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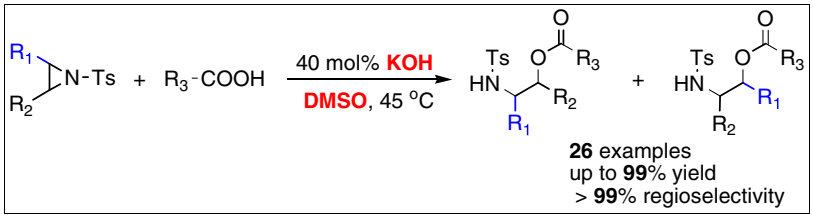
Received October 29, 2012
DOI 10.1002/jhet.1962
Published online 12 May 2014 in Wiley Online Library (wileyonlinelibrary.com).



A facile and efficient KOH-oriented regioselective ring-opening reaction for the acetolysis of *N*-tosylaziridines in DMSO was developed under mild conditions. This operationally simple protocol could tolerate a variety of functionalized carboxylic acids as well as several *N*-tosylaziridines. Moreover, this strategy provided the corresponding ring-opening products with good results.

J. Heterocyclic Chem., **52**, 284 (2015).

INTRODUCTION

Aziridines are widely recognized as significant and versatile building blocks for the synthesis of various biologically active compounds [1–26]. Their activity has led to wide research and use in the ring-opening reactions with a range of nucleophiles [27–50]. As a result, this area has attracted a great deal of attention over nearly three decades, and the pace of development has shown no sign of abating in recent years. Although there has been a great deal of interest in the ring openings of *N*-tosylaziridines with various nucleophiles, comparably, few examples on the ring openings with carboxylic acids and their derivatives are observed [51–54]. So, the discovery of new catalysts that are cheaper and more efficient and the development of new methods that are more effective remain a considerable challenge and are a long-standing problem. Inorganic bases as efficient catalysts have been widely utilized in many reactions (See Refs [51] and [52]) [55–57]. Moreover, DMSO [58–62] has been efficiently utilized as an organic catalyst or solvent in the ring-opening reactions of aziridines with some nucleophiles. Herein, we wish to report the successful application of KOH as a simple, inexpensive, and remarkable catalyst for the efficient regioselective ring openings of *N*-tosylaziridines with carboxylic acids in DMSO.

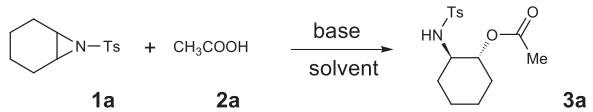
RESULTS AND DISCUSSION

Our investigations began with the screening of catalysts by selecting the ring-opening reaction of *N*-tosylcyclohexylaziridine (**1a**) with acetic acid (**2a**) as the model system. Initial studies revealed that without any catalyst, the ring-opening reaction afforded little ring-opening product in DMSO. Rising temperature up to 80°C made limited impact (Table 1, entry 1). Considering they may improve the nucleophilicity of carboxylic acids, some bases were tried to catalyze this process. It was found that weaker bases such as K_2CO_3 and NEt_3 were less effective than strong bases (Table 1, entries 2 and 3 vs. 4). KOH showed the excellent yield (Table 1, entry 4). In the presence of a catalytic amount of KOH, the reaction could be carried out smoothly and afforded the desired ring-opening product in good yield (Table 1, entry 4). Surprisingly, however, this reaction could not take place with stoichiometric amount of KOH (Table 1, entry 6) [63]. On the other hand, the examination of solvent effect indicated that anhydrous DMSO could remarkably facilitate this ring-opening reaction with the best yield (Table 1, entry 4), and NMP indicated worse result (Table 1, entry 7). However, other polar solvents failed to manifest such property (Table 1, entries 8–13 vs. 4), which suggested that the positive solvent effect of DMSO played another important role and its presence was necessary in this catalytic process. In addition, it was observed that this reaction worked most efficiently at 45°C (Table 1, entry 15 vs. 4, 14, and 16). In summary, extensive screening showed that the optimized reaction conditions were 40 mol% KOH and 1.2 equiv of acetic acid (**2a**) under air atmosphere in 1.0 mL DMSO at 45°C for 3 h (Table 1, entry 15).

With the optimized experimental conditions in hand, a series of carboxylic acids including aromatic, aliphatic, and heterocyclic acids were examined for the ring-opening reactions of *N*-tosylaziridines, and the results are summarized in Table 2. Neither the electronic property of the substitution at the aromatic ring nor the steric hindrance had obvious influence on the yields. Most aromatic carboxylic

Table 1

Ring opening of *N*-tosylcyclohexylaziridine (**1a**) with acetic acid (**2a**)[a].



1a **2a** **3a**

Entry	Base	Amount of base (mol%)	Solvent	T (°C)	*t* (h)	Yield[b] (%)
1	–	–	DMSO	25–80	24	N.D.
2	K_2CO_3	40	DMSO	25	24	23
3	TEA	40	DMSO	25	24	30
4	KOH	40	DMSO	25	24	95
5	KOH	20	DMSO	25	24	40
6	KOH	100	DMSO	25	24	trace
7	KOH	40	NMP	25	24	64
8	KOH	40	THF	25	24	N.D.
9	KOH	40	Acetone	25	24	N.D.
10	KOH	40	Dioxane	25	24	N.D.
11	KOH	40	DMF	25	24	N.D.
12	KOH	40	CH_3CN	25	24	N.D.
13	KOH	40	Toluene	25	24	N.D.
14	KOH	40	DMSO	35	12	98
15	KOH	40	DMSO	45	3	99
16	KOH	40	DMSO	55	2	90

N.D., not detected.
[a]All reactions were performed with *N*-tosylcyclohexylaziridine (**1a**) (50 mg, 0.2 mmol) and acetic acid (**2a**) (14 μL, 0.24 mmol, 1.2 equiv) in 1.0 mL DMSO under the specified conditions.
[b]Isolated yield.

acids underwent rapidly the ring-opening reactions and afforded the corresponding products in good to excellent yields (Table 2, entries 2–9). Aliphatic and unsaturated acids also offered excellent results (Table 2, entries 1, 11, and 12). Heterocyclic aromatic carboxylic acid was found to be good substrate and afforded the desired ring-opening product in 84% yield, too (Table 2, entry 10).

