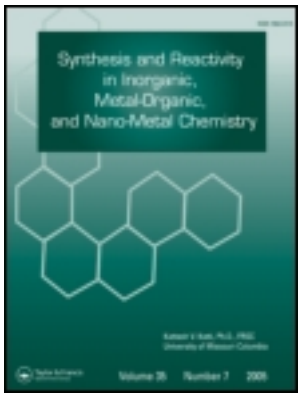

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Rapid Sulfonyloxylactonization of Alkenoic Acids Under Microwave and Ultrasound Irradiation

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Rapid Sulfonyloxylactonization of Alkenoic Acids Under Microwave and Ultrasound Irradiation

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Fast and convenient reactions were investigated under microwave and ultrasound irradiation, providing simple methods for sulfonyloxylactonization of alkenoic acids in good yields in a short time.

Keywords hypervalent iodine reagent, microwave irradiation, sulfonyloxylactonization, ultrasound irradiation

INTRODUCTION

Lactonizations have been studied extensively, and this type of transformation serves as an important key reaction in a variety of syntheses.[1–3] Among them, halolactonization and phenylselenolactonization are general used methods.[4–6] Recently, organic hypervalent iodine reagents have found broad application in organic chemistry and frequent use in synthesis due to their chemical properties and reactivity being similar to those of H (II), Tl(III), and Pb(IV), but without the toxic and environmental problems of these heavy metal congeners.[7–15] Koser et al. (1986) first reported the tosyloxylactonization of alkenoic acids with hypervalent iodine reagent, [hydroxyl(tosyloxy)iodo]benzene (HTIB, Koser's reagent), in which the mechanism was different with those of halolactonization and phenylselenolactonization.[16] The ability of HTIB to introduce the tosylate ligand into alkenoic acids prompted us to investigate the camphorsulfonyloxylactonization of alkenoic acids, and a series of new 5-camphorsulfonyloxy-4-pentanolactones and 6-camphorsulfonyloxy-5-hexanolactone was synthesized with the analogous reagent, [hydroxyl (((+)-10-camphorsulfonyl)oxy)iodo]benzene.[17] The catalytic sulfonyloxylactonization of alkenoic acids using hypervalent iodine catalyst was also just reported, which extended the scope of sulfonyloxylactonization.[18] Due to many hypervalent iodine reagents having low solubility in most organic solvents, the development of solvent-free reactions and other special reactions is a big step forward and should lead to an increasing use of this chemistry.

Microwave-assisted organic syntheses have received great attention because of their fast reaction rates, high purity of products, and ease of manipulation.[19,20] In particular, the microwave-irradiated procedures in water medium or in the absence of solvents for organic synthesis have attracted considerable interest in recent years due to their efficient and environmentally benign conditions.[21–23] Our recent interest has been in the development of new synthetic methods using hypervalent iodine reagents. As part of a program, our group found that some hypervalent iodine reagents, such as Koser's reagent, can be transformed from (diacetoxyiodo)benzene by a microwave-promoted solvent-free ligand exchange reaction.[24] In order to extend the scope of microwave-promoted reaction, we have checked the sulfonyloxylactonization of alkenoic acids under microwave irradiation, and we also have investigated the same reaction under ultrasound irradiation. We report here the novel and rapid microwave- and ultrasound-promoted sulfonyloxylactonization of alkenoic acids.

EXPERIMENTAL

Melting points were determined on a digital melting-point apparatus and were not corrected. Infrared (IR) spectra were recorded on a Thermo-Nicolet 6700 instrument, nuclear magnetic resonance (NMR) spectra were measured on a Bruker ANANCE III(500-MHz) spectrometer, and mass spectra were determined on a Thermo-ITQ 1100 mass spectrometer. Microwave irradiation was carried out with an LWMC-201 microwave reactor at full power (650 W) (Nanjing, China). Ultrasound irradiation was carried out with an ultrasonic cleaning bath (50 kHz). Alkenoic acids, (diacetoxyiodo)benzene, *p*-toluenesulfonic acid monohydrate, and (+)-10-camphorsulfonic acid are commercially available.

