

# An efficient and convenient procedure for preparation of *N*-sulfonylimines catalysed by TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> solid superacid

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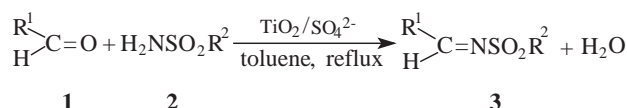
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A facile synthesis of *N*-sulfonylimines in good to excellent yields was described from aldehydes with sulfonamides catalysed by TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> solid superacid.

**Keywords:** aldehydes, sulfonamides, TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> solid superacid, *N*-sulfonylimines

During the past several years the synthesis and application of *N*-sulfonylimines from aldehydes with sulfonamides has been found to be very useful in organic chemistry. Because *N*-sulfonylimines are powerful synthetic intermediates<sup>1,2</sup> and they are also used in numerous reactions such as inverse electron-demand Diels–Alder reactions,<sup>3-5</sup> addition reactions as carbonyl equivalents<sup>6</sup> and in ene reactions.<sup>7</sup> Several synthetic methods for the preparation of *N*-sulfonylimines have been reported in the literature, for example, the rearrangement of oxime *O*-sulfonates,<sup>8</sup> Lewis acid or solid acid catalysed reactions of sulfonamides with aldehydes or acetals,<sup>9-12</sup> utilisation of tellurium metal and chloramines T,<sup>13</sup> addition of *N*-sulfinyl sulfonamides to aldehydes in the presence of BF<sub>3</sub>·Et<sub>2</sub>O,<sup>14</sup> using methyl orthosilicate,<sup>15</sup> halogen-mediated conversion of *N*-(trimethylsilyl)imines in the presence of corresponding sulfonyl chloride<sup>16</sup> and catalysed by HCOOH and NaHCO<sub>3</sub>.<sup>17</sup> Some of these methods have not been entirely satisfactory having such drawbacks as long reaction times, expensive and hazardous reagents, cumbersome experimental and requiring the use of a microwave oven.

TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> solid superacid have been used as an efficient catalyst for a variety of organic reactions.<sup>18,19</sup> Recently, we have developed easy and efficient procedures for the preparation of some organic products catalysed by TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> solid




**Scheme 1**

superacid.<sup>20-22</sup> As a part of ongoing work on solid superacid catalysis, we now describe a facile and efficient method for the synthesis of *N*-sulfonylimines from aldehydes with sulfonamides catalysed by TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> solid superacid (Scheme 1).

Representative results of this study are summarised in Table 1. As shown in Table 1, aromatic aldehydes as well as aliphatic aldehydes in the presence of TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> were heated with sulfonamide in refluxing toluene, the corresponding *N*-sulfonylimines were obtained in good to excellent yield. It is believed that the products have the thermodynamically favoured *E*-structure.<sup>16</sup> The reaction proceeded cleanly and work-up was simple, involving only filtration of the catalyst and removal of solvent to obtain the product in high purity.

It should be noted that in the absence of catalyst lower yields of product were observed even with prolonged reaction time. For example, for entry 2 without catalyst after 2 hours only 32% yield of product was obtained in refluxing toluene, whereas 93% yield was obtained with catalyst for 0.5h.

**Table 1** Conversion of aldehydes into *N*-sulfonylimines in the presence of TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup>