Subsequently, a variety of *N*-tosylaziridines such as aromatic, aliphatic, and condensed-ring ones were investigated under the optimal conditions, and the corresponding major products **3** (aromatic *N*-tosylaziridines) or **4** (aliphatic *N*-tosylaziridines) were provided in good to excellent yields, as shown in Table 3. In the case of substituted aromatic *N*-tosylaziridines, both the electronic property and the steric hindrance of the substitution at the aromatic ring had no obvious influence on the yields and up to 99% yield was obtained (Table 3, entries 1–8, 11, and 12). In addition, condensed-ring *N*-tosylaziridines were also found to be suitable substrates (Table 3, entries 9 and 10). It was noteworthy that excellent yields could be attained with substituted aliphatic *N*-tosylaziridines (Table 3, entries 13 and 14). Inspiringly, most *N*-tosylaziridines gave single regioisomers or good regioselectivity except for **1c**, **1e**, **1g**, **1h**, **1j**, **1k**, and **1l**, which provided worse regioselectivity (Table 3, entries 2, 4, 6, 7, 9–11).

For the purpose of examining the synthetic potential of the present approach, a gram-scaled synthesis of ring-opening products was performed for this catalytic system. As shown in Scheme 1, in the presence of 40 mol% of KOH, 10 mmol of *N*-tosylaziridine (**1m**) reacted with 1.2 equiv of **2h** to provide **3x** as the single product in a total yield of 83% (3.656 g). Similarly, subgram quantities of products **3o** and **4o** with the isomer **3o** as the major product (4.318 g, 95% total yield, **3o**:**4o** = 91:9) were obtained.

CONCLUSIONS

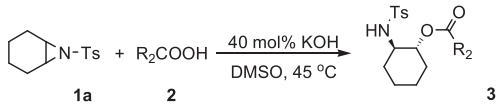
In conclusion, we developed facile, efficient, and high atom economy KOH-oriented regioselective ring-opening reactions for the acetolysis of *N*-tosylaziridines in DMSO. The combined use of KOH and DMSO was crucial and important to the desired ring-opening reaction. More importantly, good to excellent yields have been provided for a wide variety of *N*-tosylaziridines and various carboxylic acids, and good to high regioselectivity were obtained in terms of most *N*-tosylaziridines under mild reaction conditions.

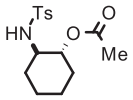
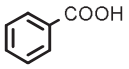
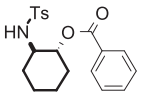
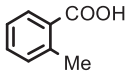
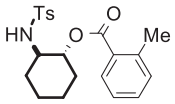
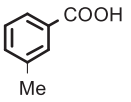
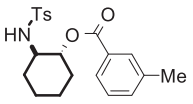
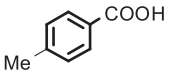
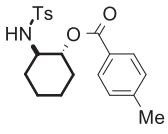
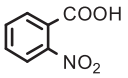
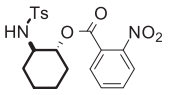
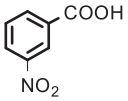
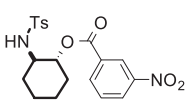
EXPERIMENTAL

General information. ^{1}H-NMR spectra were taken with Bruker AVANCE III 600 MHz NMR spectrometers. The

Table 2

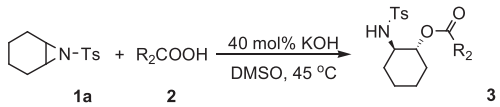
Ring-opening reactions of *N*-tosylcyclohexylaziridine (**1a**) with various carboxylic acids[a].



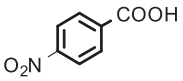
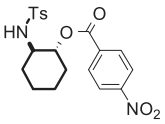
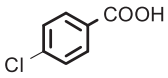
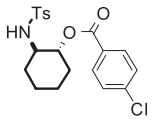
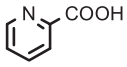
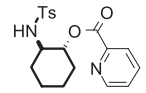
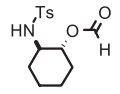
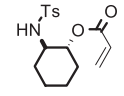
Entry	Carboxylic acid (**2**)		Product		*t* (h)	Yield[b] (%)
1	CH_3COOH	**2a**		**3a**	3	99
2		**2b**		**3b**	2	98
3		**2c**		**3c**	1.5	98
4		**2d**		**3d**	2	97
5		**2e**		**3e**	2	98
6		**2f**		**3f**	3	95
7		**2g**		**3g**	6	97

(*Continued*)

Table 2
(*Continued*)



1a 2 3

Entry	Carboxylic acid (**2**)	Product	*t* (h)	Yield[b] (%)
8	 **2h**	 **3h**	3	95
9	 **2i**	 **3i**	4	94
10	 **2j**	 **3j**	5	84
11	HCOOH **2k**	 **3k**	4	80
12	H_2C=HCOOH **2l**	 **3l**	1.5	94

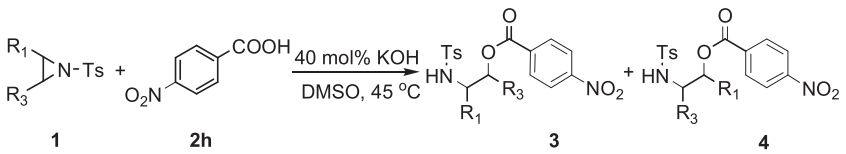
[a]All reactions were performed with *N*-tosylcyclohexylaziridine (**1a**) (50 mg, 0.2 mmol), carboxylic acids (**2**) (0.24 mmol, 1.2 equiv) and KOH (4.5 mg, 40 mol%) under air atmosphere in DMSO (1.0 mL) at 45°C.
[b]Isolated yield.