Typical Procedure for Sulfonyloxylactonization of Alkenoic Acids Under Microwave Irradiation

4-Pentenoic acid **1a** (50 mg, 0.5 mmol, 1.0 equiv), (diacetoxyiodo)benzene (161 mg, 0.5 mmol, 1.0 equiv), and

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p-toluenesulfonic acid monohydrate **2a** (114 mg, 0.6 mmol, 1.2 equiv) were mixed in a 10-mL glass tube. The mixture tube was placed inside in an alumina bath and irradiated for 40 s in a microwave reactor at full power (650 W). After cooling, the solid residue was then separated on a silica gel plate using 3:1 hexane–ethyl acetate as eluant to give 115 mg of 5-tosyloxy-4-pentanolactone **3a** (85% yield).

Typical Procedure for Sulfonyloxylactonization of Alkenoic Acids Under Ultrasound Irradiation

4-Pentenoic acid **1a** (50 mg, 0.5 mmol, 1.0 equiv), (diacetoxyiodo)benzene (161 mg, 0.5 mmol, 1.0 equiv), *p*-toluenesulfonic acid monohydrate **2a** (95 mg, 0.5 mmol, 1.0 equiv), and 2 mL tetrahydrofuran (THF) were placed in a glass flask and irradiated for 2 min in an ultrasonic cleaning bath (50 KHz). The mixture was then separated on a silica gel plate using 3:1 hexane–ethyl acetate as eluant to give 117 mg of 5-tosyloxy-4-pentanolactone **3a** (86% yield).

3a: M.p. 79–81°C (Lit.[16] 80.5–81.5°C). ^{1}H-NMR (500 MHz, $CDCl_3$): 7.78 (d, J = 8.5 Hz, 2H), 7.37 (d, J = 8.5 Hz, 2H), 4.72–4.65 (m, 1H), 4.19 (dd, J = 11.0, 3.0 Hz, 1H), 4.13 (dd, J = 11.0, 4.0 Hz, 1H), 2.61–2.48 (m, 2H), 2.46 (s, 3H), 2.39–2.31 (m, 1H), 2.15–2.10 (m, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): 176.0, 145.4, 132.2, 130.0, 127.9, 76.4, 70.0, 27.8, 23.5, 21.6. MS (EI, *m/z*,%): 271 (M+1$^+$, 100).

3b: M.p. 91–95°C (Lit. [16] 92–95°C). ^{1}H-NMR (500 MHz, $CDCl_3$): 7.77 (d, J = 8.5 Hz, 2H), 7.37 (d, J = 8.0Hz, 2H), 4.68–4.62 (**3b$_1$**) and 4.58–4.52 (**3b$_2$**) (m, 1H), 4.21(dd, J = 11.0, 3.5 Hz, 0.73 H), 4.14–4.09 (m, 1.27 H), 2.77–2.66 (m, 1H), 2.51–2.47 (**3b$_1$**) and 2.41–2.35 (**3b$_2$**) (m, 1H), 2.46 (s, 3H), 2.06–2.00 (**3b$_1$**) and 1.74–1.66 (**3b$_2$**) (m, 1H), 1.27 (d, J = 7.0Hz, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): 178.2, 145.4 (d, J = 12.5 Hz), 132.3, 130.0 (d, J = 6.3 Hz), 127.9 (d, J = 6.3 Hz), 74.5, 74.1, 70.2, 69.4, 35.0, 33.6, 32.1, 31.7, 21.6, 16.0, 15.0. MS (EI, *m/z*,%): 285 (M+1$^+$, 100).

