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RESEARCH LETTER

Convenient synthesis of sulfonyl azides using PEG-400 as an efficient and eco-friendly reaction medium

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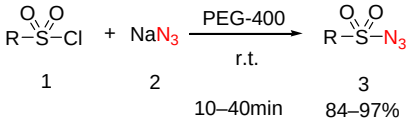
Sulfonyl azides have efficiently been synthesized via a convenient and environmentally benign procedure, in which sulfonyl chlorides undergo nucleophilic substitution reaction with sodium azide in PEG-400 under mild conditions. The sulfonyl azides were obtained in 84–97% isolated yields.



R= alkyl, aryl, cinnamyl and heteroaryl

Keywords: sulfonyl azides; PEG-400; reaction medium; sodium azide

Introduction

Sulfonyl azides are very valuable reagents in organic chemical transformations such as the preparation of α-diazocarbonyl compounds [1], the hydro-hydrazination or/and hydroazidation of olefins [2,3], the aziridination of olefins [4], the radical amination [5,6], and metal-catalyzed coupling reactions [7]. Due to a wide range of applications, there are many methods available for the preparation of sulfonyl azides. For example, sulfonyl azides were prepared by reacting sulfonyl anhydrides [8], α-disulfones [9], or 1-sulfonylbenzotriazole [10] with sodium azide. These procedures may suffer from the unavailability of starting materials or their difficulty in preparation. Additionally, diazotization of sulfonyl hydrazides with NO^+ has also been employed but still requires the availability of the hydrazides [11]. However, the most practical laboratory methods for preparing sulfonyl azides by nucleophilic substitution reaction of sulfonyl chlorides with sodium azide in various solvents are such as alcohol/H_2O, acetone/H_2O, DME/H_2O, and so on [3,12–17]. Since nucleophilic substitution reactions of sulfonyl chloride involve a nonpolar organic compound and a polar ionic salt, sodium azide, the heterogenous reactions are often troublesome because the polar and nonpolar reagents are often not soluble in a single solvent system. Consequently, to improve the yields and to facilitate the product isolation, the nucleophilic displacement reactions are carried out under phase-transfer catalysis conditions [18,19]. However, these methodologies often suffer from complex procedures, long reaction times, and low yields. Thus, there is a great demand for the development of new convenient and eco-friendly synthetic methods toward assessing sulfonyl azides.

In the recent years, polyethylene glycols (PEGs) have attracted great interest and have been explored as a novel, powerful, eco-friendly reaction medium for various organic transformations [20–25] due to their relatively inexpensive, thermally stable, readily recyclable, and biodegradable. In a continuation of our work [20] to explore PEG as an efficient and eco-friendly reaction medium, we report here a convenient and practical synthesis of sulfonyl azides by using sodium azide in PEG-400 at room temperature (Scheme 1).

Results and discussion

Initially, we examined the effectivity of PEG-400 for the model reaction of 4-Tosyl chloride and sodium azide (Entry 4, Table 1). In a typical experimental

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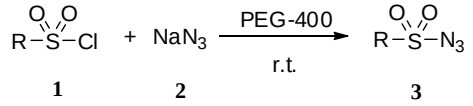


1 2 3

Scheme 1. Synthesis of sulfonyl azides from sulfonyl chlorides.

procedure, a screening of different solvents (CH_3CN, THF, CH_2Cl_2, toluene, and so on) for the model reaction revealed that PEG-400 was the most active reaction medium.