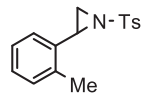
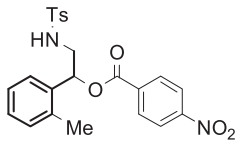
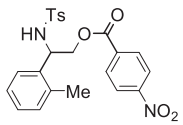
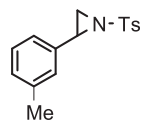
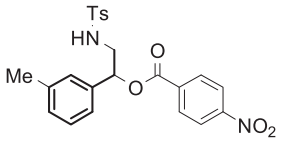
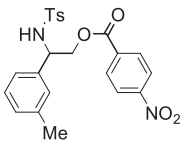
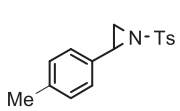
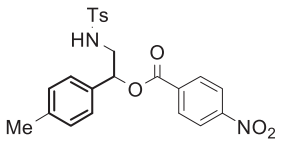
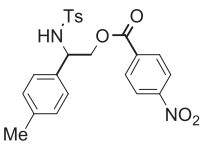
chemical shifts are reported in parts per million (ppm) downfield to the $CDCl_3$ resonance ($\delta = 7.27$). Spectra are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet), coupling constants (Hz), integration, and assignment. ^{13}C-NMR data were collected at 150 MHz with complete proton decoupling. The chemical shifts are reported in parts per million downfield to the central $CDCl_3$ resonance ($\delta = 77.0$). Coupling constants in ^{1}H-NMR spectra are given in Hertz (Hz). High-resolution mass spectra were performed on a micrOTOF-Q II instrument with an ESI source. Melting points were measured with an RD-II melting point apparatus and are uncorrected. Unless otherwise noted, reagents obtained from commercial sources were used without further purification. All solvents were purchased from commercial sources and used with further purification. Deuterated solvents were purchased from Aladdin. Column chromatography was performed on silica gel (200–300 mesh). All yields were referred to isolated yields (average of two runs) of compounds.

General procedure. To a mixture of *N*-tosylaziridine (0.2 mmol), carboxylic acid (0.24 mmol, 1.2 equiv) and KOH (4.5 mg, 0.08 mmol, 40 mol%) was added DMSO (1 mL). The reaction mixture was then stirred at 45°C until the *N*-tosylaziridine

Table 3

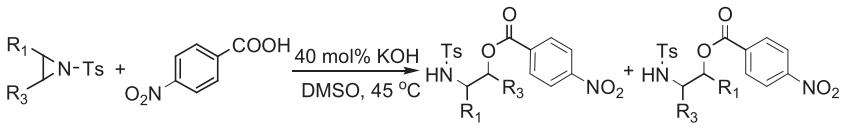
Ring-opening reactions of various *N*-tosylaziridines (**1**) with 4-nitrobenzoic acid (**2h**)[a].

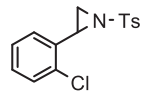
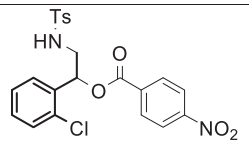
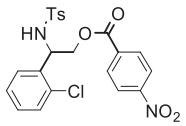
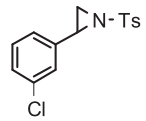
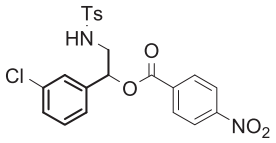
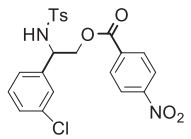
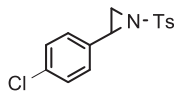
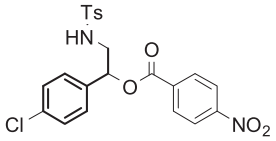
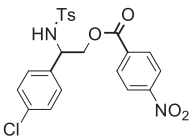


Entry	*N*-Tosylaziridine (**1**)	Product	*t* (h)	Yield[b] (%)
1	 **1b**	 **3m**  **4m**	4	96[c] (**3m**:**4m** = 70:30)
2	 **1c**	 **3n**  **4n**	3	96[c] (**3n**:**4n** = 54:46)
3	 **1d**	 **3o**  **4o**	3	97[d] (**3o**:**4o** = 91:9)

(*Continued*)

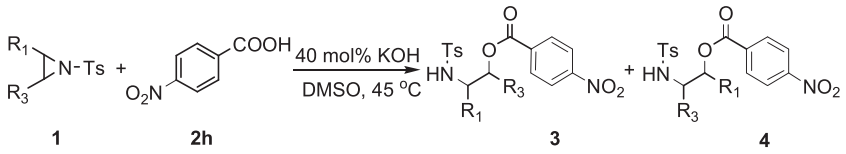
Table 3
(*Continued*)



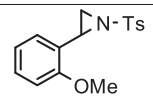
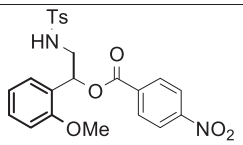
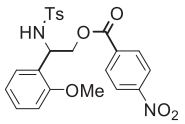
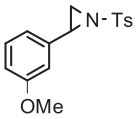
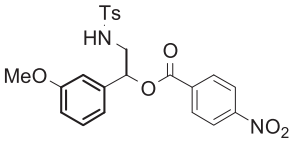
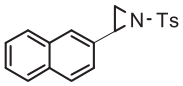
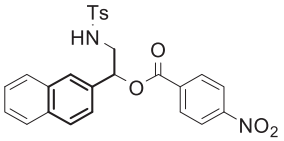
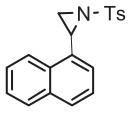
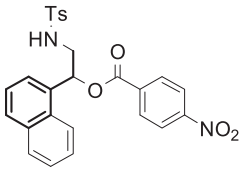
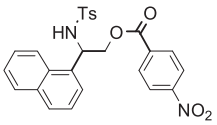
Entry	*N*-Tosylaziridine (**1**)	Product	*t* (h)	Yield[b] (%)
4	 **1e**	 **3p**  **4p**	5	98[c] (**3p**:**4p** = 62:38)
5	 **1f**	 **3q**  **4q**	4	96[c] (**3q**:**4q** = 90:10)
6	 **1g**	 **3r**  **4r**	3.5	97[c] (**3r**:**4r** = 64:36)

(*Continued*)

Table 3
(*Continued*)

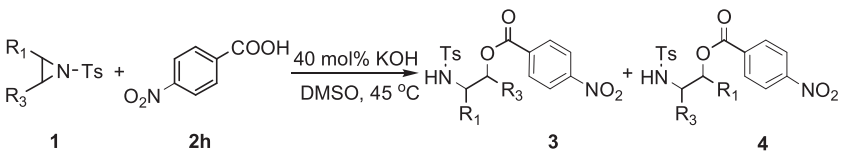


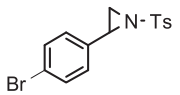
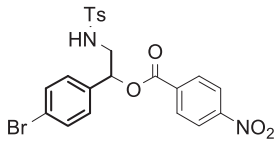
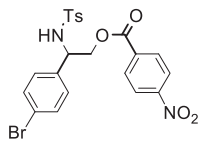
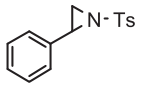
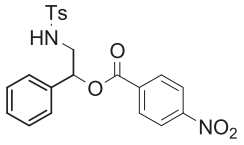
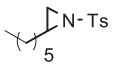
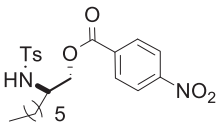
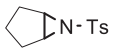
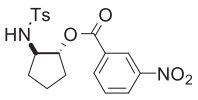
1 **2h** **3** **4**

