); ^{13}C NMR ($CDCl_3$): δ 12.6, 18.0, 29.4, 32.0, 33.6, 63.9, 119.9, 124.2, 126.2, 133.3, 133.8, 136.4, 157.6, 169.3; MS m/z (%): 311 (M^+,0.8), 247 (M^+-SO_2, 20.9).

(2b). Pale yellow oil; IR (neat): 3051, 2931, 1734, 1457, 1339 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.78 (t, *J* = 7.0 Hz, 3H, *CH*$_3$), 1.16-1.25 (complex, 6H), 1.53 (m, 2H, $CH_3(CH_2)_3$*CH*$_2$), 2.81 (t, *J* = 6.7 Hz, 2H, O=C*CH*$_2$), 3.95-4.04 (complex, 4H, N*CH*$_2$ and O*CH*$_2$), 7.78-7.92 (complex, 4H); ^{13}C NMR ($CDCl_3$): δ 12.9, 21.4, 24.4, 27.4, 30.3, 32.0, 33.6, 64.1, 119.9, 124.1, 126.1, 133.4, 133.9, 136.6, 157.6, 169.2; MS m/z (%): 339 (M^+, 1.3), 275 (M^+-SO_2, 29.7).

(2c). Yellow oil; IR (neat): 3061, 2954, 1733, 1338,1303,1182 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.85 (t, *J* = 6.9 Hz, 2H, O=C*CH*$_2$), 4.03 (t, *J* = 6.9 Hz, 2H, N*CH*$_2$), 4.70 (m, 2H, O*CH*$_2$), 6.23 (m, 1H, ArCH=*CH*), 6.54 (d, 1H, *J* = 15.7 Hz, ArC*H*), 7.19-7.30 (complex, 5H), 7.76-7.93 (complex, 4H); ^{13}C NMR ($CDCl_3$): δ 32.06, 33.6, 64.5, 119.9, 121.7, 124.2, 125.6, 126.1, 126.7, 127.0, 127.5, 133.3, 133.5, 133.8, 136.8, 157.5, 168.5; MS m/z (%): 307 (M^+-SO_2, 15.4).

(2d). Pale yellow oil; IR (neat): 3033, 2982, 1734, 1457, 1340 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.18 (d, *J* = 6.6 Hz, 6H, 2*CH*$_3$), 2.77 (t, *J* = 6.7, 2H, O=C*CH*$_2$), 4.01 (t, *J* = 6.7 Hz, 2H, N*CH*$_2$), 4.97 (q, *J* = 6.6, 1H, O*CH*), 7.79-7.95 (complex, 4H); ^{13}C NMR ($CDCl_3$): δ 20.7, 32.2, 33.6, 67.5, 119.9, 124.2, 126.2, 133.3, 133.8, 136.6, 157.6, 168.7; MS m/z (%): 297 (M^+, 1.9), 233 (M^+-SO_2, 38.5).

(2e). Colorless solid; m.p.: 62-64 °C; IR (neat): 2938, 1733, 1339 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.19-1.36 (complex, 6H), 1.65-1.81 (complex, 4H), 2.81 (t, *J* = 6.8 Hz, 2H, O=C*CH*$_2$), 4.04 (t, *J* = 6.8 Hz, 2H, N*CH*$_2$), 4.76 (m, 1H, O*CH*), 7.81-8.00 (complex, 4H); ^{13}C NMR ($CDCl_3$): δ 23.6, 25.2, 31.4, 33.2, 34.6, 73.3, 120.9, 125.1, 127.1, 134.3, 134.8, 137.5, 158.6, 169.6; MS m/z (%): 273 (M^+-SO_2, 32.9).

Table 1. Evaluation of Bases in the Michael Addition of Phthalimide with *n*-Butyl Acrylate in the Presence of TBAB

Entry	Base	Time (min)		Yield (%)[a]	
		Δ[b]	MW[c]	Δ	MW
1	DABCO	30	5	94	96
2	DMAP	30	5	74	77
3	NBu_3	30	5	76	79
4	1-Methylimidazole	30	5	78	84
5	Cs_2CO_3	40	6	65	70
6	K_2CO_3	60	7	51	64
7	NaOH	50	7	45	53
8	CaO	100	10	13	27
9	MgO	100	10	9	22

[a]Isolated yield. [b]Thermal conditions at 100 °C. [c]Microwave conditions at 300 W.