3c: Oil (Lit.[25]). ^{1}H-NMR (500 MHz, $CDCl_3$): 7.79 (d, J = 8.0 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 4.61–4.57 (**3c$_1$**) and 4.17–4.13 (**3c$_2$**) (m, 1H), 4.22–4.17 (m, 2H), 2.81–2.71 (m, 1H), 2.61 (dd, J = 17.5, 8.5 Hz) (**3c$_1$**) and 2.52–2.45 (m) (**3c$_2$**) (1H), 2.46 (s, 3H), 2.28 (dd, J = 17.5, 8.0 Hz) (**3c$_1$**) and 2.18 (dd, J = 17.5, 8.0 Hz) (**3c$_2$**) (1H), 1.17 (d, J = 6.5 Hz) (**3c$_1$**) and 1.11 (d, J = 6.5 Hz) (**3c$_2$**) (3H). ^{13}C-NMR (125 MHz, $CDCl_3$): 175.6, 175.2, 145.4 (d, J = 6.3 Hz), 132.1 (d, J = 23.8 Hz), 130.1 (d, J = 22.5 Hz), 127.9 (d, J = 2.5 Hz), 82.9, 78.8, 68.6, 67.7, 36.2 (d, J = 23.8 Hz), 31.8 (d, J = 25.0 Hz), 21.6, 17.0, 13.5. MS (EI, *m/z*,%): 285 (M+1$^+$, 100).

3d: M.p. 77–79°C (Lit. [16] 78–79°C). ^{1}H-NMR (500 MHz, $CDCl_3$): 7.81–7.78 (m, 2H), 7.38–7.35 (m, 2H), 4.52–4.48 (m, 1H), 4.15–4.09 (m, 2H), 2.60–2.55 (m, 1H), 2.47 (s, 3H), 2.46–2.43 (m, 1H), 2.00–1.94 (m, 1H), 1.88–1.82 (m, 1H), 1.72–1.65 (m, 1H). ^{13}C-NMR (125 MHz, $CDCl_3$): 170.1, 145.3, 132.3, 130.0, 128.0 (d, J = 6.3 Hz), 77.0, 70.1, 29.4, 24.0, 21.7, 18.1. MS (EI, *m/z*,%): 285 (M+1$^+$, 100).

3e: Oil (Lit.[17]). ^{1}H-NMR (500 MHz, $CDCl_3$): 4.82–4.79 (m, 1H), 4.51 (ddd, J = 22.0, 11.5, 3.0 Hz, 1H), 4.37 (ddd, J = 21.5, 11.0, 4.5 Hz, 1H), 3.62 (dd, J = 15.5, 6.0 Hz, 1H), 3.08 (dd, J = 15.0, 4.0 Hz, 1H), 2.69–2.52 (m, 2H), 2.43–2.37 (m, 3H), 2.20–2.02 (m, 3H), 1.97 (d, J = 17.5 Hz, 1H), 1.75–1.65 (m, 1H), 1.51–1.45 (m, 1H), 1.10 (s, 3H), 0.88 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): 214.2, 176.1, 76.7 (d, J = 7.5 Hz), 70.1, 69.9, 57.8 (d, J = 2.5 Hz), 48.1, 47.2, 42.6 (d, J = 2.5 Hz), 42.4 (d, J = 1.3 Hz), 27.9, 26.8, 24.8 (d, J = 7.5 Hz), 23.3 (d, J = 8.8 Hz), 19.5 (t, J = 2.5 Hz). IR (film): ν = 2963, 1781, 1746, 1456, 1418, 1360, 1282, 1167, 1070, 962 cm^{-1}. MS (EI, *m/z*,%): 331 (M+1$^+$, 100). HRMS: $C_{15}H_{22}O_6S$ calcd.: 330.1137, found: 330.1125.