Entry	*N*-Tosylaziridine (**1**)	Product	*t* (h)	Yield[b] (%)
7	 **1h**	 **3s**  **4s**	4	72[c] (**3s**:**4s** = 51:49)
8	 **1i**	 **3t**	3	86[c] (**3t**:**4t** = 75:25)
9	 **1j**	 **3u**	10	86[c] (**3u**:**4u** = 56:44)
10	 **1k**	 **3v**  **4v**	5	99[d] (**3v**:**4v** = 61:39)
11			2.5	92[c] (**3w**:**4w** = 52:48)

(*Continued*)

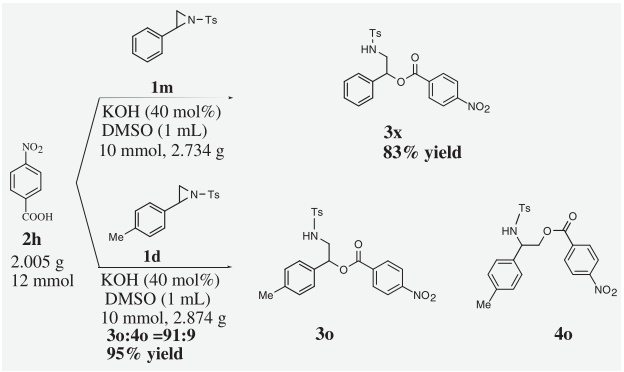
Table 3
(*Continued*)



Entry	*N*-Tosylaziridine (**1**)	Product	*t* (h)	Yield[b] (%)
	 **1l**	 **3w**		
		 **4w**		
12	 **1m**	 **3x**	4	80
13	 **1p**	 **4y**	5	98
14	 **1q**	 **3z**	10	92

[a]All reactions were performed with *N*-tosylaziridines (**1**) (0.2 mmol), 4-nitrobenzoic acid (**2h**) (40 mg, 0.24 mmol, 1.2 equiv) and KOH (4.5 mg, 40 mol%) under air atmosphere in DMSO (1.0 mL) at 45°C.
[b]Isolated yield.
[c]The ratios of the two isomer products were determined by ^{1}H-NMR.
[d]Combined yield of isolated **3** and **4**.

Scheme 1. Scaled-up version of the ring-opening reactions.



was consumed completely that was monitored by TLC. Subsequently, the mixture was washed with 0.36 *M* of K_2CO_3 solution (10 mL) to remove the remains of the carboxylic acid. The water phase was extracted with CH_2Cl_2 (3 × 5 mL). The organic extracts were combined and dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure to obtain the remainder. At last, the resulting crude product was purified by column chromatography on silica gel with petroleum ether/ethyl acetate to afford the desired corresponding product in high yield.

2-(4-Methylphenylsulfonamido)cyclohexyl acetate (3a). White solid; mp 124–126°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.16–1.27 (m, 4H), 1.59–1.62 (m, 2H), 1.76 (s, 3H), 1.90–1.91 (m, 2H), 2.36 (s, 3H), 3.12–3.14 (m, 1H), 4.52–4.53 (m, 1H), 5.36–5.37 (d, 1H), 7.24–7.26 (d, *J* = 8.4 Hz, 2H), 7.71–7.72 (d, *J* = 8.4 Hz, 2H) ppm.

2-(4-Methylphenylsulfonamido)cyclohexyl benzoate (3b). White solid; mp 156–158°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.28–1.48 (m, 4H), 1.69–1.76 (m, 2H), 2.00–2.04 (m, 1H), 2.18 (s, 3H), 2.22–2.24 (m, 1H), 3.28–3.32 (m, 1H), 4.79–4.83 (m, 1H), 5.03–5.04 (d, 1H), 6.89–6.91 (d, *J* = 7.8 Hz, 2H), 7.35–7.38 (t, *J* = 7.8 Hz, 2H), 7.54–7.58 (m, 3H), 7.75–7.76 (m, 2H) ppm.

2-(4-Methylphenylsulfonamido)cyclohexyl 2-methylbenzoate (3c). White solid; mp 130–132°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.25–1.50 (m, 4H), 1.69–1.79 (m, 2H), 1.99–2.02 (m, 1H), 2.19 (s, 3H), 2.21 (m, 1H), 2.45 (s, 3H), 3.29–3.31 (m, 1H), 4.79–4.83 (m, 1H), 5.08–5.09 (d, *J* = 7.2 Hz, 1H), 6.90–6.91 (d, *J* = 8.4 Hz, 2H), 7.16–7.19 (m, 2H), 7.38–7.39 (d, *J* = 1.2 Hz, 1H), 7.57–7.58 (d, *J* = 8.4 Hz, 2H), 7.64–7.64 (d, *J* = 1.2 Hz, 1H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.4, 22.0, 23.8, 24.2, 31.3, 34.0, 57.3, 74.3, 125.5, 126.5, 128.8, 129.3, 131.0, 131.6, 132.2, 138.2, 140.6, 142.7, 167.6 ppm. MS (ESI): Calcd for $C_{21}H_{25}NO_4S$ + Na 410.1402, found 410.1403.

2-(4-Methylphenylsulfonamido)cyclohexyl 3-methylbenzoate (3d). White solid; mp 129–131°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.25–1.49 (m, 4H), 1.68–1.76 (m, 2H), 1.99–2.01 (m, 1H), 2.16 (s, 3H), 2.17–2.20 (m, 1H), 2.37 (s, 3H), 3.29–3.31 (m, 1H), 4.79–4.83 (m, 1H), 5.20–5.21 (d, *J* = 7.2 Hz, 1H), 6.89–6.91 (d, *J* = 8.4 Hz, 2H), 7.23–7.26 (m, 1H), 7.34–7.35 (d, *J* = 7.2 Hz, 1H), 7.55–7.56 (m, 4H) ppm.