To investigate the generality and scope of the reaction, various sulfonyl chlorides were subjected to the reaction conditions and no additional catalyst and solvent were required. The results are summarized in Table 1. As presented in Table 1, all aryl and aliphatic sulfonyl chlorides gave sulfonyl azides in excellent yields in 10–40 minutes. Aryl sulfonyl chlorides containing both electron-donating, such as methyl, methoxyl, and electron-withdrawing groups, like nitro, acetamido, underwent the conversion smoothly. Aryl sulfonyl chlorides with electron-withdrawing groups such as NO_2 required slightly more long time (Entries 8–10, Table 1). With more sterically hindered sulfonyl chlorides, satisfactory yields were still obtained from the nucleophilic substitution (Entries 5, 6, and 10, Table 1). 2-Nitrobenzenesulfonyl chloride took the longest time caused by the electronic and steric hindrance effect (Entry 10, Table 1). The presence of various functional groups such as halides, nitro, acetamino, and methoxyl on the aryl sulfonyl chlorides was tolerated (Entries 7–13, Table 1). *Trans*-β-styrenesulfonyl chloride and 2-thiophenesulfonyl chloride have also been successfully converted into their corresponding sulfonyl azides in high yields (Entries 14 and 15, Table 1). In short, the products were all formed in excellent yields and no side products were detected. The structures of all products were identified by their physical and spectral data. Infrared spectra of all compounds have strong characteristic band at 2120–2160 cm^{-1} (N_3), 1310–1370 cm^{-1}, and 1100–1170 cm^{-1} (SO_2).

Experimental

All reagents were purchased from commercial sources unless otherwise stated. Petroleum ether/ethyl acetate (8:1) (TLC) was carried out on silica gel 60 F_{254} precoated plates (0.20–0.25 mm thickness) and visualized with UV light (254 nm). Melting points were determined with X-6 (Beijing Fukai Co. Ltd.) melting point apparatus and were uncorrected. ^1H NMR and ^{13}C NMR (600 and 150 MHz, respectively) spectra were recorded in $CDCl_3$. ^1H NMR chemical shifts are reported in ppm (δ) relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard ($CDCl_3$, δ 7.26 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, and m = multiplet), coupling constants (Hz), and integration. ^{13}C NMR chemical shifts are reported in ppm from TMS with the solvent resonance as the internal standard ($CDCl_3$, δ 77.0 ppm). Compounds **1l** [27], **1m** [27], and **1n** [28] were prepared by following the reported methods.

Typical procedure for synthesis of sulfonyl azides

A typical experimental procedure is as follows: a mixture of sulfonyl chloride (2 mmol) and NaN_3 (2.4 mmol) in PEG-400 (2 mL) was vigorously stirred at room temperature for the appropriate time (Table 1) until TLC indicated total disappearance of sulfonyl chloride. After completion, the reaction mixture was poured into water and extracted with dry ether. The organic layer was removed under reduced pressure and afforded pure sulfonyl azides in excellent yield [Caution: Sufficient care has to be exercised while treating organic azides because of their explosive nature]. The crude products were generally sufficient purity to be used without further purification. The pure compounds can also be obtained by flash silica gel column chromatography with petroleum ether/ethyl acetate (8:1) or crystallized from methanol. The PEG-400 was recovered from the aqueous layer and reused without loss of activity.

Characterization data of selected known compounds and new compound

The products are all known except **3m** and were identified by comparing their physical and spectral data with literature values. Spectral data for selected and new compounds are described in the following subsections.

1-Butanesulfonyl azide (3a)

Pale yellow liquid. ^1H NMR ($CDCl_3$): δ_H 0.99 (t, J = 7.4 Hz, 3H), 1.48–1.57 (m, 2*H*), 1.88–1.93 (m, 2*H*), 3.32 (t, J = 7.9 Hz, 2*H*). ^{13}C NMR ($CDCl_3$): δ_C 13.4, 21.3, 25.3, 55.7. IR (KBr), ν|cm^{-1}: 3306, 2965, 2877, 2378, 2136, 1467, 1364, 1242, 1198, 1159, 1100, 1079, 917, 794, 735.

Phenylmethanesulfonyl azide (3b)

Colorless solid, m.p. 53–54 °C. ^1H NMR ($CDCl_3$): δ_H 4.53 (s, 2*H*), 7.43–7.48 (m, 5*H*). ^{13}C NMR ($CDCl_3$): δ_C 61.9, 126.6, 129.3, 129.9, 130.9. IR (KBr), ν|cm^{-1}:

Table 1. Synthesis of sulfonyl azides from sulfonyl chlorides using PEG-400 as an efficient reaction medium.[a]