RESULTS AND DISCUSSION

In order to determine the optimal reaction conditions, we have studied the synthesis of compound **1b** as a model reaction (Scheme 1). At first, the effect of various organic and inorganic bases was examined under thermal and microwave conditions. The results are summarized in Table 1. When DABCO was used, higher yields and shorter reaction times were observed under both thermal and microwave conditions (Table 1).

In another study, the reaction of phthalimide with *n*-butyl acrylate in the presence of TBAB and DABCO was examined at different microwave powers (100-700 W). The best result

was obtained at 300 W.

In order to find the optimal temperature for thermal reactions, the reaction of phthalimide with *n*-butyl acrylate was examined at 60-130 °C. A higher yield and shorter reaction time were observed for the Michael adduct formation at 100 °C. No addition to carbonyl groups was observed for either method.

The role of TBAB was also evaluated under both reaction conditions. The absence of TBAB in the reaction media afforded low reaction yields, even after prolonged reaction times and increased reaction temperatures or microwave power. Thus, the presence of TBAB in the reactions is critical. Molten TBAB (m.p.: 102-103 °C) creates a homogeneous reaction media and is assumed to behave as an ionic liquid [18]. In addition, TBAB absorbs microwave irradiation, increasing the temperature of the reaction media. Other quaternary ammonium salts were also examined in the reaction, and TBAB gave the best results (Table 2).

To compare the efficiency of the solution *vs*. solvent-free preparation, a mixture of phthalimide (10 mmol), DABCO (10 mmol) and *n*-butyl acrylate (15 mmol) in DMSO (20 ml) was either heated in an oil-bath (100 °C) for 12 h or irradiated in a microwave oven (300 W) for 20 min. However; low yields of the product were obtained, even after elongated reaction times, or increased temperature or microwave power. In addition, no improvement in the yield of the reaction was observed by changing the volume of the solvent. Using solvents, such as DMF, HMPTA, and *o*-xylene, the product was isolated in low yields (Table 3). Therefore, the solvent-free reaction is more efficient.

To investigate the versatility and scope of our method, the reactions were examined with various α,β-unsaturated esters. The results are depicted in Table 4. As indicated in Table 4, all the reactions proceeded efficiently under both conditions and the desired Michael adducts were obtained in good to excellent yields.

To study the structural effect of the alkoxy group (-OR) of α,β-unsaturated esters (Michael acceptors) in this reaction, we have investigated the reaction of phthalimide and saccharin with esters containing sterically hindered alkoxy groups under both microwave and thermal conditions (Table 4). As shown in Table 4, the bulkiness of the alkoxy group had no significant effect on the yields and reaction times under microwave conditions. Under thermal conditions, the alkoxy group did not affect the reaction yields; however, longer reaction times were required when phthalimide or saccharin was introduced to esters possessing sterically hindered alkoxy groups. Using phenolic α,β-unsaturated esters, the reaction

Table 2. Evaluation of Quaternary Ammonium Salts in the Michael Addition of Phthalimide with *n*-Butyl Acrylate in the Presence of DABCO

Entry	Quaternary ammonium salt	Time (min)		Yield (%)[a]	
		Δ[b]	MW[c]	Δ	MW
1	-	120	20	18	25
2	TBAB	30	5	94	96
3	TBAI	40	8	75	79
4	TBACl	30	5	81	86
5	TBAF	30	5	36	39
6	$TBAHSO_4$	30	5	42	44

[a]Isolated yield. [b]Thermal conditions at 100 °C. [c]Microwave conditions at 300 W.

Table 3. Comparison of Compound **1b** Synthesis in Solution *vs*. Solvent-free Conditions

Entry	Solvent	Time (min)		Yield (%)[a]	
		Δ[b]	MW[c]	Δ	MW
1	DMSO	720	20	45	58
2	DMF	720	20	38	46
3	HMPTA	720	25	32	34
4	*o*-Xylene	720	25	16	20
5	TBAB[d]	30	5	94	96

[a]Isolated yield. [b]Thermal conditions at 100 °C. [c]Microwave conditions at 300 W. [d]Molten TBAB homogenizes the reaction media.