3f: Oil (Lit.[17]). ^{1}H-NMR (500 MHz, $CDCl_3$): 4.79–4.72 (**3h$_1$**) and 4.68–4.62 (**3h$_2$**) (m, 1H), 4.51 (ddd, J = 19.0, 11.5, 3.0 Hz, 1H), 4.37–4.31 (m, 1H), 3.66–3.59 (m, 1H), 3.09–3.03 (m, 1H), 2.83–2.78 (**3h$_1$**) and 2.77–2.70 (**3h$_2$**) (m, 1H), 2.56–2.49 (m, 1H), 2.46–2.37 (m, 2H), 2.16–2.13 (m, 1H), 2.10–2.03 (m, 1H), 1.97 (d, J = 18.0 Hz, 1H), 1.79–1.68 (m, 2H), 1.50–1.44 (m, 1H), 1.31 (d, J = 5.5 Hz, **3h$_1$**) and 1.30 (d, J = 6.0 Hz, **3h$_2$**) (d, 3H), 1.10 (**3h$_1$**) and 1.09 (**3h$_2$**) (s, 3H), 0.89 (**3h$_1$**) and 0.88 (**3h$_2$**) (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): 214.4, 214.3, 179.2, 178.3, 74.9, 74.5 (d, J = 7.5 Hz), 70.5, 70.4, 69.7, 69.4, 57.9 (d, J = 3.8 Hz), 48.2 (d, J = 3.8 Hz), 47.4, 47.3 (d, J = 6.3 Hz), 42.7, 42.5 (d, J = 2.5 Hz), 35.2, 33.7, 32.2 (d, J = 8.8 Hz), 31.6 (d, J = 6.3 Hz), 26.9, 24.9 (t, J = 3.8 Hz), 19.6 (d, J = 5.0 Hz), 16.1, 15.1 (d, J = 3.8 Hz). IR (film): ν = 2966, 1775, 1747, 1456, 1360, 1286, 1168, 1069, 972, 931 cm^{-1}. MS (EI, *m/z*,%): 345 (M+1$^+$, 48), 109 (100). HRMS: $C_{16}H_{24}O_6S$ calcd.: 344.1294, found: 344.1289

3g: Oil (Lit.[17]). ^{1}H-NMR (500 MHz, $CDCl_3$): 4.75–4.30 (m, 3H), 3.63 (dd, J = 15.0, 4.5 Hz, 1H), 3.07 (dd, J = 15.5, 4.0 Hz, 1H), 2.85–2.78 (m, 1H), 2.72–2.66 (**3i$_1$**) and 2.56–2.49 (**3i$_2$**) (m, 1H), 2.45–2.22 (m, 3H), 2.15–2.13 (m, 1H), 2.11–2.02 (m, 1H), 1.97 (d, J = 18.5 Hz, 1H), 1.75–1.65 (m, 1H), 1.51–1.44 (m, 1H), 1.23 (dd, J = 6.5, 3.0 Hz, **3i$_1$**) and 1.17 (d, J = 7.5 Hz, **3i$_2$**) (3H), 1.10 (s, 3H), 0.88 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): 214.3, 175.7(d, J = 2.5 Hz), 175.4, 83.3 (d, J = 2.5 Hz), 79.2 (d, J = 5.0 Hz), 68.8, 68.6, 68.2, 68.0, 57.9 (d, J = 3.8 Hz), 48.2, 47.4 (d, J = 3.8 Hz), 47.2 (d, J = 2.5 Hz), 42.7 (d, J = 3.8 Hz), 42.5 (d, J = 2.5 Hz), 36.4 (d, J = 3.8 Hz), 36.2, 31.9, 31.8, 31.7, 26.9, 24.9 (d, J = 2.5 Hz), 24.8, 19.6 (d, J = 3.8 Hz), 18.0, 13.5. IR (film): ν = 2965, 1785, 1747, 1456, 1418, 1361, 1283, 1214, 1166, 1054, 975, 933 cm^{-1}. MS (EI, *m/z*,%): 345 (M+1$^+$, 61), 109 (100). HRMS: $C_{16}H_{24}O_6S$ calcd.: 344.1294, found: 344.1277.

