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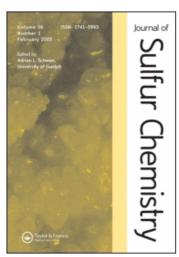
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RESEARCH ARTICLE

Silphox [POCl_{3-n} (SiO₂)_n] as a new, efficient and heterogeneous reagent for the preparation of N-sulfonyl imines under solvent-free conditions

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An efficient, simple and clean solvent-free procedure for the preparation of N-sulfonyl imines is described. The reaction of sulfonamides with different aromatic aldehydes in the presence of silicaphosphinoxide (Silphox) [POCl_{3-n}(SiO₂)_n] as a new and heterogeneous reagent affords the title compounds in good to high yields.

Keywords: Silphox; N-sulfonyl imine; Sulfonamides; Aldehyde; Solvent-free

N-Sulfonyl imines are versatile synthetic intermediates in organic synthesis. As electron deficient imines, they find elegant application in inverse electron demand Diels-Alder chemistry [1–3], stable and reactive alkenes in ene reactions [4, 5], aza-aldehyde equivalents in addition reactions [6] and valuable precursors for the preparation of optically active 2-imidazolines [7]. The preparation of N-sulfonyl imines has attracted much attention in recent years for the versatile usage of the C=N double bond as controllable electrophiles [8].

There are several methods available for the preparation of N-sulfonyl imines including rearrangement of oxime O-sulfinates [9], Lewis acid catalyzed reactions of sulfonamides with aldehydes precursors [10–13] the addition of N-sulfinyl sulfonamides to aldehydes in the presence of boron-trifluoride etherate [14, 15] the utilization of $in\ situ$ generated N, N'-ditosyltellurodiimide from tellurium metal and chloramines T [16], halogen-mediated conversion of N-(trimethylsilyl) imines in the presence of corresponding sulfonyl chloride [17], or two step synthesis using sulfamic acid [18]. The reported methods are associated with one or more of the following drawbacks: (i) long reaction times, (ii) unsatisfactory yields, and (iii) the use of expensive and hazardous reagents. Furthermore, some methods need two-step procedure [34]. Therefore, it seems highly desirable to find a simple and efficient protocol for N-sulfonyl imines synthesis.

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SCHEME 1 Preparation of N-sulfonyl imines from sulfonamides and aryl aldehydes.

The combinations of heterogeneous catalysts with the use of solvent-free conditions represent a suitable way toward the so-called ideal synthesis [19].

According to the applications of heterogeneous phosphorus-containing reagents in organic synthesis [20–27], and also along with our previous studies on solvent-free organic reactions [28–33] herein, we introduce silicaphosphinoxide (Silphox), [POCl_{3-n}(SiO₂)_n] as a new, efficient and heterogeneous reagent for the synthesis of N-sulfonyl imines from sulfonamides and aromatic aldehydes under solvent-free conditions (scheme 1).

1. Discussion section

In order to select the best supporting bed and conditions for the preparation of a suitable reagent, different types of silica gel and alumina were reacted with excess amount of POCl₃ under N₂ atmosphere at various temperatures (table 1). In all reactions, the reagents obtained after washing with dry CH₂Cl₂ and drying showed a considerable weight increasing. As table 1 indicates, the weight increasing was considerably higher when plate silica gel was applied at room temperature (table 1, entry 4). Therefore, the reagent obtained by the reaction of plate silica gel with POCl₃ was chosen (table 1, entry 4) and 1 g of it was added to 10 mL of cool distilled water and the produced HCl was titrated with aqueous NaOH. The results showed that all chlorine atoms of POCl₃ were not replaced with silica gel. Thus, POCl_{3-n}(SiO₂)_n was represented as general structure for Silphox.

To realize the capability of our reagent, the reagent was examined for the synthesis of *N*-sulfonyl imines from sulfonamides and aromatic aldehydes. In order to optimize reaction conditions, the reaction of 4-methylbenzenesulfonamide with benzaldehyde was selected as a model reaction to provide compound **1**. At first, the effect of various amount of Silphox as well as different temperatures were checked. The results are summarized in table 2. As it is shown in table 2, higher yield and shorter reaction time were obtained when 1 g Silphox was used for the reaction of 5 mmol 4-methylbenzenesulfonamide with 5 mmol benzaldehyde at 110°C (table 2, entry 3). The reaction was also carried out in reflux CH₃CN; however, these conditions gave low yield of product in relatively long reaction time. Therefore, the solvent-free conditions are more efficient.