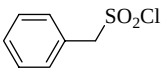
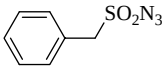
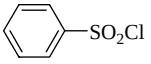
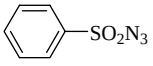
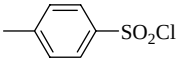
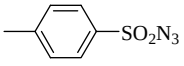
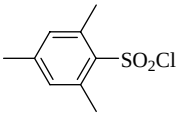
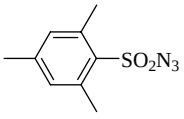
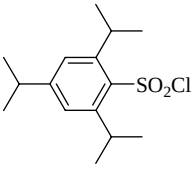
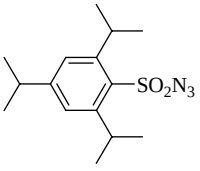
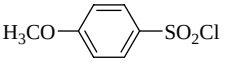
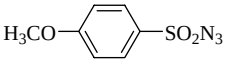
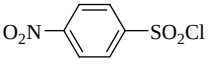
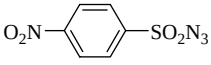
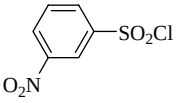
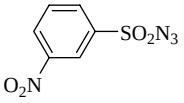
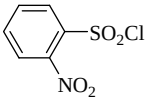
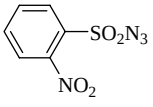
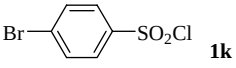
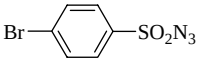
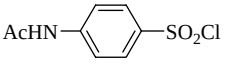
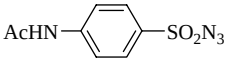
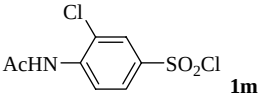
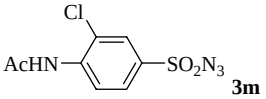
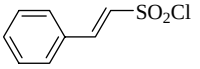
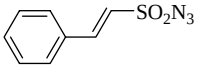
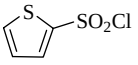
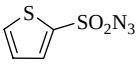
Entry	Sulfonyl chloride	Sulfonyl azide	Time (mins)	Yield[b]	m.p. (°C) Found	m.p. (°C) Reported[c]
1	$CH_3(CH_2)_2SO_2Cl$ **1a**	$CH_3(CH_2)_2SO_2N_3$ **3a**	10	94	Oil	Oil [10]
2	 **1b**	 **3b**	10	97	53–54	53.5–54 [26]
3	 **1c**	 **3c**	10	96	13–14	13.5–14.5 [11]
4	 **1d**	 **3d**	10	94	22–23	22.5–23.5 [11]
5	 **1e**	 **3e**	10	90	Oil	Oil [7]
6	 **1f**	 **3f**	30	84	42–43	41–43 [14]
7	 **1g**	 **3g**	10	95	50–51	51.5–52 [17]
8	 **1h**	 **3h**	30	90	100–101	101.5–102 [17]
9	 **1i**	 **3i**	30	95	78–79	80.5–81 [17]
10	 **1j**	 **3j**	40	85	67–68	68–71 [15]
11	 **1k**	 **3k**	10	97	54–55	54.5–56 [17]
12	 **1l**	 **3l**	10	93	107–108	108–110 [16]

Table 1 (*Continued*)

Entry	Sulfonyl chloride	Sulfonyl azide	Time (mins)	Yield[b]	m.p. (°C) Found	m.p. (°C) Reported[c]
13	 **1m**	 **3m**	10	92	95–97	–
14	 **1n**	 **3n**	30	91	31–32	31.5–33 [26]
15	 **1o**	 **3o**	10	90	30–31	30–32 [10]

[a]Reaction conditions: sulfonyl chloride (2.0 mmol), sodium azide (2.4 mmol), and PEG-400 (2 mL) at room temperature.
[b]Isolated yield.
[c]The compound reported in the literature.

3436, 3294, 2979, 2137, 1599, 1496, 1456, 1407, 1355, 1270, 1179, 1159, 1136, 1031, 884, 793, 748.

Benzenesulfonyl azide (3c)

Colorless solid, m.p. 13–14 °C. ^{1}H NMR ($CDCl_3$): δ_H 7.63 (t, $J=7.5$, $J=7.9$ Hz, 2*H*), 7.74 (t, $J=7.5$ Hz, 1*H*), 7.97 (d, $J=7.9$ Hz, 2*H*). ^{13}C NMR ($CDCl_3$): δ_C 127.5, 129.7, 134.8, 138.5. IR (KBr), $\nu|cm^{-1}$: 3273, 3069, 2921, 2128, 1732, 1583, 1449, 1373, 1313, 1170, 1087, 1020, 930, 753.