3h: Oil (Lit.[17]). ^{1}H-NMR (500 MHz, $CDCl_3$): 4.73–4.69 (m, 1H), 4.51 (ddd, J = 20.0, 11.5, 3.5 Hz, 1H), 4.33 (ddd, J = 19.5, 11.5, 5.5 Hz, 1H), 3.63 (dd, J = 15.5, 8.0 Hz, 1H), 3.08 (d, J = 15.0 Hz, 1H), 2.45–2.37 (m, 2H), 2.20–2.13 (m, 2H), 2.10–2.01 (m, 1H), 1.99–1.95 (m, 2H), 1.75–1.67 (m, 1H), 1.51–1.45 (m, 1H), 1.32 (s, 3H), 1.30 (s, 3H), 1.10 (s, 3H), 0.89 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): 214.3 (d, J = 8.8 Hz), 180.8, 73.6 (d, J = 3.8 Hz), 69.9, 69.6, 57.9, 48.2, 47.4 (d, J =

10.0 Hz), 42.7, 42.5 (d, $J = 2.5$ Hz), 40.0 (d, $J = 2.5$ Hz), 38.4 (d, $J = 7.5$ Hz), 26.9, 24.9 (d, $J = 3.8$ Hz), 24.8 (d, $J = 3.8$ Hz), 24.7 (d, $J = 2.5$ Hz), 19.6. IR (film): $\nu = 2967$, 1775, 1747, 1457, 1361, 1280, 1207, 1169, 1130, 1067, 983, 926 cm^{-1}. MS (EI, *m/z*,%): 359 ($M+1^+$, 25), 109 (100). HRMS: $C_{17}H_{26}O_6S$ calcd.: 358.1451, found: 358.1441.

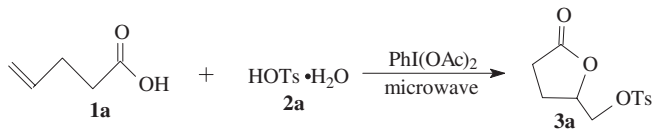
3i: Oil (Lit.[17]). ^{1}H-NMR (500 MHz, $CDCl_3$): 4.65–4.55 (m, 1H), 4.45–4.34 (m, 2H), 3.64 (dd, $J = 15.0$, 8.5 Hz, 1H), 3.09 (d, $J = 15.0$ Hz, 1H), 2.65–2.60 (m, 1H), 2.52–2.35 (m, 3H), 2.15–1.85 (m, 6H), 1.80–1.70 (m, 2H), 1.51–1.42 (m, 1H), 1.10 (s, 3H), 0.89 (s, 3H). ^{13}C-NMR (125 MHz, $CDCl_3$): 214.4, 170.1, 70.4, 70.2, 57.9, 48.2 (d, $J = 2.5$ Hz), 47.4 (d, $J = 2.5$ Hz), 42.7 (d, $J = 3.8$ Hz), 42.5, 29.5, 26.9, 24.9 (d, $J = 11.3$ Hz), 23.9 (d, $J = 6.3$ Hz), 19.6, 18.2. IR (KBr): $\nu = 2961$, 1744, 1456, 1360, 1240, 1169, 1081, 1054, 963 cm^{-1}. MS (EI, *m/z*,%): 345 ($M+1^+$, 34), 109 (100). HRMS: $C_{16}H_{24}O_6S$ calcd: 344.1294, found: 344.1288.

