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Tetrahedron

Tetrahedron 62 (2006) 4769–4773

Electrooxidation of alcohols in an *N*-oxyl-immobilized rigid network polymer particles/water disperse system

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Received 17 February 2006; revised 6 March 2006; accepted 7 March 2006
Available online 27 March 2006

Abstract—The electrooxidation of alcohols in an aqueous disperse system with *N*-oxyl-immobilized poly(*p*-phenylene benzobisthiazole) network polymer particles (*PBZT$_{NT}$-N-Oxyl*) as a disperse phase was performed successfully in a simple beaker-type undivided cell under a constant current condition to afford the corresponding ketones, aldehydes, and/or carboxylic acid in moderate to good yields. Recycle use of both the *PBZT$_{NT}$-N-Oxyl* particles and the aqueous media could be achieved successfully by immobilization of additional *N*-oxyl moiety on the polymer particles in an appropriate interval. Notably, the shape and the particle size of *PBZT$_{NT}$-N-Oxyl* were not appreciably changed even after 60 times recycle use.

1. Introduction

Electrooxidation of alcohols mediated with *N*-oxyl compounds has been intensively investigated as a prominent tool for organic synthesis. The *N*-oxyl-mediated electrooxidation has been mainly carried out in polar organic solvents, such as acetonitrile or dichloromethane, containing rather high concentration of supporting electrolytes under a regulated potential condition in a divided cell.[1] The procedure is not necessarily satisfactory in terms of operational simplicity, manufacturing cost, and environmental stress. Torii and co-workers have developed an organic/aqueous two-phase system,[2] e.g., CH_2Cl_2/H_2O, in which the electrooxidation of alcohols is performed successfully by use of a simple beaker-type undivided cell under a constant current condition; hence, the operations are remarkably simple. However, there still remain serious problems in terms of environmental stress arising from the use of the harmful organic solvents, e.g., CH_2Cl_2.

In the last decade, various kinds of solid-supported reagents and catalysts have been developed for the oxidation of alcohols.[3] For example, *N*-oxyl-immobilized polymer particles[4] and silica gel[5] were prepared and employed as solid-supported catalysts. In this concern, we developed the electrooxidation of alcohols in a disperse system with *N*-oxyl-immobilized polymer particles, e.g., polyethylene[6] and poly(ethylene-*co*-acrylic acid),[7] or silica gel[8] as a disperse phase and an aqueous 20 wt % NaBr/satd $NaHCO_3$ solution as a disperse medium, in which most of alcohols would be adsorbed on the disperse phase and oxidize on the water/solid interface. Recycle use of the disperse phase and the aqueous disperse media was also investigated to offer a formally closed oxidation system. The disperse phases, e.g., polyethylene and poly(ethylene-*co*-acrylic acid) particles, and silica gel, however, suffered gradual degradation during the course of the recycle use. After repeating the use of the *N*-oxyl-immobilized polymer particles for several times, significant degradation into fine pieces brought about difficulty in the separation of the solid particles and the aqueous disperse media. In a practical sense, therefore, mechanically more tough solid particles (disperse phase) are desirable. In our continuing studies, we investigated the use of poly(*p*-phenylene benzobisthiazole) network polymer (PBZT$_{NT}$,[9] Fig. 1) as the disperse phase. This network polymer is constituted with

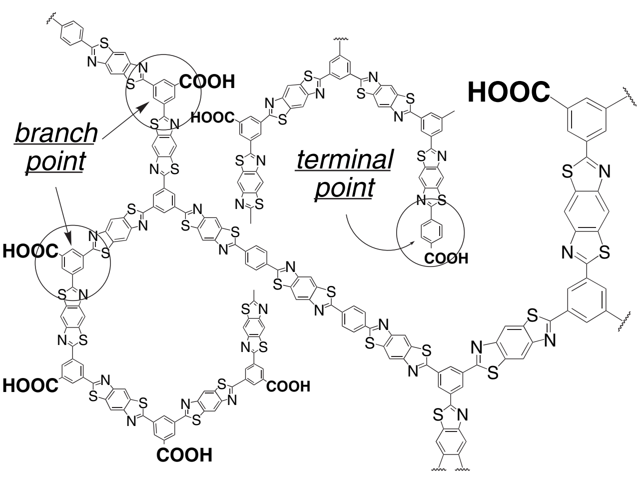

Figure 1. Structure of PBZT$_{NT}$.