4-Toluenesulfonyl azide (3d)

Colorless solid, m.p. 22–23 °C. ^{1}H NMR ($CDCl_3$): δ_H 2.48 (s, 3*H*), 7.41 (d, $J=8.1$ Hz, 2*H*), 7.84 (d, $J=8.3$ Hz, 2*H*). ^{13}C NMR ($CDCl_3$): δ_C 21.7, 127.5, 130.3, 135.6, 146.2. IR (KBr), $\nu|cm^{-1}$: 3273, 3067, 2926, 2127, 1595, 1494, 1450, 1371, 1308, 1167, 1121, 1086, 1018, 814, 748.

2,4,6-Trimethylbenzene-1-sulfonyl azide (3e)

Tan liquid. ^{1}H NMR ($CDCl_3$): δ_H 2.3 (s, 3*H*), 2.67 (s, 6*H*), 7.02 (s, 2*H*). ^{13}C NMR ($CDCl_3$): δ_H 21.1, 22.7, 132.2, 133.3, 139.9, 144.6. IR (KBr), $\nu|cm^{-1}$: 3276, 2981, 2924, 2382, 2122, 1602, 1566, 1455, 1366, 1291, 1191, 1166, 1051, 965, 854, 745.

2,4,6-Triisopropylbenezensulfonyl azide (3f)

Colorless solid, m.p. 42–43 °C. ^{1}H NMR ($CDCl_3$): δ_H 1.26 (d, $J=7.1$ Hz, 6*H*), 1.30 (dd, $J=6.6$, $J=6.8$ Hz, 12*H*), 2.91–2.96 (m, 1*H*), 4.22–4.26 (m, 2*H*), 7.22 (s, 2*H*). ^{13}C NMR ($CDCl_3$): δ_C 23.4, 24.3, 24.7, 29.7, 29.8, 34.3, 124.1, 124.3, 139.3, 150.4, 150.9, 155.6. IR (KBr), $\nu|cm^{-1}$: 3435, 3055, 2961, 2930, 2870, 2121, 1598, 1462, 1434, 1385, 1378, 1364, 1350, 1261, 1175, 1104, 1059, 889, 802, 740.

4-Methoxysulfonyl azide (3g)

Colorless solid, m.p. 50–51 °C. ^{1}H NMR ($CDCl_3$): δ_H 3.91 (s, 3*H*), 7.05 (d, $J=8.9$ Hz, 2*H*), 7.90 (d, $J=8.9$ Hz, 2*H*). ^{13}C NMR ($CDCl_3$): δ_C 55.9, 114.8, 128.6, 129.9, 164.6. IR (KBr), $\nu|cm^{-1}$: 3273, 3096, 2984, 2129, 1590, 1495, 1369, 1318, 1266, 1187, 1163, 1110, 1085, 1020, 832, 805, 744.

4-Nitrobenzenesulfonyl azide (3h)

Tan solid, m.p. 100–101 °C. ^{1}H NMR ($CDCl_3$): δ_H 8.17 (d, $J=8.8$ Hz, 2*H*), 8.46 (d, $J=8.8$ Hz, 2*H*). ^{13}C NMR ($CDCl_3$): δ_H 124.9, 128.9, 143.8, 151.0. IR (KBr), $\nu|cm^{-1}$: 3107, 2920, 2143, 1606, 1531, 1377, 1350, 1311, 1178, 1160, 1085, 854, 769, 744.

2-Nitrobenzenesulfonyl azide (3j)

Tan solid, m.p. 68–71 °C. ^{1}H NMR ($CDCl_3$): δ_H 7.83 (ddd, 1*H*), 7.88 (ddd, 1*H*), 7.92 (dd, 1*H*), 8.20 (dd, 1*H*). ^{13}C NMR ($CDCl_3$): δ_H 125.4, 131.7, 132.7, 133.0, 135.7. IR (KBr), $\nu|cm^{-1}$: 3320, 3100, 2923, 2381, 2157, 1594, 1550, 1438, 1363, 1315, 1261, 1194, 1145, 1120, 967, 853, 755, 737.