RESULTS AND DISCUSSION

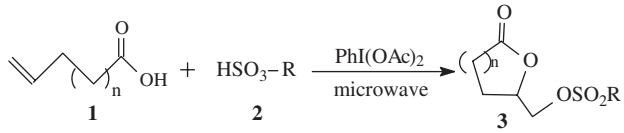
At the beginning, we mixed equal equivalents of 4-pentenoic acid (**1a**), (diacetoxyiodo)benzene (DIB), and *p*-toluenesulfonic acid monohydrate ($HOTs{\cdot}H_2O$) (**2a**) to examine the sulfonyloxylactonization of 4-pentenoic acid. We found that when the mixture was heated in a glass tube by a microwave irradiation for only 20 s, the sulfonyloxylactonization was nearly complete and the desired product of 5-tosyloxy-4-pentanolactone (**3a**) was afforded in 78% of yield (Scheme 1). Then a series of experiments was performed on the reaction of 4-pentenoic acid with DIB and $HOTs{\cdot}H_2O$ to determine the optimum reaction conditions. It was found when one equivalent of 4-pentenoic acid and DIB with 1.2 equivalents of $HOTs{\cdot}H_2O$ was heated in a microwave reactor for 40 s, the highest yield of 85% was reached. Under the optimum reaction conditions, the sulfonyloxylactonization of alkenoic acids (**1**) with *p*-toluenesulfonic acid monohydrate (**2a**) or (+)-10-camphorsulfonic acid (**2b**) was investigated (Scheme 2); the good results are summarized in Table 1.

It is shown from Table 1 that all 4-pentenoic acids reacted with DIB and **2a** or **2b** rapidly, and gave the corresponding 5-sulfonyloxy-4-pentanolactones in good to excellent yields (entries **1–3**, **5–8**). Among them, **3b**, **3c**, **3f**, and **3g** were found to be the mixtures of diastereomers by ^{1}H-nuclear magnetic resonance (NMR) technique. Similar treatment of 5-hexenoic acid with **2a** or **2b** provided 6-sulfonyloxy-5-hexanolactones (**3d**, **3i**) in middle yields (entries **4**, **9**), which meant that the five-membered lactone ring was formed more easily than the six-membered lactone ring in the sulfonyloxylactonization. We also checked the reactions of 3-butenoic acid and *trans*-3-hexenoic acid with **2a** in the same reaction conditions; the products were not the desired sulfonyloxylactones, and two corresponding unsaturated lactones were obtained.

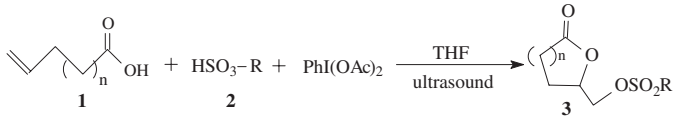
The sonochemical reaction was carried out in a thermostatted (20°C) ultrasonic cleaning bath of frequency 50 kHz in air. It was found that when the mixture of equal equivalents



SCH. 1.



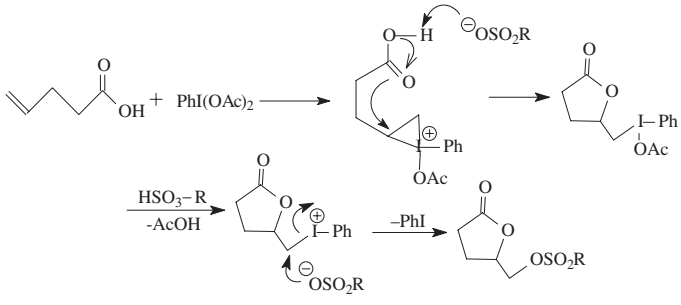
SCH. 2.



SCH. 3.

of 4-pentenoic acid (**1a**), (diacetoxyiodo)benzene (DIB), and *p*-toluenesulfonic acid monohydrate ($HOTs{\cdot}H_2O$) (**2a**) in THF was irradiated under ultrasound only 2 min, an 86% yield of 5-tosyloxy-4-pentanolactone (**3a**) was afforded. After several reactions, the optimal reaction conditions were determined: The suitable equivalent ratio for **2a**, DIB, and $HOTs{\cdot}H_2O$ was 1:1:1, THF was the most preferred solvent, and in only 2 min of ultrasound irradiation all reactions were complete. Under the optimal reaction conditions, the sulfonyloxylactonization of alkenoic acids was investigated (Scheme 3); the good results are summarized in Table 2.

