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One-step Barton decarboxylation by micellar catalysis – application to the synthesis of maleimide derivatives

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F. Mangin,[a] E. Banaszak-Léonard,[a] C. Len[a,b]*

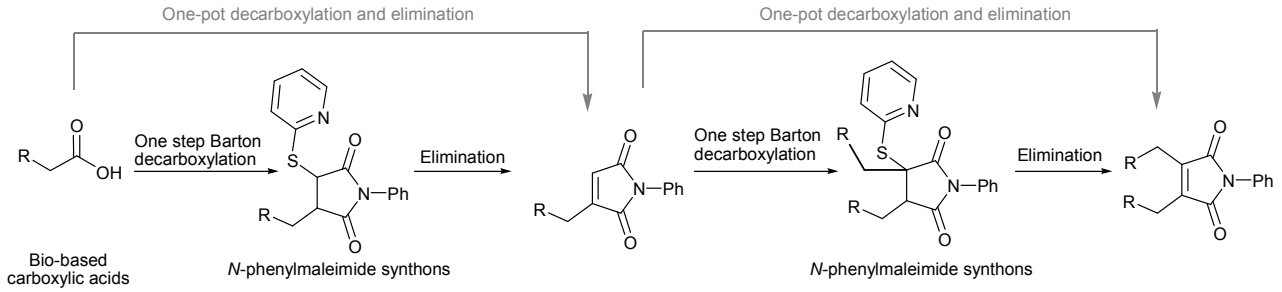
Maleimides being studied or used in various applications, for the first time, a facile entry to Barton decarboxylation in aqueous media is described to obtain in one step substituted *N*-phenylmaleimide synthons. The radicals, generated by the ultrasonic lysis of *N*-hydroxy-2-thiopyridone esters, reacted respectively with electron deficient olefin phenylmaleimide in a one-step radical addition. These esters were obtained from natural fatty acid derivatives including unsaturated ones. Various activating conditions (UV, sonication, heating, microwaves), reactants and solvent adjustment, as well as surfactants were tested to improve the yield of the reaction. One of the products obtained was then transformed into a new electron-trap monosubstituted maleimide and was able to react again to obtain a disubstituted *N*-phenylmaleimide derived from biomass.

Introduction

With the concept of Green Chemistry and its twelve principles,[1] organic chemists are strongly encouraged to reduce the environmental impact of their reaction in terms of media and energy. To reach this goal, new aspects of water influence on organic chemistry have been explored, and organic reactions in aqueous media have been readapted. To overcome the problem of organic reactant solubility in water, various strategies have been studied: the use of organic co-solvents[2] or ionic liquids[3] as well as additives like phase transfer agents,[4] cyclodextrins,[5–7] polymers[8] or surfactants.[9–12]

Among the main organic reactions, initial free radical two-step Barton decarboxylation and recent advances offer the possibility to create various structures.[13–32] It is noteworthy that solvent and especially water plays an important role in free radical chemistry.[33,34]

Maleimide derivatives can be considered as either interesting target compounds[35–40] or organic synthons/monomers.[41–50] Different pathways for substituted *N*-phenylmaleimide synthesis have been thus developed in the literature: starting from unsubstituted[40,47] or substituted[51] *N*-phenylmaleimides ; itaconic,[52] citraconic,[36,37] or dimethylmaleic anhydrides[39] ; phenylisocyanide[53,54] ; or phenyliminovinylidenphosphoran.[51]



Scheme 1 Present work.