Entry	R <sup>1</sup>	R <sup>2</sup>	Product	Time/min	Yields <sup>a</sup> /%	M.p./°C	
						Found	Reported
1	C <sub>6</sub> H <sub>5</sub>	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3a</b>	40	96	114	110 <sup>16</sup>
2	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>	<b>3b</b>	30	93	139–140	139–141 <sup>10</sup>
3	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3c</b>	70	96	116–118	
4	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	<b>3d</b>	50	86	136	
5	4-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3e</b>	65	95	128–129	128–129 <sup>13</sup>
6	4-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	<b>3f</b>	70	90	145	147–149 <sup>10</sup>
7	2-HOC <sub>6</sub> H <sub>4</sub>	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3g</b>	70	91	125	
8	2-HOC <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	<b>3h</b>	80	87	119–120	
9	3,4-(OCH <sub>2</sub> O)C <sub>6</sub> H <sub>3</sub>	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3i</b>	25	92	121	114–116 <sup>13</sup>
10	4-ClC <sub>6</sub> H <sub>4</sub>	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3j</b>	60	70	172–173	
11	4-ClC <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	<b>3k</b>	65	69	128–129	127–130 <sup>10</sup>
12	3-ClC <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	<b>3l</b>	50	90	102–104	
13	2,4-Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3m</b>	60	73	112–113	
14	3-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3n</b>	80	56	144–145	143–145 <sup>10</sup>
15	C <sub>6</sub> H <sub>5</sub> CH=CH	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3o</b>	30	97	110	108–109 <sup>16</sup>
16	C <sub>6</sub> H <sub>5</sub> CH=CH	C <sub>6</sub> H <sub>5</sub>	<b>3p</b>	25	95	112–113	
17		4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	<b>3q</b>	30	93	101–103	101–102 <sup>13</sup>

<sup>a</sup>Yield of pure isolated products.

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† This is a Short Paper, there is therefore no corresponding material in *J Chem. Research (M)*.

The reaction worked better in refluxing toluene than in refluxing benzene, which may be due to its higher boiling temperature than that of benzene.

We were pleased to find that the conversion rate of *p*-toluenesulfonamide with aldehydes was a little higher than benzene sulfonamide and the aldehydes with electron-withdrawing groups (4-chlorobenzaldehyde, 2,4-dichlorobenzaldehyde and 3-nitrobenzaldehyde) provided lower conversion rates (especially for 3-nitrobenzaldehyde, <60%) than the aldehydes with donating group (4-tolualdehyde, anisaldehyde, salicylaldehyde and piperonal), this indicated that electron-donating groups had increased reaction yields. On the other hand electron-withdrawing groups had decreased yields.

Mention must be made here that the yields of products from **3j** to **3n** were lower except for **3l**. The explanation for this result may be due to the 3-nitrobenzaldehyde with a strongly electron-withdrawing nitro group and the aldehydes (4-chlorobenzaldehyde and 2,4-dichlorobenzaldehyde) with weakly electron-withdrawing chloro groups situated *ortho* or *para* which will reduce activity more than the 3-chlorobenzaldehyde with the weakly electron-withdrawing chloro group situated *meta*. We also found that the reaction yield of 4-chlorobenzaldehyde (1.00 mmol) with 4-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NH<sub>2</sub> (1.00 mmol) was 70% in refluxing toluene for 60 mins but it increased to 91% from 4-chlorobenzaldehyde (1.00 mmol) with 4-CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NH<sub>2</sub> (1.50 mmol) on refluxing for 30 minutes.

The catalyst was easily regenerated by washing with ethanol followed by drying at 100°C for 4 h. The catalyst could be reused five times for the synthesis *N*-(*p*-toluenesulfonyl)benzaldimine (**3a**) without significant loss of activity.

In conclusion, we have provided a facile and efficient method for the synthesis of *N*-sulfonylimine catalysed by TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> solid superacid. This method possesses these advantages of the operational simplicity, short reaction times, use of inexpensive materials, lack of corrosion and pollution and high yields.