Table 1. Preparation of different reagents via the reaction of different kinds of silica gel and alumina with excess amount of POCl₃.

Entry	Supporting bed	Temperature (°C)	Time (h)	Weight increasing (%)
1	Al ₂ O ₃ (basic)	RT	2	35–38
2	Al ₂ O ₃ (acidic)	RT	2	23–25
3	Column silica gel	RT	2	32–35
4	Plate silica gel	RT	2	52-55
5	Plate silica gel	RT	4	52-55
6	Plate silica gel	50–55°C	1	50-53
7	Plate silica gel	50–55°C	2	50–53

Table 2.	Reaction of 4-methylbenzenesulfonamide (5 mmol) with benzaldehyde (5 mmol)		
in different reaction conditions.			

Entry	Conditions	Time(h)	Yield ^a (%)
1	Silphox (1 g)/r.t.	8	trace
2	Silphox (1 g)/80°C	8	45
3	Silphox (1 g)/110°C	3	85
4	Silphox $(0.5 \text{ g})/110^{\circ}\text{C}$	3	68
5	Silphox (1 g)/CH ₃ CN (20 mL, reflux)	8	35

^aIsolated yield.

Table 3. The comparative synthesis of compound 1 from 4-methylbenzenesulfonamide (5 mmol) and benzaldehyde (5 mmol) using the reported methods versus the present method.

Entry	Reagent and Conditions	Time	Yield ^a (%)	Ref.
1	Silphox (1 g)/110°C	3 h	85	_
2	TiCl ₄ , NEt ₃ /0°C (CH ₂ Cl ₂)	25 min	58	10
3	Si(OEt) ₄ /160°C	6 h	68	12
4	CaCO ₃ , K10 Clay, CH(OMe) ₃ /Microwave	6 min	69	13
5 ^b	a-PhCH=NOH b-TsCN, NEt ₃ /0°C (CCl ₄)	30 min	59	9
6	$PhCHO + Ph_3P = NTs/RuCl_2(PPh_3)_3 (CH_2Cl_2)$	6 h	75	34
7	Silphos (1 g)/CH ₃ CN (reflux)	8 h	29	22
8	Silphos (1 g)/110°C (Solvent-free)	5 h	60	22
9	Plate silica gel (1 g)/r.t.	6 h	0	_
10	Plate silica gel (1 g)/110°C	6 h	trace	_
11	POCl ₃ (0.3 g)/100°C	5 h	51	-

^a Isolated yield. ^bThe oxime has been prepared by the reaction of benzaldehyde with hydroxylamine hydrochloride.

To recognize the capability of the present method in comparison with reported methods for the preparation of *N*-sulfonyl imines from sulfonamides and aldehydes, the synthesis of compound **1** was tested with the reported methods. The results are depicted in table 3. As it is clear from table 3, the present method afforded higher yields. The model reaction was also performed using silphos under solvent-free as well as solution conditions (table 3, entries 7 and 8). This reagent produced the product in lower yield and longer reaction time with respect to our reagent Silphox. Moreover, the reaction of 4-methylbenzenesulfonamide and benzaldehyde was examined in the presence of plate silica gel as well as POCl₃ separately (table 3, entries 9–11). The results showed that these reagents were not efficient for this transformation.

After optimization of the reaction conditions, the reactions of sulfonamides were examined with various structurally diverse aromatic aldehydes (table 4). As it is clear from table 4, the reactions proceeded efficiently and the desired products were obtained in good to high yields.

Aromatic aldehydes containing both electron-withdrawing and electron-donating substituents afforded the corresponding *N*-sulfonyl imines in good yields. However, aryl aldehydes possessing electron-withdrawing or hindered substituents generally necessitates longer reaction times and decreased the reaction yields (table 4, entries 5, 8, 9, 15 and 16). Mention must be made here that the yields of *N*-sulfonyl imines obtained from the reaction of 4-methylbenzenesulfonamide with aldehydes were higher than benzenesulfonamide (for comparison see table 4, entries 1 and 12).