However, these syntheses gave products with moderate yields and are usually performed in organic solvents. For the last 30 years, only few publications described the synthesis of substituted *N*-phenylmaleimides using a two-step Barton decarboxylation in organic solvent,[14,18,55,56] But surprisingly, radical Barton decarboxylation has never been performed in aqueous solution, and especially in one-step. In the present study, we propose the first one-step Barton decarboxylation in

[a] *Sorbonne Universités, Université de Technologie de Compiègne, Ecole Supérieure de Chimie Organique et Minérale, EA 4297 Transformations Intégrées de la Matière Renouvelable, Centre de Recherche Royallieu, CS 60319, F-60203 Compiègne cedex, France.*
Fax: +33 (0)3 44 97 15 91; Tel: +33 (0)3 44 23 88 28; E-mail: christophe.len@utc.fr
[b] *Department of Chemistry, University of Hull, Hull HU6 7RX, England*
† Footnotes relating to the title and/or authors should appear here.
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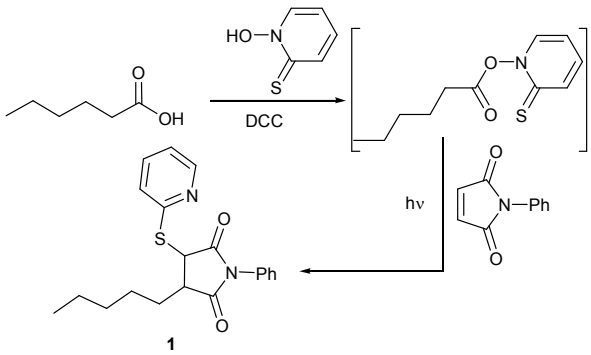
water. An easy access to maleimide derivatives from bio-based molecules is studied (Scheme 1).

Results and discussion

Physicochemical parameters implied the lack of reaction for acetic acid, propionic acid, butyric acid, or valeric acid. So we first adapted the general reaction mostly found in literature for Barton decarboxylation starting from caproic acid (hexanoic acid). Variations of the amounts of solvent, reactants and number of steps were also studied (Scheme 2, Table 1). Among the different organic solvents, dichloromethane (CH_2Cl_2) was selected and used with hexanoic acid (1.03 mmol), Dicyclohexylcarbodiimide (DCC, 1.2 equiv.) and 2-mercaptopyridine-*N*-oxide (1.2 equiv.). *N*-Phenylmaleimide as electron-trap was added in a second step (Method A) or directly at the beginning of the reaction (Method B). The one-step method B gave better results than the two-step one since time of reaction was divided by two (2 h *vs* 4 h) and yield was better (77% vs 58%, Table 1, entries 1 and 2). Moreover, decreasing the solvent volume and phenylmaleimide quantity did not influence dramatically the yield of the reaction (Table 1, entries 2 and 3).



Scheme 2 General pathway of the first Barton decarboxylation.

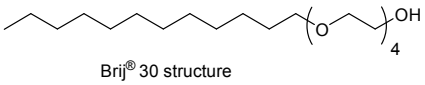
Table 1 Adjustment of the solvent, reactant quantities and number of steps for the synthesis of compound **1**[a]

Entry	Solvent quantity	Electron trap quantity	Method	Yields (%)[b]
1	15 mL	5 equiv.	A	58
2	15 mL	5 equiv.	B	77
3	5 mL	1.5 equiv.	B	72

[a] Reagents and conditions: Method A: protected from the light, caproic acid, DCC, 2-mercaptopyridine-*N*-oxide, CH_2Cl_2, rt, 2 h, then *N*-phenylmaleimide, hν, 2 h; Method B: without any protection from the light, one-step caproic acid, DCC, 2-mercaptopyridine-*N*-oxide, CH_2Cl_2, *N*-phenylmaleimide, hν, 2 h. [b] HPLC yields obtained *via* calibration curve with external standard.