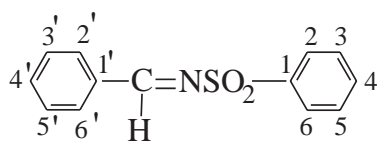
## Experimental

Melting points are uncorrected. IR spectra were recorded on a BIO-RAD FTS-40 IR spectrometer using KBr pellets. <sup>1</sup>H NMR spectra were determined on a Varian VXP-400s spectrometer using CDCl<sub>3</sub> as solvent and tetramethylsilane (TMS) as internal reference. CHN analyses were recorded on a Perkin-Elmer 2400 CHN elemental analyser. The products were also characterised by comparison of their melting points with literature values. The catalyst TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> solid superacid was prepared as follows. Ti(OH)<sub>4</sub> was obtained by hydrolysing TiCl<sub>4</sub> with aqueous ammonium hydroxide, washing the precipitates, drying them at 120°C for 4 h, and powdering the precipitates below a 100 mesh. The hydroxide was treated with 1M H<sub>2</sub>SO<sub>4</sub> for 4 h, filtering, drying, calcined in furnace at 475°C for 4 h, and finally stored in a desiccator until use.

**General procedure for the preparation of *N*-sulfonylimines.** A mixture of aldehyde (1.00 mmol), sulfonamide (1.00 mmol), TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> (50 mg) and toluene (5 ml) was stirred under refluxing for 25–80 min (Table 1) in a Dean-Stark apparatus. The reaction was monitored by TLC. After completion of the reaction, the reaction mixture was cooled and TiO<sub>2</sub>/SO<sub>4</sub><sup>2-</sup> was filtered off with toluene washing. The solvent was evaporated under reduced pressure to provide the crude product and the crude product was purified by recrystallisation from hexane-ethyl acetate mixtures or by flash chromatography on silica gel (Et<sub>2</sub>O-hexane as eluent) to give the pure product.

Spectroscopic and analytical data for the products:

The number of carbon atom in aryl like Scheme 2.



Scheme 2

**3a:** IR (KBr):  $\nu_{\max}$  3008, 2948, 1652, 1600, 1576, 1454, 1350, 1160, 816, 750, 682 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  9.03 (1H, s, HC=N), 7.92 (2H, d, *J*=7.6 Hz, 2',6'-ArH), 7.89 (2H, d, *J*=8.0 Hz, 2,6-SO<sub>2</sub>ArH), 7.60 (1H, t, *J*=7.6 Hz, 4'-ArH), 7.48 (2H, t, *J*=7.6 Hz, 3',5'-ArH), 7.34 (2H, d, *J*=8.0 Hz, 3,5-SO<sub>2</sub>ArH), 2.43 (3H, s, CH<sub>3</sub>); Anal. Calcd. for C<sub>14</sub>H<sub>13</sub>NO<sub>2</sub>S (259.33): C, 64.84; H, 5.05; N, 5.40. Found: C, 64.82; H, 5.10; N, 5.36.

**3c:** IR (KBr):  $\nu_{\max}$  3010, 2920, 1652, 1596, 1580, 1490, 1326, 1164, 844, 820 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  9.00 (1H, s, HC=N), 7.90 (2H, d, *J*=8.0 Hz, 2,6-SO<sub>2</sub>ArH), 7.83 (2H, d, *J*=7.6 Hz, 2',6'-ArH), 7.35 (2H, d, *J*=8.0 Hz, 3,5-SO<sub>2</sub>ArH), 7.30 (2H, d, *J*=7.6 Hz, 3',5'-ArH), 2.45 (6H, s, CH<sub>3</sub>); Anal. Calcd. for C<sub>15</sub>H<sub>15</sub>NO<sub>2</sub>S (273.35): C, 65.91; H, 5.53; N, 5.12. Found: C, 65.87; H, 5.64; N, 5.10.

**3d:** IR (KBr):  $\nu_{\max}$  3012, 2956, 1646, 1592, 1558, 1496, 1330, 1168, 816, 772, 704 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  9.04 (1H, s, HC=N), 8.02 (2H, d, *J*=7.6 Hz, 2,6-SO<sub>2</sub>ArH), 7.84 (2H, d, *J*=8.0 Hz, 2',6'-ArH), 7.65 (1H, t, *J*=7.6 Hz and *J*=7.6 Hz, 4-SO<sub>2</sub>ArH), 7.57 (2H, t, *J*=7.6 Hz and *J*=7.6 Hz, 3,5-SO<sub>2</sub>ArH), 7.31 (2H, d, *J*=8.0 Hz, 3',5'-ArH), 2.45 (3H, s, CH<sub>3</sub>); Anal. Calcd. for C<sub>14</sub>H<sub>13</sub>NO<sub>2</sub>S (259.33): C, 64.84; H, 5.05; N, 5.40. Found: C, 64.83; H, 5.10; N, 5.34.