2-(4-Methylphenylsulfonamido)cyclohexyl 4-methylbenzoate (3e). White solid; mp 144–146°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.28–1.47 (m, 4H), 1.62–1.66 (m, 2H), 1.92–1.94 (m, 1H), 2.09 (s, 3H), 2.10–2.14 (m, 1H), 2.37 (s, 3H), 3.21–3.22 (m, 1H), 4.70–4.74 (m, 1H), 5.11–5.12 (d, *J* = 7.2 Hz, 1H), 6.82–6.83 (d, *J* = 8.4 Hz, 2H), 7.07–7.08 (d, *J* = 7.8 Hz, 2H), 7.49–7.50 (d, *J* = 8.4 Hz, 2H), 7.55–7.57 (d, *J* = 7.8 Hz, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.3, 21.6, 23.8, 24.2, 31.3, 34.1, 57.3, 74.4, 126.6, 127.0, 128.8, 129.3, 129.8, 138.1, 142.6, 143.6, 166.9 ppm. MS (ESI): Calcd for $C_{21}H_{25}NO_4S$ + Na 410.1402, found 410.1402.

2-(4-Methylphenylsulfonamido)cyclohexyl 2-nitrobenzoate (3f). White solid; mp 135–137°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.22–1.37 (m, 4H), 1.47–1.53 (m, 1H), 1.70–1.72 (m, 1H), 1.77–1.79 (m, 1H), 2.04–2.09 (m, 2H), 2.23 (s, 3H), 3.38–3.41 (m, 1H), 4.82–4.86 (m, 1H), 4.90–4.92 (d, *J* = 7.8 Hz, 1H), 7.02–7.03 (d, *J* = 8.4 Hz, 2H), 7.61–7.62 (d, *J* = 8.4 Hz, 2H), 7.98–8.00 (d, *J* = 9.0 Hz, 2H), 8.20–8.22 (d, *J* = 8.4 Hz, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.5, 23.4, 24.1, 30.2, 32.8, 56.3, 76.2, 123.6, 126.7, 127.4, 129.4, 130.2, 131.6, 132.9, 138.4, 142.8, 147.9, 165.2 ppm. MS (ESI): Calcd for $C_{20}H_{22}N_2O_6S$ + Na 441.1096, found 441.1088.

2-(4-Methylphenylsulfonamido)cyclohexyl 3-nitrobenzoate (3g). White solid; mp 138–140°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.29–1.39 (m, 3H), 1.51–1.53 (m, 1H), 1.71–1.77 (m, 2H), 2.04–2.07 (m, 2H), 2.17 (s, 3H), 3.37–3.39 (m, 1H), 4.87–4.88 (m, 1H), 5.07–5.08 (d, *J* = 8.4 Hz, 1H), 6.97–6.98 (d, *J* = 7.8 Hz, 2H), 7.59–7.61 (d, *J* = 8.4 Hz, 3H), 8.19–8.20 (d, *J* = 7.8 Hz, 1H), 8.38–8.40 (m, 1H), 8.56 (t, *J* = 1.8 Hz, 1H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.3, 23.7, 24.2, 31.1, 33.5, 56.9, 75.8, 124.6, 126.6, 127.2, 129.4, 131.7, 135.5, 138.4, 142.8, 148.1, 164.4 ppm. MS (ESI): Calcd for $C_{20}H_{22}N_2O_6S$ + Na 441.1096, found 441.1096.

2-(4-Methylphenylsulfonamido)cyclohexyl 4-nitrobenzoate (3h). White solid; mp 150–152°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.27–1.37 (m, 3H), 1.45–1.52 (m, 1H), 1.6–1.75 (m, 2H), 2.01–2.09 (m, 2H), 2.23 (s, 3H), 3.38–3.40 (m, 1H), 4.82–4.86 (m, 1H), 5.04–5.06 (d, *J* = 8.4 Hz, 1H), 7.02–7.03 (d, *J* = 7.8 Hz, 2H), 7.61–7.63 (d, *J* = 8.4 Hz, 2H), 7.98–8.00 (d, *J* = 7.8 Hz, 2H), 8.19–8.21 (d, *J* = 9.0 Hz, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.4, 23.7, 24.3, 31.1, 33.6, 56.9, 75.8, 123.2, 126.6, 129.5, 130.9, 135.2, 138.5, 142.9, 150.5, 164.7 ppm. MS (ESI): Calcd for $C_{20}H_{22}N_2O_6S$ + Na 441.1096, found 441.1093.

2-(4-Methylphenylsulfonamido)cyclohexyl 4-chlorobenzoate (3i). White solid; mp 137–139°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.25–1.36 (m, 3H), 1.46–1.50 (m, 1H), 1.67–1.74 (m, 2H), 2.06–2.09 (m, 2H), 2.22 (s, 3H), 3.33–3.35 (m, 1H), 4.81–4.85 (m, 1H), 5.09–5.11 (d, *J* = 7.8 Hz, 1H), 6.99–7.01 (d, *J* = 8.4 Hz, 2H), 7.24–7.26 (m, 1H), 7.41–7.42 (d, *J* = 3.6 Hz, 2H), 7.63–7.68 (m, 3H) ppm.

2-(4-Methylphenylsulfonamido)cyclohexyl picolinate (3j). White solid; mp 220–222°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.24–1.41 (m, 4H), 1.55–1.57 (m, 1H), 1.70–1.75 (m, 2H), 2.03–2.06 (m, 1H), 2.18 (s, 3H), 3.38–3.44 (m, 1H), 4.89–4.92 (m, 1H), 5.05–5.06 (d, *J* = 7.8 Hz, 1H), 6.91–6.93 (d, *J* = 8.4 Hz, 2H), 7.45–7.47 (m, 1H), 7.60–7.61 (d, *J* = 7.8 Hz, 2H), 7.75–7.78 (q, 1H), 7.88–7.89 (d, *J* = 7.8 Hz, 1H), 8.70–8.71 (q, 1H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.4, 23.8, 24.2, 31.1, 34.0, 57.1, 75.6, 125.3, 126.7, 129.3, 136.7, 138.3, 142.5, 147.6, 149.8, 165.1 ppm. MS (ESI): Calcd for $C_{19}H_{22}N_2O_4S$ + Na 397.1198, found 397.1192.