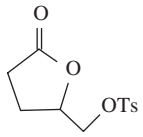
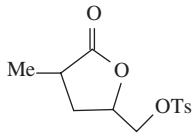
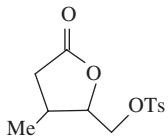
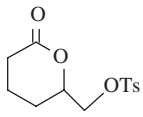
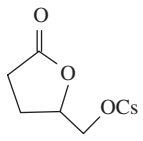
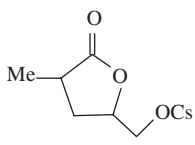
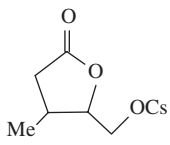
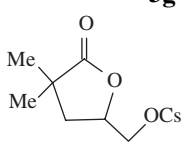
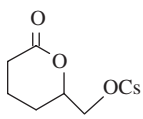
Table 2 shows that good to excellent yields of five-membered sulfonyloxylactones were obtained for a series of 4-pentenoic acids (entries **1–3**, **5–8**). The yields of six-membered 6-sulfonyloxy-5-hexanolactones were increased to 77% and 73% for **3d** and **3i**, respectively (entries **4**, **9**) compared with Table 1, which meant that ultrasound irradiation favoured the synthesis of sulfonyloxylactones.



SCH. 4.

'The plausible mechanism is similar to the literature procedure,[16] which included the electrophilic addition of hypervalent

TABLE 1
The sulfonylactonization of alkenoic acids under microwave irradiation

Entry	Alkenoic acid (1)	Sulfonic acid (2)	Sulfonyloxylactone (3)[a]	Yield (%)[b]
1	$CH_2=CH(CH_2)_2CO_2H$ **1a**	*p*-Tosylic acid monohydrate **2a**	 **3a**	85
2	$CH_2=CHCH_2CH(Me)CO_2H$ **1b**	**2a**	 **3b**	86
3	$CH_2=CHCH(Me)CH_2CO_2H$ **1c**	**2a**	 **3c**	80
4	$CH_2=CH(CH_2)_3CO_2H$ **1d**	**2a**	 **3d**	63
5	**1a**	(+)-10-Camphorsulfonic acid **2b**	 **3e**	81
6	**1b**	**2b**	 **3f**	83
7	**1c**	**2b**	 **3g**	74
8	$CH_2=CHCH_2C(Me)(Me)CO_2H$ **1e**	**2b**	 **3h**	84
9	**1d**	**2b**	 **3i**	56

[a]Ts, *p*-Me-$C_6H_4SO_2$; Cs, (+)-10-camphorylsulfonyl.
[b]Isolated yield.

TABLE 2
The sulfonylactonization of alkenoic acids under ultrasound irradiation

Entry	Alkenoic acid (1)	Sulfonic acid (2)	Sulfonyloxylactone (3)[a]	Yield (%)[b]
1	**1a**	**2a**	**3a**	86
2	**1b**	**2a**	**3b**	88
3	**1c**	**2a**	**3c**	85
4	**1d**	**2a**	**3d**	77
5	**1a**	**2b**	**3e**	83
6	**1b**	**2b**	**3f**	85
7	**1c**	**2b**	**3g**	80
8	**1e**	**2b**	**3h**	85
9	**1d**	**2b**	**3i**	73

[a]Ts, *p*-Me-$C_6H_4SO_2$; Cs, (+)-10-camphorylsulfonyl.
[b]Isolated yield.

iodine reagent DIB on the double bond, then an intramolecular nucleophilic displacement, followed by another nucleophilic displacement to give the sulfonyloxylactone (Scheme 4).

In summary, rapid and convenient methods for sulfonyloxylactonization of alkenoic acids are afforded by the microwave- and ultrasound-promoted reactions. They are simple, fast, and afford good yields for synthesis of sulfonyloxylactones.

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