Keywords: Electrooxidation; Alcohols; Polymer-supported *N*-oxyl; Disperse system; Aqueous solution.

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doi:10.1016/j.tet.2006.03.020

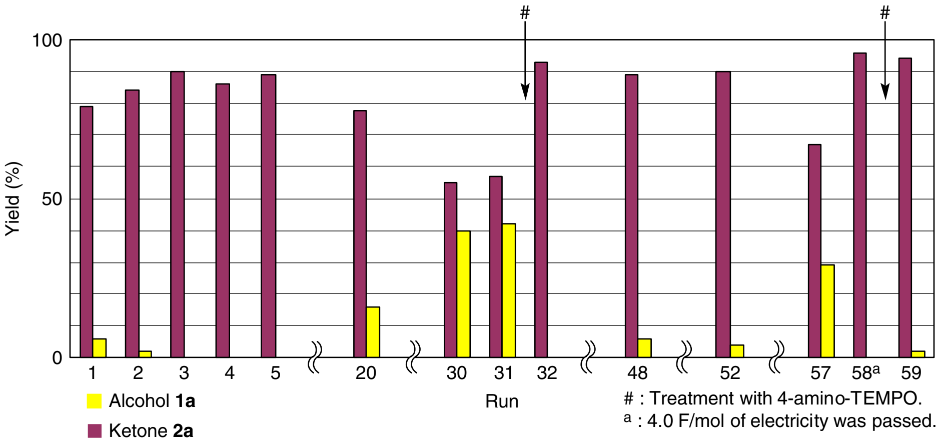
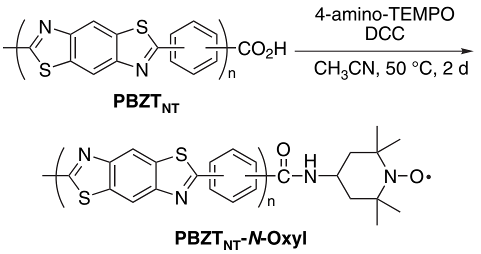


Figure 2. Recycle use of the *PBZT$_{NT}$-N-Oxyl*.

both straight chain segments and the branch point. The straight chain segments consist of benzobisthiazole and phenyl rings and is called PBZT. The PBZT is a rigid-rod polymer and the PBZT fiber is well known for its exceptional mechanical properties as well as its high thermal and chemical stabilities.[10] The PBZT$_{NT}$ also shows high mechanical, thermal, and chemical stability. In addition, many voids exist in the network and there are carboxyl groups at the branch and/or terminal point of the polymer chain, which can be used for immobilization of the *N*-oxyl moieties on the polymer particles. Herein, we describe that the PBZT$_{NT}$ particles were strong enough to survive without appreciable change in their shape and size even after more than 60 times recycle use for the electrooxidation of alcohols (Fig. 2).

2. Results and discussion

The *N*-oxyl-immobilized poly(*p*-phenylene benzobisthiazole) network polymer particles (*PBZT$_{NT}$-N-Oxyl*) were prepared by treatment of the PBZT$_{NT}$ particles with 4-amino-2,2,6,6-tetramethylpiperidine-*N*-oxyl (4-amino-TEMPO) in acetonitrile in the presence of dicyclohexylcarbodiimide (DCC) at 50 °C for 2 d (Scheme 1). The polymer particles were separated by filtration, washed successively with acetonitrile, water, ethanol, and ether, and dried under reduced pressure to afford the *PBZT$_{NT}$-N-Oxyl*. The weight of polymer particles increased from 400 to 450 mg, suggesting that ca. 0.6 mmol/g of the *N*-oxyl moiety was immobilized on the PBZT$_{NT}$.



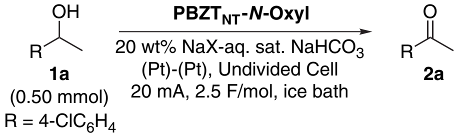
Scheme 1.