2-(4-Methylphenylsulfonamido)cyclohexyl formate (3k). White solid; mp 116–118°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.25–1.36 (m, 4H), 1.65–1.69 (m, 2H), 1.92–2.01 (m, 2H), 2.41 (s, 3H), 3.24–3.26 (m, 1H), 4.64–4.68 (m, 1H), 4.90–4.91 (d, *J* = 7.8 Hz, 1H), 7.28–7.29 (d, *J* = 8.4 Hz, 2H), 7.53 (s, 1H), 7.73–7.74 (d, *J* = 8.4 Hz, 2H) ppm.

2-(4-Methylphenylsulfonamido)cyclohexyl acrylate (3l). White solid; mp 106–108°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.21–1.36

(m, 4H), 1.63–1.70 (m, 2H), 1.94–1.96 (m, 1H), 2.05–2.08 (m, 1H), 2.39 (s, 3H), 3.21–3.23 (m, 1H), 4.62–4.64 (m, 1H), 5.00–5.01 (d, J = 7.2 Hz, 1H), 5.71–5.78 (m, 2H), 6.21–6.24 (dd, J = 16.8, 1.2 Hz, 1H), 7.23–7.24 (d, J = 7.8 Hz, 2H), 7.69–7.70 (d, J = 8.4 Hz, 2H) ppm.

***2-(4-Methylphenylsulfonamido)-1-o*-tolylethyl 4-nitrobenzoate (3m).** White solid; mp 164–166°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.17 (s, 3H), 2.39 (s, 3H), 3.38–3.50 (m, 1H), 4.41–4.44 (m, 1H), 5.27–5.30 (t, J = 7.2 Hz, 1H), 6.20–6.22 (q, 1H), 7.02–7.04 (d, J = 8.4 Hz, 1H), 7.16–7.19 (m, 1H), 7.20–7.27 (m, 2H), 7.30–7.31 (d, J = 7.8 Hz, 1H), 7.53–7.54 (d, J = 8.4 Hz, 1H), 7.70–7.72 (d, J = 8.4 Hz, 1H), 8.07–8.10 (dt, J = 9.0, 1.8 Hz, 1H), 8.15–8.17 (dt, J = 9.0, 1.8 Hz, 1H), 8.22–8.24 (d, J = 8.4 Hz, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 19.1, 21.4, 46.9, 73.1, 123.5, 125.5, 126.6, 127.0, 128.2, 128.8, 129.4, 129.9, 130.9, 134.9, 135.0, 135.4, 143.7, 150.6, 164.5 ppm. MS (ESI): Calcd for $C_{23}H_{22}N_2O_6S$ + Na 477.1096, found 477.1093.

Mixture of 3n and 4n. Inseparable white solid (**3n**:**4n** = 54:46); mp 137–144°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.21 (s, 3H), 2.30 (s, 3H), 2.31 (s, 3H), 2.39 (s, 3H), 3.41–3.54 (m, 2H), 4.42–4.49 (m, 1H), 4.50–4.52 (m, 1H), 4.73–4.74 (td, J = 7.8, 4.8 Hz, 1H), 5.49–5.52 (t, J = 4.8 Hz, 1H), 5.92–5.93 (d, J = 7.8 Hz, 1H), 5.95–5.97 (m, 1H), 6.90 (s, 1H), 6.98–7.01 (m, 2H), 7.03–7.06 (m, 2H), 7.10–7.13 (t, J = 7.2 Hz, 4H), 7.21–7.23 (m, 3H), 7.56–7.57 (d, J = 8.4 Hz, 2H), 7.69–7.71 (d, J = 8.4 Hz, 2H), 8.06–8.08 (m, J = 8.4 Hz, 2H), 8.14–8.16 (dt, J = 9.0, 2.4 Hz, 2H), 8.18–8.20 (m, 4H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.2, 21.4, 21.5, 47.7, 56.9, 67.7, 75.9, 123.4, 127.0, 127.1, 128.8, 129.4, 129.8, 130.9, 134.8, 135.0, 136.4, 136.6, 137.0, 137.4, 138.5, 138.7, 143.4, 143.6, 150.6, 163.8, 164.5 ppm. MS (ESI): Calcd for $C_{23}H_{22}N_2O_6S$ + Na 477.1096, found 477.1091.

***2-(4-Methylphenylsulfonamido)-1-p*-tolylethyl 4-nitrobenzoate (3o).** White solid; mp 140–142°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.17 (s, 3H), 2.39 (s, 3H), 3.43–3.52 (m, 1H), 4.42–4.53 (m, 1H), 5.18–5.20 (t, J = 6.4 Hz, 1H), 5.95–5.97 (dd, J = 6.4, 4.2 Hz, 1H), 7.05–7.08 (m, 2H), 7.14–7.16 (d, J = 7.8 Hz, 2H), 7.21–7.27 (m, 2H), 7.57–7.58 (d, J = 8.4 Hz, 1H), 7.68–7.70 (t, J = 8.4 Hz, 1H), 8.06–8.08 (d, J = 9.0 Hz, 1H), 8.15–8.17 (d, J = 9.0 Hz, 1H), 8.20–8.22 (q, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.2, 21.5, 47.6, 75.7, 123.5, 126.4, 127.0, 129.6, 129.8, 130.9, 133.4, 135.1, 137.0, 139.0, 143.7, 150.6, 163.8 ppm. MS (ESI): Calcd for $C_{23}H_{22}N_2O_6S$ + Na 477.1096, found 441.1093.

2-(4-Methylphenylsulfonamido)-1-(2-chlorophenyl)ethyl 4-nitrobenzoate (3p). White solid; mp 175–177°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.28 (s, 3H), 4.42–4.45 (dd, J = 11.4, 4.8 Hz, 1H), 4.56–4.59 (dd, J = 11.4, 7.8 Hz, 1H), 5.22–5.25 (m, 1H), 5.82–5.84 (d, J = 7.8 Hz, 1H), 7.05–7.06 (d, J = 7.8 Hz, 2H), 7.14–7.19 (m, 2H), 7.26–7.33 (m, 2H), 7.59–7.61 (d, J = 8.4 Hz, 2H), 8.04–8.06 (d, J = 8.4 Hz, 2H), 8.21–8.23 (d, J = 8.4 Hz, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.4, 54.4, 66.3, 123.5, 127.0, 127.2, 128.7, 129.5, 129.9, 130.9, 132.5, 134.3, 134.6, 136.9, 143.5, 150.7, 164.5 ppm. MS (ESI): Calcd for $C_{22}H_{19}ClN_2O_6S$ + Na 497.0550, found 497.0550.