Table 4. Michael Addition of Phthalimide and Saccharin to α,β-Unsaturated Esters in the Presence of TBAB and DABCO

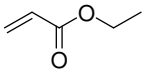
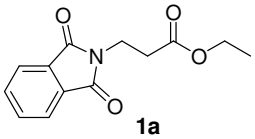
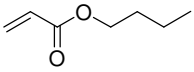
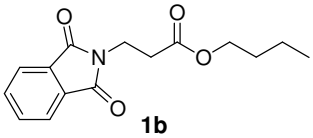
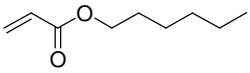
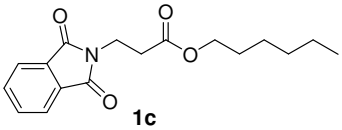
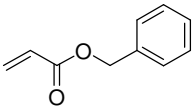
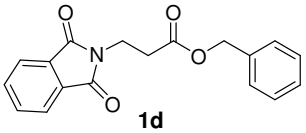
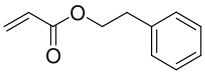
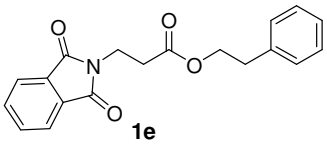
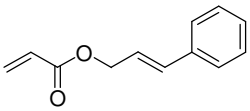
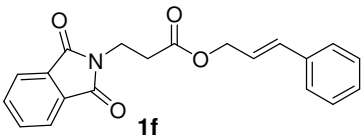
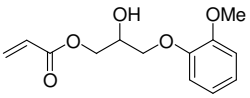
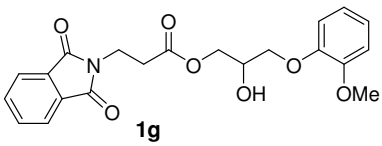
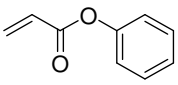
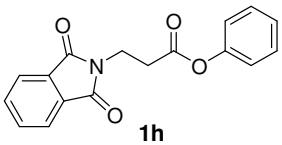
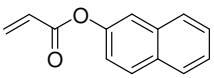
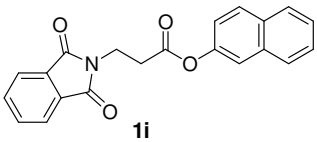
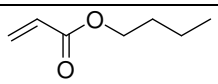
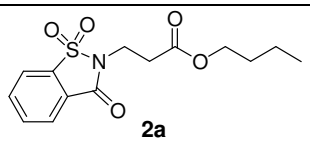
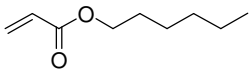
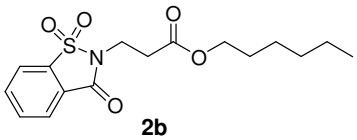
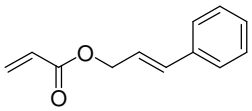
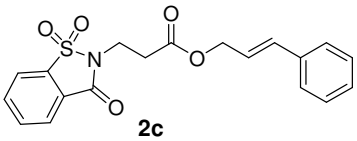
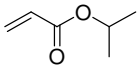
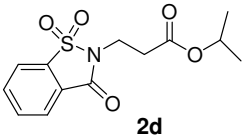
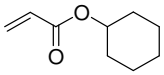
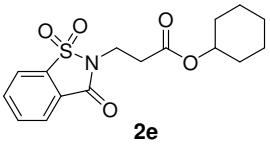
Entry	Ester	Product	Time (min)		Yield[a] (%)	
			Δ[b]	MW[c]	Δ	MW
1[d]		 **1a**	30	5	93	96
2		 **1b**	30	5	94	96
3		 **1c**	45	6	92	95
4		 **1d**	45	6	91	94
5		 **1e**	45	6	90	94
6		 **1f**	60	7	89	92
7		 **1g**	70	7	90	92
8		 **1h**	15	3	90	92
9		 **1i**	25	4	88	91

Table 4. Continued

10		 **2a**	600	1	45	31
11		 **2b**	720	1	43	30
12		 **2c**	720	1	45	30
13		 **2d**	660	1	43	28
14		 **2e**	720	1	42	29

[a]Isolated yield. [b]Thermal conditions at 100 °C. [c]Microwave conditions at 300 W. [d]In this reaction, under thermal conditions, the α,β-unsaturated ester/phthalimide (mol/mol) ratio was 2/1.

times were shorter in both thermal and microwave methods (Table 4, entries 8 and 9). In general, for the Michael addition of phthalimide to α,β-unsaturated esters, the microwave method was more efficient than the thermal method. However, for the Michael addition of saccharin, thermal conditions gave higher yields.

CONCLUSIONS

In conclusion, we have developed an efficient, rapid, and operationally simple method for the Michael addition of phthalimide to various α,β-unsaturated esters in solvent-free conditions. The method was not as effective for the addition of saccharin to α,β-unsaturated esters and the adducts were isolated in much lower yields than those resulting from the addition of phthalimide to various α,β-unsaturated esters.

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