This chemistry was applied unsuccessfully toward the synthesis of N-sulfonyl ketimines as well as enolizable N-sulfonyl aldimines and unfortunately this method shares a common

Table 4. Preparation of *N*-sulfonyl imines from sulfonamides and aldehydes in the presence of silphox under solvent-free conditions at $110\,^{\circ}$ C.

Entry	Product	M. p. (Lit.)	Time (h)	Yield ^a (%)
1	Me————————————————————————————————————	108–109 (109) [27]	3	85
2	Me — S – N — Me (2)	112–114 (111–112) [16]	3	75
3	Me————————————————————————————————————	113–115 [35]	3	84
4	Me————————————————————————————————————	127–129 (128–129) [16]	3	85
5	Me————————————————————————————————————	124–126 [17]	4	76
6	Me — S — N — Br (6)	181–183 (182–185) [13]	3	83
7	Me - S - N - CI (7)	171–173 (172–173) [16]	3	92
8	Me————————————————————————————————————	128–129 (128–129) [12]	4	75
9	Me — S – N – CN (9)	172–173 [35]	4	70
10	Me————————————————————————————————————	100–101 (101) [16]	3	78
11	Me————————————————————————————————————	127–129 [19]	3	80

Table 1. Continued.

Entry	Product	M. p. (Lit.)	Time (h)	Yield ^a (%)
12	O S-N O (12)	77–78 (78–81) [13]	3	76
13	O O O O O O O O O O	130–132 (131–132) [13]	3	72
14	O S-N O (14)	206–208 (208–209) [35]	3	70
15	O CI S-N O (15)	123–124 (125–126) [16]	4	68
16	O S - N NO ₂ NO ₂ (16)	161–163 (162–164) [36]	4.5	65

^aIsolated yield.

limitation with several other methods of N-sulfonyl imine synthesis [8, 10, 12, 16, 17]. Methods of overcoming this limitation are currently under investigation.

The *N*-sulfonyl imines were readily separated from the by-products of the reaction, allowing their isolation in a high state of purity, and in good yield. Crude yields were typically 73–97%. These crude products could then be purified by recrystallization (see experimental section).

In summary, we have introduced a new and efficient reagent for the easy preparation of N-sulfonyl imines from sulfonamides and aryl aldehydes. The application of our reagent in this transformation suffers some advantages such as high yields, simplicity, easy workup, low cost and compliance with green chemistry protocols.

2. Experimental section

2.1 General

All chemicals were prepared from Merck or Fluka chemical companies. The progress of reactions was followed with TLC using silica gel SILG/UV 254 plates. Acidic and basic Al $_2$ O $_3$ 90 (0.063–0.2 mm), column silica gel (type 60, 63–200 µm) and plate silica gel (type 60, 15–40 µm) were used as support. Infrared spectra were run on a Perkin Elmer 781 and a Shimadzu FT-IR 8300 spectrophotometer. The 1 H NMR (250 MHz) and 13 C NMR (62.5 MHz) were run on a Bruker Avanced DPX-250, FT-NMR spectrometer in pure deuterated solvents with tetramethyl silane as an internal standard. Melting points determined in open capillary tubes in a Buchi-535 circulating oil melting point apparatus without further corrections.

- **2.1.1** General procedure for the preparation of silphox. Under a nitrogen atmosphere, to a flask containing dry plate silica gel (6.0 g, 0.1 mol) was added POCl₃ (64 g, 0.5 mol) at room temperature and stirred slowly with a mechanical stirrer for 30 min. The mixture was then heated to 60°C while it was stirring under pressure of nitrogen for 3 h to remove all produced HCl. Afterward, the reaction mixture was cooled to room temperature, filtered and the resulting precipitate was washed with dry CH₂Cl₂ (50 mL) to give Silphox as a white solid (9.1–9.3 g), which was stored in a capped bottle. The reagent can be kept without any change for months. The presence of chloride in the reagent was determined by titration of the produced HCl from the addition of Silphox (1 g) to cool distilled water (10 mL) with 0.1 M aq. NaOH. The results obtained from several runs showed the formation of 5.7–5.9 mmol of HCl for 1 g Silphox. This shows that not all the chlorine atoms of POCl₃ were replaced by silica gel.
- 2.1.2 General procedure for synthesis of N-sulfonyl imines. To a 100-mL single-necked flask equipped with magnetic stirring bar and short-path distilling head was added equivalent amounts (typically 5 mmol) of the appropriate sulfonamide and aldehyde. The resulting mixture was heated and stirred in an oil-bath (110°C) for 10 min, then, Silphox (1 g) was added to it and stirring was continued for 3–4.5 h (table 4). Afterward, the reaction mixture was cooled to room temperature and the solid mixture was poured on a Celite pad and washed with acetone (50 mL). The solvent was evaporated and the crude product was dissolved in warm ethyl acetate (10 mL), treated with n-hexane (35–50 mL), and was allowed to stand at room temperature for 5–6 h. During this time, crystals of product formed which were collected by filtration, washed with n-hexane and dried.