**3e:** IR (KBr):  $\nu_{\max}$  3010, 2932, 1668, 1596, 1580, 1506, 1320, 1154, 1090, 834, 800 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  8.95 (1H, s, HC=N), 7.91 (2H, d, *J*=8.0 Hz, 2,6-SO<sub>2</sub>ArH), 7.88 (2H, d, *J*=7.6 Hz, 2',6'-ArH), 7.34 (2H, d, *J*=8.0 Hz, 3,5-SO<sub>2</sub>ArH), 6.98 (2H, d, *J*=7.6 Hz, 3',5'-ArH), 3.92 (3H, s, OCH<sub>3</sub>), 2.45 (3H, s, CH<sub>3</sub>); Anal. Calcd. for C<sub>15</sub>H<sub>15</sub>NO<sub>2</sub>S (289.35): C, 62.26; H, 5.22; N, 4.84. Found: C, 62.19; H, 5.30; N, 4.86.

**3f:** IR (KBr):  $\nu_{\max}$  3030, 2920, 1674, 1594, 1560, 1510, 1325, 1160, 1078, 808, 766, 692 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  8.98 (1H, s, HC=N), 8.01 (2H, d, *J*=7.6 Hz, 2,6-SO<sub>2</sub>ArH), 7.91 (2H, d, *J*=8.8 Hz, 2',6'-ArH), 7.63 (1H, t, *J*=7.2 Hz and *J*=7.2 Hz, 4-SO<sub>2</sub>ArH), 7.55 (2H, dd, *J*=7.6 Hz and *J*=7.2 Hz, 3,5-SO<sub>2</sub>ArH), 6.98 (2H, d, *J*=8.8 Hz, 3',5'-ArH), 3.93 (3H, s, OCH<sub>3</sub>); Anal. Calcd. for C<sub>14</sub>H<sub>13</sub>NO<sub>2</sub>S (275.33): C, 61.07; H, 4.76; N, 5.09. Found: C, 61.02; H, 4.80; N, 5.06.

**3g:** IR (KBr):  $\nu_{\max}$  3360, 3046, 2920, 1666, 1616, 1600, 1554, 1300, 1172, 1094, 810, 742 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  10.84 (1H, s, -OH), 9.10 (1H, s, HC=N), 7.87 (2H, d, *J*=8.0 Hz, 2,6-SO<sub>2</sub>ArH), 7.52 (2H, m, 4',6'-ArH), 7.36 (2H, d, *J*=8.0 Hz, 3,5-SO<sub>2</sub>ArH), 7.01 (2H, m, 3',5'-ArH), 2.45 (3H, s, CH<sub>3</sub>); Anal. Calcd. for C<sub>14</sub>H<sub>13</sub>NO<sub>2</sub>S (275.33): C, 61.07; H, 4.76; N, 5.09. Found: C, 61.05; H, 4.81; N, 5.09.

**3h:** IR (KBr):  $\nu_{\max}$  3320, 3020, 1658, 1600, 1550, 1494, 1342, 1160, 1092, 758, 692 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  10.83 (1H, s, -OH), 9.14 (1H, s, HC=N), 8.01 (2H, d, *J*=8.0 Hz, 2,6-SO<sub>2</sub>ArH), 7.68 (1H, t, *J*=8.0 Hz, 4-SO<sub>2</sub>ArH), 7.59 (2H, m, 4',6'-ArH), 7.54 (2H, dd, *J*=8.0 Hz and *J*=8.0 Hz, 3,5-SO<sub>2</sub>ArH), 7.03 (2H, m, 3',5'-ArH); Anal. Calcd. for C<sub>13</sub>H<sub>11</sub>NO<sub>2</sub>S (261.31): C, 59.75; H, 4.25; N, 5.36. Found: C, 59.71; H, 4.35; N, 5.29.