In order to decrease the impact of organic solvent, substitution of dichloromethane by water as green solvent was studied. The same manipulation (Method B) with a complete substitution of CH_2Cl_2 by water (5 mL), *N*-phenylmaleimide (1.5 equiv.) was performed and gave only 12% of the titled product **1**. In order to increase the mass transfer of organic reagents in water media, Brij® 30 (HLB = 10, Table 2) was chosen for its medium Hydrophilic-Lipophilic-Balance and alternative technologies were developed. Starting with classical 1 wt% of surfactant in water (5 or 30 mL), four modes of activation were chosen: conventional heating at 35°C, UV irradiation (500 W), monomode microwave at 70°C and sonication (200 W) with an ultrasonic probe. It was noteworthy that the reaction time was determined by HPLC measurement when a maximal yield of titled product **1** was observed. In our hands, conventional heating and UV irradiation were conducted for 2h, and microwave and ultrasounic activations were performed for 20 min (Table 2). Two hours of conventional heating in 5 mL of micellar media (Table 2, entry 1) afforded a 34% yield of compound **1** leading to a really poor reproductibility. A poor dispersion of the reactants in the solution was observed. This observation was made, whatever the activation modes were (UV irradiation, microwave, sonication) when the solvent volume was 5 mL. Pouring starting materials in 30 mL of Brij® 30 solution led (Table 2, entry 2) to visually-seen good dispersion of the reagents into the media. However under conventional or microwave heating, yields obtained remained poor (15%). By UV irradiation with a 500 W vapour-mercury lamp, the reaction was performed at room temperature without increase of temperature yielding compound **1** in 30% yield. As expected, our best first result for the free radical Barton decarboxylation was obtained under sonication. In this case, maleimide derivative **1** was obtained in 60% yield. In contrast with UV irradiation, an ice-bath cooling was necessary to avoid the thermic degradation of Barton ester.

Table 2 Yields[a] of molecule **1** and activation mode comparison



Brij® 30 structure

Entry	1	2
Solvent Volume	5 mL	30 mL
Conventional Heating[b]	34%	15%
UV Irradiation[b]	35%	30%
Microwave Irradiation[c]	14%	15%
Sonication[c,d]	18%	60%

[a] Reagent and conditions: caproic acid (1.03 mmol), *N*-phenylmaleimide (1.5 equiv.), 2-mercaptopyridine-*N*-oxide (1.2 equiv.), DCC (1.2 equiv.), Brij® 30 (1 wt%), water, HPLC yielding *via* calibration curve with external standard. [b] 2 h of reaction. [c] 20 min of reaction. [d] ice bath cooling, 200W with an ultrasonic probe.

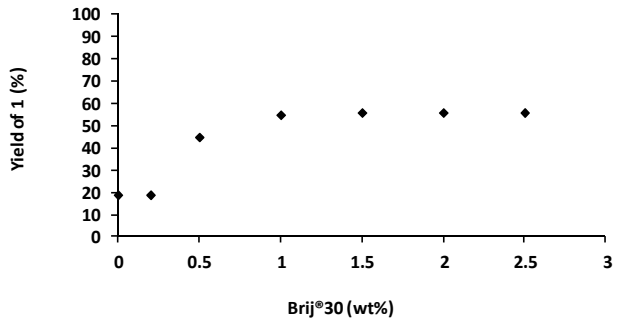


Fig. 1 Variation of the weight percentage of Brij® 30 for the synthesis of compound **1**.

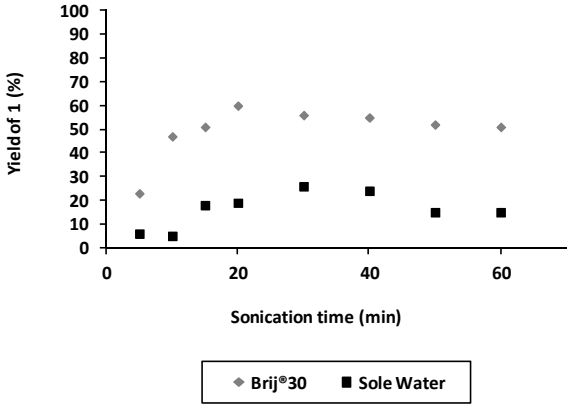


Fig. 2 Variation of the sonication time (Brij® 30, 1 wt%) and comparison to sole water for the synthesis of compound **1**.