Electrolysis was carried out in a simple beaker-type undivided cell fitted with two platinum electrodes (1×1 cm^2 each). A typical procedure is as follows: a mixture of *PBZT$_{NT}$-N-Oxyl* (450 mg), and alcohol **1a** (R=4-Cl–C$_6$H$_4$; 0.5 mmol) in an aqueous satd NaHCO$_3$ containing 20 wt % NaBr was electrolyzed at 20 mA/cm^2 under vigorous stirring at 0 °C. After passage of 2.5 F/mol (1.67 h) of electricity, the *PBZT$_{NT}$-N-Oxyl* was separated by filtration and washed with EtOAc. The filtrates were extracted three times with EtOAc and the extracts and the washings were combined. Evaporation of the solvent afforded the corresponding ketone **2a** in 94% yield (Table 1, entry 1). The presence of both NaBr and NaHCO$_3$ is indispensable, since in the absence of each of them, the yields of **2a** decreased to 16–43% (entries 2 and 3). Neither NaCl nor NaI was effective since only 14% yield or no appreciable amount of the ketone **2a** was obtained by similar electrolysis with NaCl or NaI (entries 4 and 5).

Next, the required amount of *PBZT$_{NT}$-N-Oxyl* was examined (Table 2). When the electrooxidation of **1a** (0.5 mmol) was carried out with 400–200 mg of *PBZT$_{NT}$-N-Oxyl*, the corresponding ketone **2a** was obtained in good yield (entries 1–3). With less than 100 mg of the *PBZT$_{NT}$-N-Oxyl*, the yield of **2a** significantly decreased to 55–35%, suggesting that more than 200 mg of *PBZT$_{NT}$-N-Oxyl* would be required for the adsorption and the oxidation of 0.5 mmol of alcohol **1a** on the polymer surface.

The aqueous solution recovered after extractive workup process could also be used repeatedly. As shown in Figure 3, no significant change in the current efficiency and in the

Table 1. Effects of halide salts and NaHCO$_3$[a]

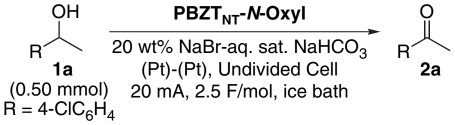


Entry	NaX	NaHCO$_3$	Yield **2a** (%)[b]	Recovered **1a** (%)[b]
1	NaBr	Satd	94	4
2	None	Satd	16	75
3	NaBr	None	43	51
4	NaCl	Satd	14	74
5	NaI	Satd	—	94

[a] Electrooxidation of **1a** (0.50 mmol) using *PBZT$_{NT}$-N-Oxyl* (450 mg) in aqueous solution was carried out under a constant current (20 mA, 2.5 F/mol, 1.67 h) in an undivided cell under ice bath.

[b] Yields were determined by GC analysis using acetophenone as an internal standard.

Table 2. Effects of the amount of *PBZT$_{NT}$-N-Oxyl* [a]



Entry	*PBZT$_{NT}$-N-Oxyl* (mg)	*N*-Oxyl (mmol)	Yield **2a** (%)[b]	Recovered **1a** (%)[b]
1	450	0.27	94	4
2	400	0.24	94	4
3	200	0.12	94	4
4	100	0.06	55	41
5	50	0.03	35	63

[a] Electrooxidation of **1a** (0.50 mmol) using *PBZT$_{NT}$-N-Oxyl* in aqueous solution was carried out under a constant current (20 mA, 2.5 F/mol, 1.67 h) in an undivided cell under ice bath.

[b] Yields were determined by GC analysis using acetophenone as an internal standard.

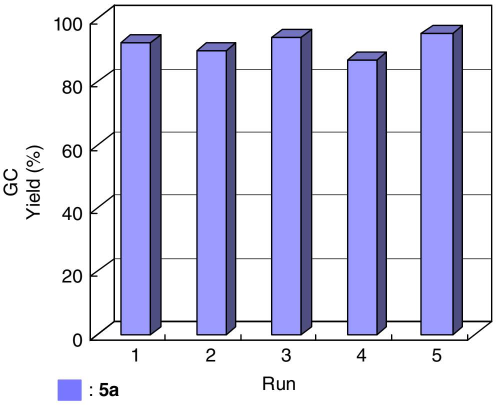
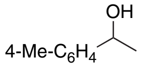
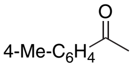
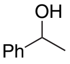
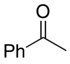
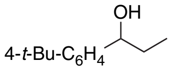
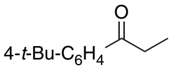
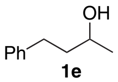
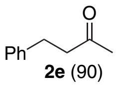
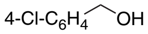
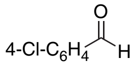
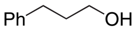
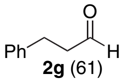
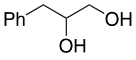
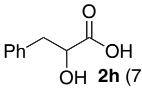
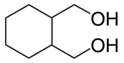
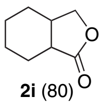


Figure 3. Recycle use of both the *PBZT$_{NT}$-N-Oxyl* and the aqueous media.