2-(4-Methylphenylsulfonamido)-1-(3-chlorophenyl)ethyl 4-nitrobenzoate (3q). White solid; mp 146–148°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.30 (s, 3H), 3.44–3.48 (m, 1H), 4.42–4.45 (m, 1H), 4.56–4.59 (m, 1H), 6.17–6.18 (d, J = 7.8 Hz, 1H), 7.06–7.07 (d, J = 7.8 Hz, 1H), 7.09–7.13 (m, 2H), 7.16–7.17 (m, 1H), 7.23–7.24 (d, J = 7.8 Hz, 1H), 7.27–7.28 (m, 1H), 7.55–7.57 (m, 1H), 7.68–7.70 (q, 1H), 8.04–8.05 (m, 1H), 8.15–8.17 (q, 3H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.4, 56.4, 674, 124.4, 125.0, 126.9, 127.2, 128.4, 129.5, 129.9, 130.1, 130.9, 134.6, 137.0, 138.8, 143.7, 150.6, 164.4 ppm. MS (ESI): Calcd for $C_{22}H_{19}ClN_2O_6S$ + Na 497.0550, found 497.0550.

Mixture of 3r and 4r. Inseparable white solid (**3r**:**4r** = 64:36); mp 134–137°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.32 (s, 3H), 2.40 (s, 2H), 3.44–3.48 (m, 1H), 4.41–4.49 (m, 2H), 4.74–4.77 (m, 1H), 5.63 (t, 1H), 5.97–5.99 (m, 1H), 6.13–6.14 (d, J = 7.8 Hz, 1H), 7.06–7.07 (d, J = 8.4 Hz, 2H), 7.14–7.15 (d, J = 8.4 Hz, 2H), 7.16–7.17 (m, 2H), 7.22–7.27 (m, 4H), 7.55–7.57 (d, J = 8.4 Hz, 2H), 7.67–7.68 (d, J = 8.4 Hz, 1H), 8.03–8.05 (d, J = 8.4 Hz, 2H), 8.17–8.18 (m, 4H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.5, 29.6, 47.4, 56.3, 67.4, 75.2, 123.5, 126.9, 127.9, 128.2, 128.9, 129.1, 129.5, 129.8, 130.9, 134.3, 134.6, 134.9, 135.0, 135.4, 136.8, 137.1, 143.7, 143.8, 150.7, 163.7, 164.4 ppm. MS (ESI): Calcd for $C_{22}H_{19}ClN_2O_6S$ + Na 497.0550, found 497.0556.

2-(4-Methylphenylsulfonamido)-1-(2-methoxyphenyl)ethyl 4-nitrobenzoate (3s). White solid; mp 150–152°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.38 (s, 3H), 3.44–3.49 (m, 1H), 3.53–3.57 (m, 1H), 3.82 (s, 3H), 4.95–4.97 (t, J = 6.4 Hz, 1H), 6.37–6.39 (q, 1H), 6.86–6.88 (m, 1H), 6.94–7.02 (m, 1H), 7.21–7.22 (d, J = 8.4 Hz, 2H), 7.27–7.31 (m, 2H), 7.67–7.68 (d, J = 8.4 Hz, 2H), 8.20–8.22 (m, 2H), 8.26–8.28 (m, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.5, 46.3, 55.8, 70.9, 110.8, 120.9, 123.5, 124.6, 126.5, 127.0, 129.2, 129.6, 130.9, 135.2, 137.3, 143.4, 150.7, 156.0, 163.7 ppm. MS (ESI): Calcd for $C_{23}H_{22}N_2O_7S$ + Na 493.1045, found 493.1044.

Mixture of 3t and 4t. Inseparable white solid (**3t**:**4t** = 75:25); mp 87–89°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.27 (s, 3H), 2.38 (s, 3H), 3.47–3.48 (m, 1H), 3.50–3.55 (m, 1H), 3.82 (s, 3H), 3.91 (s, 3H), 4.42–4.43 (m, 2H), 4.64–4.68 (m, 1H), 5.10–5.13 (m, 1H), 5.77–5.78 (d, J = 7.8 Hz, 1H), 5.92–5.95 (m, 1H), 6.37–6.39 (m, 1H), 6.74–6.78 (m, 1H), 6.87–6.93 (m, 1H), 6.93–7.03 (m, 5H), 7.19–7.25 (m, 3H), 7.27–7.31 (m, 3H), 7.52–7.54 (m, 2H), 7.67–7.68 (d, J = 8.4 Hz, 2H), 7.75–7.77 (m, 3H), 8.09–8.11 (d, J = 9.0 Hz, 1H), 8.20–8.26 (m, 6H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.4, 21.5, 47.6, 55.3, 67.7, 75.6, 112.4, 113.9, 118.5, 118.9, 123.5, 127.0, 129.5, 129.8, 130.1, 130.9, 134.8, 137.0, 137.3, 138.0, 138.3, 143.4, 143.7, 150.6, 159.9, 163.7, 164.5 ppm. MS (ESI): Calcd for $C_{23}H_{22}N_2O_7S$ + Na 493.1045, found 493.1047.

2-(4-Methylphenylsulfonamido)-1-(naphthalen-7-yl)ethyl 4-nitrobenzoate (3u). White solid; mp 184–186°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.35 (s, 3H), 3.53–3.63 (m, 2H), 5.36–5.38 (t, 1H), 6.15–6.17 (q, J = 4.2, 3.6 Hz, 1H), 7.15–7.16 (d, J = 8.4 Hz, 2H), 7.23–7.27 (m, 1H), 7.35–7.41 (dd, J = 8.4, 1.8 Hz, 1H), 7.47–7.49 (m, 2H), 7.50–7.56 (d, J = 8.4 Hz, 1H), 7.65–7.69 (d, J = 8.4 Hz, 2H), 7.76–7.81 (m, 3H), 8.16–8.21 (m, 3H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.5, 47.6, 75.9, 123.5, 126.1, 126.7, 126.7, 126.9. 127.7, 128.1, 128.9, 129.4, 129.8, 130.9, 133.0, 133.4, 133.7, 135.0, 136.9, 143.7, 150.6, 163.8 ppm. MS (ESI): Calcd for $C_{26}H_{22}N_2O_6S$ + Na 513.1096, found 513.1095.