The required quantity of Brij® 30 promoter was then investigated leading to a yielding curve (Fig. 1). As shown, up to 0.2 wt% of Brij® 30 surfactant, compound **1** was obtained in 19% yield. The effect of surfactant was optimal at 1 wt% and more Brij® 30 did not increase the yield obtained of maleimide derivative **1**. To control the contribution of the aqueous surfactant solution over sole water, ultrasonic timings were studied (Fig. 2). Unsurprisingly, whatever the sonication time, Brij®30 gave better results than sole water, as all the reactants were not soluble in non-micellar aqueous media. However, surfactant used in 1 wt% seemed to reach a maximum efficiency with 20 min of ultrasonic activation, still cooled with an ice bath.

In order to select the best promoter, variation of the media was studied (Table 3). In addition to the blank reaction performed in water (Table 3, entry 1), CH_2Cl_2 (Table 3, entry 2) gave only 3% yields of maleimide derivative **1**.

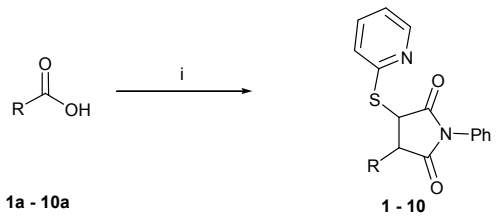
Phase-transfer agents such as tetrabutyl ammonium bromide (TBAB), 18-crown-6 ether and *β*-cyclodextrin, led to low yields from 8 to 23% (Table 3, entries 3-5). Ionic surfactants such as SDS, CTAB did not appear to be the best family for the Barton decarboxylation reaction as they only permitted to afford compound **1** in 11 and 13 % yields respectively (Table 3, entries 6 and 7). Concerning the PEG-based saturated Brij® 30 (HLB = 10), Brij® 700 (HLB = 18), Brij® 72 (HLB = 5), Brij® 76 (HLB = 12) or insaturated Brij® 98 (HLB = 15), the best results were obtained with Brij® 30 and Brij®98, leading to the conclusion that HLB was not the major influent parameter to take into account for the success of the reaction (Table 3, entries 8-12). Monoglyceride and sugar-based fatty esters such as Tween® 20 and Monomuls® afforded compound **1** in yields lower than 43% (Table 3, entries 13 and 14). Finally, aromatic ones (Triton™X, Igepal® CO family) led to the formation of maleimide derivative **1** with homogeneous moderate range yields (around 45%, Table 3, entries 15-18). In our hands, the surfactant Brij® 30 was chosen as the best one for our Barton decarboxylation model. Using our optimized reaction conditions (caproic acid (1.03 mmol), *N*-phenylmaleimide (1.5 equiv.), 2-mercaptopyridine-*N*-oxide (1.2 equiv.), DCC (1.2 equiv.), Brij® 30 (300 mg), water (30 mL), sonication for 20 min at 200 W, cooled with an ice bath), the scope of the reaction was then investigated (Table 4).

Table 3 Media condition variations for the synthesis of compound 1 [a]

Entry	Media	HLB	Yield (%)[b]
1	Water	-	19
2	CH_2Cl_2	-	3
3	TBAB	-	23
4	18-Crown-6	-	8
5	*β*-Cyclodextrin	-	13
6	CTAB	10	13
7	SDS	40	11
8	Brij® 30	10	60
9	Brij® 700	18	27
10	Brij® 72	5	16
11	Brij® 76	12	38
12	Brij® 98	15	55
13	Tween® 20	16	43
14	Monomuls®	4	28
15	Triton™X-100	12	42
16	Igepal® CO-520	10	48
17	Igepal® CO-630	13	47
18	Igepal® CO-720	14	42

[a] Reagent and conditions: (i) caproic acid (1.03 mmol), *N*-phenylmaleimide (1.5 equiv.), 2-mercaptopyridine-*N*-oxide (1.2 equiv.), DCC (1.2 equiv.), promoter (300 mg), water (30 mL), sonication 20 min at 200 W, ice bath. [b] HPLC yielding *via* calibration curve with external standard.