Table 3. Electrooxidation of alcohols in the *PBZT$_{NT}$-N-Oxyl* dispersed water system[a]

Entry	Substrate	F/mol	Products (Yield, %)[b]
1	 **1b**	2.5	 **2b** (84)
2	 **1c**	2.5	 **2c** (73)
3	 **1d**	4.0	 **2d** (68)
4	 **1e**	4.0	 **2e** (90)
5	 **1f**	2.5	 **2f** (30)[c]
6[d]	**1f**		**2f** (86)
7[e,f]	 **1g**	2.5	 **2g** (61)
8[f]	 **1h**	4.5	 **2h** (73)
9[d]	 **1i**	4.5	 **2i** (80)

[a] Electrooxidation of **1a** (0.50 mmol) using *PBZT$_{NT}$-N-Oxyl* (450 mg) in aqueous solution was carried out under a constant current (20 mA) in an undivided cell under ice bath.

[b] Isolated yields.

[c] 4-Chlorobenzoic acid (7%) was obtained.

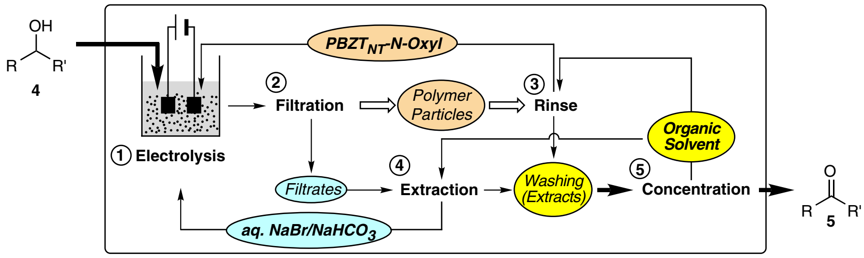
[d] Methyl ethyl ketone (1.5 mL) was used as a co-solvent.

[e] 3-Phenylpropanoic acid (28%) was obtained.

[f] Acetonitrile (1.5 mL) was used as a co-solvent.

conversion yields was observed when both the aqueous solution and *PBZT$_{NT}$-N-Oxyl* were recovered and reused for the electrooxidation of **1a**. The recycling system, formally offers a totally closed system as illustrated in Scheme 2.

It is of interest to note that *PBZT$_{NT}$-N-Oxyl* could be easily recovered and used repeatedly. Thus, *PBZT$_{NT}$-N-Oxyl* was recovered by filtration after the electrolysis and used for the subsequent electrolysis. The results of the recycle use of *PBZT$_{NT}$-N-Oxyl* are shown in Figure 2. The yields of the ketone **2a** varied in the range of 79–90% through runs 1–20. After 30 times recycle use (run 30), however, the yield of **2a** decreased to 55% and 40% of **1a** was recovered. It is likely that some of the *N*-oxyl moieties would be peeled off from the *PBZT$_{NT}$-N-Oxyl* during the repetition of the electrolysis. Indeed, when the recovered *PBZT$_{NT}$-N-Oxyl* was treated again with 4-amino-TEMPO to immobilize the *N*-Oxyl moiety, the efficiency of the *N*-oxyl-mediated oxidation of **1a** was almost completely recovered, affording **2a** in 93% yield (run 32). After additional 25 times recycle use (run 57), the yield of **2a** decreased to 67%. It is of interest to note that the yield of **2a** increased to over 90% by only passage of excess amount (3.0 F/mol) of electricity (run 58). Above all, it is likely that the immobilization process of the *N*-oxyl moiety on the PBZT$_{NT}$ particles in an appropriate interval enables the recycle use of the *PBZT$_{NT}$-N-Oxyl* for almost semi-permanent times (run >60).



Scheme 2. A totally closed electrolysis system.