2-(4-Methylphenylsulfonamido)-1-(naphthalen-5-yl)ethyl 4-nitrobenzoate (3v). White solid; mp 189–191°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.37 (s, 3H), 3.62–3.66 (m, 2H), 5.18–5.20 (t, J = 6.4 Hz, 1H), 6.79–6.81 (dd, J = 4.2, 3.6 Hz, 1H), 7.19–7.20 (d, J = 8.4 Hz, 2H), 7.42–7.44 (t, J = 7.8 Hz, 1H), 7.52–7.55 (m, 3H), 7.66–7.67 (d, J = 8.4 Hz, 2H), 7.81–7.83 (m, 1H), 7.87–7.88 (m, 1H), 7.98–8.00 (d, J = 8.4 Hz, 1H), 8.22–8.26 (m, 4H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 21.5, 47.2, 73.3, 122.4, 123.6, 124.0, 125.2, 126.2, 127.0, 127.0, 129.2, 129.5, 129.8, 130.0, 131.0, 132.2, 133.8, 135.0, 137.0, 143.7, 150.7, 163.9 ppm. MS (ESI): Calcd for $C_{26}H_{22}N_2O_6S$ + Na513.1096, found 513.1097.

Mixture of 3w and 4w. Inseparable white solid (**3w**:**4w** = 52:48); mp 152–156°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.32 (s, 3H), 2.40 (s, 3H), 3.46–3.48 (m, 2H), 4.41–4.43 (m, 1H), 4.46–4.49 (m, 1H), 4.71–4.75 (m, 1H), 5.50–5.53 (m, 1H), 5.95–5.96 (m, 1H), 6.02–6.03 (m, 1H), 7.06–7.07 (m, 4H), 7.19–7.22 (t, 2H), 7.24–7.27 (t, 2H), 7.33–7.34 (d, J = 8.4 Hz, 2H), 7.43–7.45 (d, J = 9.0 Hz, 2H), 7.54–7.56 (d, J = 7.8 Hz, 2H), 7.66–7.67 (d, J = 7.8 Hz, 2H), 8.04–8.05 (d, J = 8.4 Hz, 2H), 8.14–8.16 (d, J = 8.4 Hz, 2H), 8.18–8.21 (dd, J = 9.0, 7.2 Hz, 4H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 122.4, 123.1, 123.5, 126.9, 128.1, 128.5, 129.5, 129.8, 130.9, 131.9, 132.1, 134.5, 134.7, 135.5, 135.9, 136.8, 137.1, 143.7, 150.7, 163.6, 164.4 ppm. MS (ESI): Calcd for $C_{22}H_{19}BrN_2O_6$ + Na 541.0045, found 541.0041.

2-(4-Methylphenylsulfonamido)-1-phenylethyl 4-nitrobenzoate (3x). White solid; mp 143–145°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 2.32 (s, 3H), 4.43–4.45 (q, 1H), 4.48–4.51 (q, 1H), 4.76–4.79 (m, 1H), 5.80–5.82 (d, 1H), 7.08–7.13 (m, 3H), 7.19–7.20 (m, 1H), 7.27–7.28 (d, J = 8.4 Hz, 2H), 7.57–7.58 (d, J = 8.4 Hz, 2H), 8.06–8.07 (m, 2H) , 8.20–8.21 (m, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): 21.5, 47.7, 56.8, 67.8, 75.9, 123.4, 127.0, 128.8, 128.9, 129.5, 129.8, 130.9, 134.8, 135.0, 136.5, 136.8, 136.9, 137.3, 143.4, 143.7, 150.6, 163.8, 164.4 ppm. MS (ESI): Calcd for $C_{22}H_{20}N_2O_6S$ + Na 463.0940, found 463.0940.

2-(4-Methylphenylsulfonamido)octyl 4-nitrobenzoate (3y). White solid; mp 116–118°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 0.82–0.84 (t, J = 7.2 Hz, 3H), 1.13–1.20 (m, 8H), 1.46–1.53 (m, 2H), 2.35 (s, 3H), 3.60–3.63 (dd, J = 7.8, 4.8 Hz, 1H), 4.25–4.28 (m, 2H), 5.18–5.19 (d, J = 8.4 Hz, 1H), 7.20–7.22 (d, J = 7.8 Hz, 2H), 7.74–7.75 (d, J = 7.8 Hz, 2H), 8.11–8.12 (d, J = 9.0 Hz, 2H), 8.21–8.22 (d, J = 8.4 Hz, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 13.9, 21.4, 22.4, 25.4, 28.8, 31.5, 32.3, 52.9, 67.3, 123.4, 126.9, 129.7, 130.8, 135.0, 138.5, 143.5, 150.6, 164.5 ppm. MS (ESI): Calcd for $C_{22}H_{28}N_2O_6S$ + Na 471.1566, found 471.1562.

2-(4-Methylphenylsulfonamido)cyclopentyl 4-nitrobenzoate (3z). White solid; mp 143–145°C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 1.54–1.58 (m, 1H), 1.70–1.77 (m, 4H), 2.08–2.13 (m, 1H), 2.29 (s, 3H), 3.70–3.74 (m, 1H), 5.13–5.16 (m, 1H), 5.37–5.38 (d, J = 6.4 Hz, 1H), 7.14–7.16 (d, J = 7.8 Hz, 2H), 7.72–7.73 (d, J = 7.8 Hz, 2H), 8.04–8.05 (m, 2H), 8.24–8.26 (m, 2H) ppm. ^{13}C-NMR (150 MHz, $CDCl_3$): δ 20.7, 21.4, 29.5, 31.0, 59.6, 81.0, 123.4, 127.1, 129.6, 130.8, 135.2, 137.4, 143.4, 150.6, 164.4 ppm. MS (ESI): Calcd for $C_{19}H_{20}N_2O_6S$ + Na 427.0940, found 427.0938.

Acknowledgment. We appreciate gratefully the Natural Science Foundation of Shanxi Province (No. 2012021007-2 and No. 2011011010-2) for financial support.

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[63] Instead of 0.24 mmol of CH_3COOH and 40 mol% KOH, 0.24 mmol of CH_3COOK was added to perform this reaction. Unfortunately, no corresponding reaction product was observed within 24 h.