Table 4 Scope of the reaction



1: R = $CH_3(CH_2)_4$ **5**: R = $CH_3(CH_2)_{10}$
2: R = $CH_3(CH_2)_5$ **6**: R = $CH_3(CH_2)_{13}$
3: R = $CH_3(CH_2)_6$ **7**: R = $CH_3(CH_2)_{14}$
4: R = $CH_3(CH_2)_8$ **8**: R = $CH_3(CH_2)_{16}$

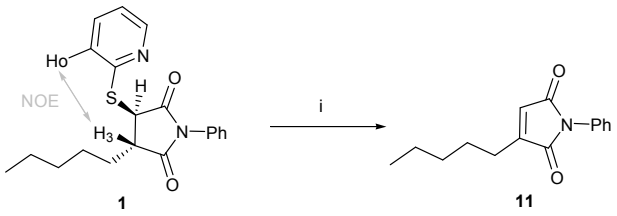
9: R = (*Z*) $CH_3(CH_2)_7CH{=}CH(CH_2)_7$
10: R = (*Z,Z*) $CH_3(CH_2)_4CH{=}CH\text{-}CH_2\text{-}CH{=}CH(CH_2)_7$

Entry	Acid	Product	Isolated Yields (%)
1	**1a**	**1**	60
2	**2a**	**2**	62
3	**3a**	**3**	58
4	**4a**	**4**	54
5	**5a**	**5**	57
6	**6a**	**6**	61
7	**7a**	**7**	66
8	**8a**	**8**	55
9	**9a**	**9**	60
10	**10a**	**10**	61

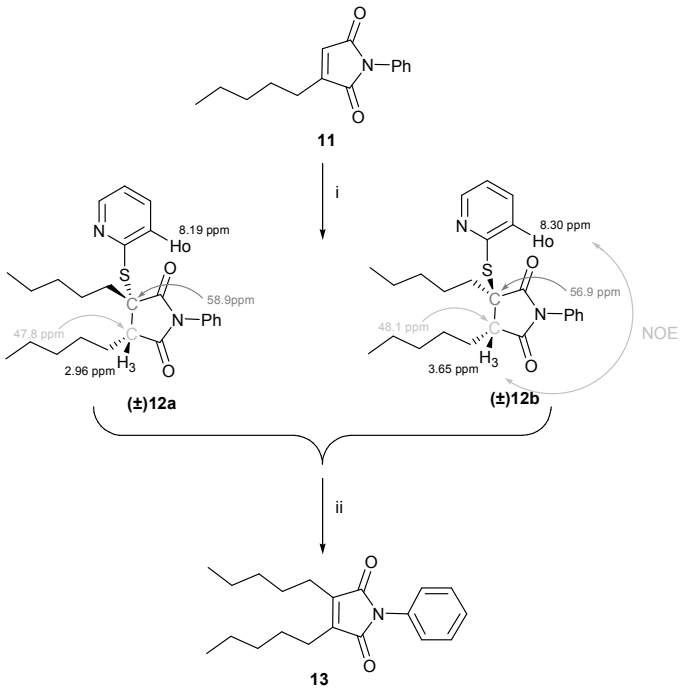
Reagents and conditions: (i) carboxylic acid (1.03 mmol), *N*-phenylmaleimide (1.5 equiv.), 2-mercaptopyridine-*N*-oxide (1.2 equiv.), DCC (1.2 equiv.), Brij® 30 (300 mg), water (30 mL), sonication for 20 min at 200 W, ice bath.

Natural saturated fatty acids (caproic acid (**1a**), caprylic acid (**3a**), capric acid (**4a**), lauric acid (**5a**), palmitic acid (**7a**) and stearic acid (**8a**)) gave their corresponding adduct with yields comprised between 54 and 66% (Table 4, entries 1, 3-5, 7 and 8). Natural unsaturated fatty acids (oleic acid (**9a**) and linoleic acid (**10a**)) were transformed into **9** and **10** in 60 and 61% yield respectively (Table 4, entries 9 and 10). Finally, free fatty acids having odd carbon atoms (C7 and C15): heptanoic acid (**2a**) and pentadecanoic acid (**6a**) gave similar yields of 62 and 61% (Table 4, entries 2 and 6). These results showed that the nature of the carboxylic acid (number of carbon atoms, unsaturation...) did not influence the Barton decarboxylation in our conditions.