The present electrooxidation in the *PBZT$_{NT}$-N-Oxyl*/Water disperse system could be successfully applied to various alcohols **1b–i**. The representative results are shown in Table 3. The electrooxidation of benzylic as well as aliphatic *sec*-alcohols **1b–e** proceeded smoothly to afford the corresponding ketones **2b–e** in good to excellent yields (entries 1–4). In contrast, the electrooxidation of *p*-chlorobenzyl alcohol (**1f**) under the standard condition afforded only 30% yield of the corresponding aldehyde **2f** together with the corresponding carboxylic acid (7%) (entry 5). When this electrooxidation was carried out in the presence of methyl ethyl ketone as a co-solvent, the yield of **2f** increased to 86% and no appreciable amount of the carboxylic acid was obtained (entry 6). The electrooxidation of aliphatic *prim*-alcohol **1g** and diols **1h** and **1i** was also performed by the use of methyl ethyl ketone or acetonitrile as a co-solvent to afford the corresponding aldehyde **2g**, carboxylic acid **2h** and lactone **2i** (entries 7–9).

3. Conclusion

In conclusion, the electrooxidation of alcohols **1** was successfully achieved in the disperse system with *N*-oxyl-immobilized poly(*p*-phenylene benzobisthiazole) network polymer (*PBZT$_{NT}$-N-Oxyl*) as a disperse phase and an aqueous 20 wt % NaBr/saturared $NaHCO_3$ as a disperse media. The disperse phase and the aqueous disperse media could be recovered and used repeatedly for the electrooxidation of alcohols, thereby offering a totally closed electrolysis system (Scheme 2).

4. Experimental

4.1. Preparation of poly(*p*-phenylene benzobisthiazole) network (PBZT$_{NT}$)

To a 500-mL reaction flask, equipped with a mechanical stirrer and an argon inlet/outlet adapters, were placed 2,5-diamino-1,4-benzenedithiol·dihydrochloride (6.17 g, 25.2 mmol) and PPA (115%, 125 g). The mixture was stirred under reduced pressure (<3 mmHg) and gradually heated up to 100 °C. On completion of degassing of hydrogen chloride, a stoichiometric amount of terephthalic acid (2.08 g, 12.6 mmol) and trimesic acid (1.75 g, 8.39 mmol) were added. The mixture was then heated to 140 °C under an argon atmosphere. The polymerization was carried out, while the solution viscosity was measured by a torque meter, and the gel point of the system was evaluated by the beginning of the precipitous climb of the viscosity. By stopping the reaction before the gelation, PBZT network particle was prepared. After the polymerization, the product was washed with sulfuric acid and water, and dried under reduced pressure at 100 °C to afford PBZT network as dark-brown powder: IR (KBr) 1708, 1100, 960, 690 cm^{-1}.

4.2. Preparation of *N*-oxyl-immobilized PBZT$_{NT}$: a typical procedure

A mixture of PBZT$_{NT}$ (400 mg), 4-amino-2,2,6,6-tetramethylpiperidine-*N*-oxyl (105 mg, 0.61 mmol) and DCC (119 mg, 0.58 mmol) in acetonitrile (10 mL) was heated at 50 °C for 2 d under an Ar atmosphere. The solid particles were separated by filtration and washed successively with acetonitrile, H_2O, MeOH, and Et_2O (20 mL each). The solids were dried under reduced pressure to afford *N*-oxyl-immobilized PBZT$_{NT}$ (451 mg, 0.6 mmol/g of the *N*-oxyl moiety was immobilized): black solids.

4.3. Electrooxidation of alcohols. A typical procedure

A mixture of *N*-oxyl-immobilized PBZT$_{NT}$ (450 mg) and 1-(4-chlorophenyl)ethanol **1a** (78.6 mg, 0.50 mmol) in an aqueous satd $NaHCO_3$ containing 20 wt % NaBr (5.0 mL) was placed in a beaker-type undivided cell. After stirring for 15 min, two platinum electrodes (1×1 cm^2) were immersed into the reaction mixture, and a constant current (20 mA, 1.67 h, 2.5 F/mol) was supplied at 0 °C under vigorous stirring. After electrolysis, the PBZT$_{NT}$ particles were separated by filtration and washed with EtOAc. The aqueous layer was extracted three times with EtOAc. The extracts and the washings were combined and dried over Na_2SO_4. Most of the solvents were evaporated and the residue was chromatographed on a silica gel column (hexane/EtOAc: 5/1) to afford 4-chloroacetophenone (**2a**, 70.0 mg, 0.45 mmol, 90%): a colorless liquid; R_f=0.46 (hexane/EtOAc: 5/1); ^{1}H NMR (200 MHz, $CDCl_3$) δ 2.60 (s, 3H, CH_3), 7.44 (d, J=8.6 Hz, 2H, Ar), 7.89 (d, J=8.6 Hz, 2H, Ar); IR (neat) 3006, 2970, 2904, 1687, 1590, 1572, 1176 cm^{-1}.