4-Bromobenzenesulfonyl azide (3k)

White solid, m.p. 78–79 °C. ^{1}H NMR ($CDCl_3$): δ_H 7.76 (d, $J=8.4$ Hz, 2*H*), 7.82 (d, $J=8.6$ Hz, 2*H*). ^{13}C NMR ($CDCl_3$): δ_C 128.9, 130.3, 133.1, 137.5. IR (KBr), $\nu|cm^{-1}$: 3246, 3094, 2923, 2148, 1571, 1470, 1392, 1376, 1168, 1083, 1064, 1008, 819, 770, 732.

4-Acetamidobenzenesulfonyl azide (3l)

White solid, m.p. 107–108 °C. ^{1}H NMR ($CDCl_3$): δ_H 2.25 (s, 1*H*), 7.78 (d, $J=9.0$ Hz, 2*H*), 7.79 (brs, 1*H*), 7.89 (d, $J=8.8$ Hz, 2*H*). ^{13}C NMR ($CDCl_3$): δ_C 24.7, 119.6, 129.6, 129.0, 132.7, 143.9, 168.9. IR (KBr), $v|cm^{-1}$: 3303, 3264, 3185, 3112, 2130, 2120, 1676, 1585, 1534, 1405, 1365, 1315, 1265, 1165, 1086, 839, 752, 707.

3-Chloro-4-acetamidobenzenesulfonyl azide (3m)

White solid, m.p. 95–97 °C. ^{1}H NMR ($CDCl_3$): δ_H 2.32 (s, 3*H*), 7.85 (dd, $J=2.2$, $J=9.0$ Hz, 1*H*), 7.87 (brs, 1*H*), 7.97 (d, $J=2.2$ Hz, 1*H*), 8.74 (d, $J=8.8$ Hz, 1*H*). ^{13}C NMR ($CDCl_3$): δ_C 25.1, 120.9, 122.6, 127.5, 128.3, 133.2, 140.3, 168.5. IR (KBr), $v|cm^{-1}$: 3401, 3119, 3070, 2340, 2134, 1712, 1575, 1505, 1392, 1375, 1306, 1171, 1098, 857, 837, 778, 745.

Trans-β-styrenesulfoyl azide (3n)

White solid, m.p. 31–32 °C. ^{1}H NMR ($CDCl_3$): δ_H 6.94 (d, $J=15.3$ Hz, 1*H*), 7.45–7.47 (m, 2*H*), 7.50 (m, 1*H*), 7.53–7.55(m, 2*H*), 7.70 (d, $J=15.3$ Hz, 1*H*). ^{13}C NMR ($CDCl_3$): δ_C 123.2, 126.8, 128.9, 129.4, 129.6, 129.8, 131.3, 132.2. IR (KBr), $v|cm^{-1}$: 3292, 3065, 2345, 2130, 1725, 1610, 1576, 1495, 1450, 1368, 1180, 1154, 1107, 1074, 975, 863, 821, 749.

2-Thiophenesulfonyl azide (3o)

Pale yellow, m.p. 30–31 °C. ^{1}H NMR ($CDCl_3$): δ_H 7.21 (dd, $J=3.9$, $J=4.9$ Hz, 1*H*), 7.80 (dd, $J=1.4$, $J=4.9$ Hz, 1*H*), 7.21 (dd, $J=1.4$, $J=3.9$ Hz, 1*H*). ^{13}C NMR ($CDCl_3$): δ_C 128.0, 134.7, 135.1, 138.2. IR (KBr), $v|cm^{-1}$: 3271, 3101, 2129, 1754, 1601, 1504, 1400, 1378, 1345, 1167, 1094, 1019, 857, 757, 746.

Conclusion

In summary, we have disclosed a simple, mild, and efficient method for the synthesis of sulfonyl azides. Compared to the previously reported methods, this protocol offers several advantages including exceedingly mild conditions, operational simplicity, more environmentally benign, short reaction time, and higher reaction yield. Further investigations on the application of PEG-400 on other catalytically synthetic reactions will be reported in due course.

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