It is noticeable that compounds **1-10** were formed with a good selectivity for the (±) *anti*-adduct. NOESY experiment was chosen to confirm this selectivity on compound **1** (Scheme 3) and the cross peak between Ho (8.29 ppm) and H_3 (3.23 ppm) indicated its *anti* configuration. Then, it is noticeable that a spontaneous elimination occurred during both the ^{1}H NMR operation, and on silica gel as previously mentioned in the literature,[57] and leading to a ^{1}H NMR peak corresponding to the double bond thus formed (See Supporting Information). In order to complete the elimination of the thiopyridine synthon, the molecule **1** was transformed into its oxidized form (sulfoxide). Conventional thermal elimination led to compound **11** in quantitative yield (scheme 3). Then, the same reaction than presented previously was carried out, excepted than the electon-trap **11** was both the starting material and the limiting reactant. Starting from maleimide derivative **11**, a mixture of two diastereoisomers (±)**12a** and (±)**12b** (plus their corresponding enantiomers) was obtained in 47% yield (Scheme 4).



Scheme 3 Elimination step. Reagent and conditions: (i) **1** (1 mmol), *m*-CPBA (10 equiv.), CH_2Cl_2 (10 mL), for 10 min at 0°C, then toluene (20 mL) under reflux for 1 h



Scheme 4 Second Barton reaction and elimination step. Reagents and conditions: (i) **11** (1 mmol), caproic acid (1.5 equiv.), 2-mercaptopyridine-*N*-oxide (1.8 equiv.), DCC (1.8 equiv.), Brij® 30 (300 mg), water (30 mL), sonication 20 min at 200 W, ice bath ; (ii) (±)**12a** and (±)**12b** (1 mmol), *m*-CPBA (10 equiv.), CH_2Cl_2 (10 mL), for 10 min at 0°C, then toluene (20 mL) under reflux for 1 h.

In contrast with the good stereoselectivity for the synthesis of **1-10**, diastereoisomers (**±**)**12a** and (**±**)**12b** were clearly seen on NMR. For (**±**)**12a** and (**±**)**12b**, the unique C*H*CO (H_3) was seen as 2 triplets (3.65 and 2.96 ppm) with integrations of 0.4 and 0.6 respectively, correlated by HSQC experiment to their corresponding carbon (47.8 and 48.1 ppm), and by HMBC experiment to their corresponding α-quaternary carbons (56.9 and 58.9 ppm). Between the two diastereoisomers (**±**)**12a** and (**±**)**12b**, a significant variation of the *ortho*-proton chemical shift (Ho) was observed (8.19 ppm and 8.30 ppm respectively). The cross peak (NOESY experiment) between Ho (8.30 ppm) and H_3 (3.65 ppm) indicated the *anti*-configuration for (**±**)**12b**. However, the spontaneous *syn*-elimination that occurred during both purification and NMR led to the integration proportions observed. Then, to complete the elimination step, conventional sulphide oxidation followed by sulfoxide moiety elimination afforded compound **13** in quantitative yield.

Conclusions

First one-step Barton decarboxylation was performed in a Brij® 30 micellar media, under ultrasonic probe activation. The radicals formed by the lyses of *N*-hydroxy-2-thiopyridone esters from natural fatty acid derivatives were trapped by a designed easy-to-use *N*-phenylmaleimide to afford the corresponding functionalized products. The facile synthesis mild conditions of the reaction described herein offer a rapid synthetic access to mono- and disubstituted *N*-phenylmaleimide derived from biomass.

Acknowledgements

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