**3j:** IR (KBr):  $\nu_{\max}$  3030, 2932, 1658, 1604, 1552, 1500, 1332, 1160, 834, 800 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  9.01 (1H, s, HC=N), 7.90 (2H, d, *J*=7.6 Hz, 2,6-SO<sub>2</sub>ArH), 7.87 (2H, d, *J*=8.0 Hz, 2',6'-ArH), 7.48 (2H, d, *J*=8.0 Hz, 3',5'-ArH), 7.37 (2H, d, *J*=7.6 Hz, 3,5-SO<sub>2</sub>ArH), 2.46 (3H, s, CH<sub>3</sub>); Anal. Calcd. for C<sub>14</sub>H<sub>12</sub>NO<sub>2</sub>SCI (293.77): C, 57.24; H, 4.12; N, 4.77. Found: C, 57.19; H, 4.18; N, 4.69.

**3l:** IR (KBr):  $\nu_{\max}$  3084, 1680, 1606, 1566, 1502, 1345, 1168, 794, 756, 729, 694 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  9.03 (1H, s, HC=N), 8.03 (2H, d, *J*=7.6 Hz, 2,6-SO<sub>2</sub>ArH), 7.97 (1H, s, 2'-ArH), 7.81 (1H, d, *J*=7.6 Hz, 6'-ArH), 7.68 (1H, d, *J*=7.6 Hz, 4'-ArH), 7.59 (3H, m, 3,4,5-SO<sub>2</sub>ArH), 7.47 (1H, t, 5'-ArH); Anal. Calcd. for C<sub>13</sub>H<sub>10</sub>NO<sub>2</sub>SCI (279.75): C, 55.81; H, 3.60; N, 5.01. Found: C, 55.77; H, 3.68; N, 4.99.

**3m:** IR (KBr):  $\nu_{\max}$  3106, 2898, 1694, 1596, 1540, 1490, 1306, 1162, 810, 746, 694 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  9.44 (1H, s, HC=N), 8.12 (1H, d, *J*=8.4 Hz, 6'-ArH), 7.91 (2H, d, *J*=8.0 Hz, 2,6-SO<sub>2</sub>ArH), 7.51 (1H, s, 3'-ArH), 7.38 (2H, d, *J*=8.0 Hz, 3,5-SO<sub>2</sub>ArH), 7.34 (1H, d, *J*=8.4 Hz, 5'-ArH), 2.47 (3H, s, CH<sub>3</sub>); Anal. Calcd. for C<sub>14</sub>H<sub>11</sub>NO<sub>2</sub>SCI<sub>2</sub> (328.22): C, 51.23; H, 3.39; N, 4.27. Found: C, 51.18; H, 3.41; N, 4.25.

**3p:** IR (KBr):  $\nu_{\max}$  3034, 1686, 1620, 1582, 1546, 1320, 1176, 780, 764, 716, 694 cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta_{\text{H}}$  8.83 (1H, d, *J*=9.2 Hz, HC=N), 8.00 (2H, d, *J*=7.2 Hz, 2,6-SO<sub>2</sub>ArH), 7.65 (1H, d, *J*=14.8 Hz, PhCH=), 7.57 (5H, m, 2'~6'-ArH), 7.47 (3H, m, 3,5-SO<sub>2</sub>ArH), 7.02 (1H, dd, *J*=14.8 Hz and *J*=9.2 Hz, =CHCHN); Anal. Calcd. for C<sub>15</sub>H<sub>13</sub>NO<sub>2</sub>S (271.34): C, 66.40; H, 4.83; N, 5.16. Found: C, 66.37; H, 4.92; N, 5.12.

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