4.3.1. 4-Methylacetophenone (2b). A colorless liquid; ^{1}H NMR (200 MHz, $CDCl_3$): δ 2.42 (s, 3H, CH_3–Ar), 2.58 (s, 3H, CH_3), 7.26 (m, 2H, Ar), 7.86 (d, J=8.2 Hz, 2H, Ar); IR (neat) 3004, 2923, 1683, 1607, 1183 cm^{-1}.

4.3.2. Acetophenone (2c). A colorless liquid; ^{1}H NMR (200 MHz, $CDCl_3$): δ 2.61 (s, 3H, CH_3), 7.43–7.60 (m, 3H, Ar), 7.96 (d, J=7.6 Hz, 2H, Ar); IR (neat) 3004, 2923, 1686, 1599, 1266, 761 cm^{-1}.

4.3.3. 1-(4-*tert*-Butylphenyl)-1-propanone (2d). A colorless liquid; ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.22 (t, J=7.2 Hz, 3H, CH_3), 1.34 (s, 9H, $(CH_3)_3C$), 2.98 (q, J=7.2 Hz, 2H, CH_2), 7.47 (d, J=8.6 Hz, 2H, Ar), 7.91 (d, J=8.6 Hz, 2H, Ar); IR (neat) 2967, 1687, 1607, 1228, 1192, 801 cm^{-1}.

4.3.4. 4-Phenyl-2-butanone (2e). A colorless liquid; ^{1}H NMR (200 MHz, $CDCl_3$): δ 2.14 (s, 3H, CH_3), 2.71–2.79 (m, 2H, CH_2–Ar), 2.86–2.95 (m, 2H, CH_2CO), 7.16–7.31 (m, 5H, Ar); IR (neat) 3027, 2923, 1717, 1497, 1454, 1162, 750 cm^{-1}.

4.3.5. 4-Chlorobenzaldehyde (2f). White solids; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.52 (d, J=8.5 Hz, 2H, Ar), 7.82 (d, J=8.5 Hz, 2H, Ar), 9.98 (s, 1H, CHO); IR (KBr) 3019, 2985, 2862, 1693, 1590, 1576, 1209 cm^{-1}.

4.3.6. 3-Phenylpropionaldehyde (2g). A colorless liquid; ^{1}H NMR (200 MHz, $CDCl_3$): δ 2.74–2.82 (m, 2H, CH_2–Ar), 2.90–3.02 (m, 2H, CH_2CO), 7.16–7.34 (m, 5H, Ar), 9.82 (s, 1H, CHO); IR (neat) 3029, 2928, 1725, 1604, 1455, 1180, 747 cm^{-1}.

4.3.7. 3-Phenylpropanoic acid-2-ol (2h). White solids; ^{1}H NMR (200 MHz, $CDCl_3$): δ 2.94–3.26 (m, 2H, CH_2), 4.49–4.55 (m, 1H, CH), 7.23–7.38 (m, 5H, Ar); IR (KBr) 3006, 2966, 1687, 1590, 1488, 1262, 1176 cm^{-1}.

4.3.8. *cis*-8-Oxabicyclo[4.3.0]nonan-7-one (2i). A colorless liquid; ^1H NMR (200 MHz, $CDCl_3$): δ 1.13–1.36 (m, 3H), 1.52–1.70 (m, 3H), 1.76–1.84 (m, 1H), 2.06–2.15 (m, 1H), 2.39–2.52 (m, 1H, CH), 2.59–2.69 (m, 1H, CH–C(O)–), 3.94 (d, $J=9$ Hz, 1H, –C(O)OCH_2–), 4.18 (dd, $J=9$ Hz, $J=5$ Hz, 1H, –C(O)OCH_2–); IR (neat) 2935, 1857, 1774, 1376, 1160, 1129 cm^{-1}.

Acknowledgment

We thank the SC-NMR Laboratory of Okayama University for ^1H and ^{13}C NMR analyses.

References and notes

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