2.2 Some selected spectral data of N-sulfonyl imines

(*E*)-*N*-Benzylidene-4-methylbenzenesulfonamide (*1*) [27] White solid; IR (KBr): 1650, 1570, 1380, 1320, 1160 cm⁻¹. ¹H NMR (CDCl₃): δ 2.33 (s, 3H), 7.25 (d, J = 8.2 Hz, 2H), 7.48 (t, J = 7.8 Hz, 2H), 7.51 (t, J = 6.2 Hz, 1H), 7.80–7.84 (m, 4H), 8.99 (s, 1H). ¹³C NMR (CDCl₃): δ 21.9, 128.4, 129.9, 130.3, 131.6, 135.4, 140.2, 145.1, 170.7.

(*E*)-*N*-(*4*-Methylbenzylidene)-*4*-methylbenzenesulfonamide (2) [16] White powder; ¹H NMR (CDCl₃): δ 2.42 (s, 6H), 7.26 (d, J = 7.5 Hz, 2H), 7.34 (d, J = 7.8 Hz, 2H), 7.82 (d, J = 7.5 Hz, 2H), 7.90 (d, J = 7.5 Hz, 2H), 8.96 (s, 1H). ¹³C NMR (CDCl₃): δ 22.0, 126.8, 128.4, 129.9, 130.2, 130.3, 131.8, 135.7, 144.9, 170.4.

(*E*)-*N*-(*4*-Isoperopyl-benzylidene)-*4*-methylbenzenesulfonamide (*3*) [35] White solid; ${}^{1}\text{H NMR (CDCl}_{3})$: δ 1.20 (d, J = 7 Hz, 6H), 2.33 (s, 3H), 2.93 (m, 1H), 7.18–7.80 (m, 8H), 8.91 (s, 1H). ${}^{13}\text{C NMR (CDCl}_{3})$: 22.4, 22.5, 32.9, 126.1, 126.7, 128.6, 129.3, 130.3, 130.9, 132.9, 156.7, 171.4.

(*E*)-*N*-(4-methoxy-benzylidene)-4-methylbenzenesulfonamide (4) [16] White powder; ${}^{1}\text{H NMR (CDCl}_{3})$: δ 2.36 (s, 3H), 3.78 (s, 3H), 6.93 (d, $J=8.5\,\text{Hz}$, 2H), 7.29 (d, $J=7.8\,\text{Hz}$, 2H), 7.81 (d, $J=7.5\,\text{Hz}$, 2H), 7.85 (d, $J=7.6\,\text{Hz}$, 2H), 8.90 (s, 1H). ${}^{13}\text{C NMR (CDCl}_{3})$: 22.4, 56.6, 115.5, 125.9, 128.7, 130.4, 134.5, 136.6, 145.2, 166.2, 170.1.

(*E*)-*N*-(2,5-Dimethoxy-benzylidene)-4-methylbenzenesulfonamide (5) [17] White solid; $^1\text{H NMR}$ (DMSO-d⁶): δ 2.39 (s, 3H), 3.69 (s,3H), 3.93 (s, 3H), 7.14–7.82 (m, 7H), 9.30 (s, 1H). $^{13}\text{C NMR}$ (DMSO-d⁶): δ 21.0, 55.5, 56.2, 110.2, 114.3, 119.8, 125.2, 127.6, 130.0, 134.9, 144.5, 153.1, 156.4, 165.5.

(*E*)-*N*-(*4*-*Bromobenzylidene*)-*4*-*methylbenzenesulfonamide* (*6*) [13] White powder; 1 H NMR (CDCl₃): δ 2.50 (s, 3H), 7.26–7.85 (m, 8H), 9.04 (s, 1H). 13 C NMR (CDCl₃): 22.7, 125.0, 128.6, 129.5, 131.2, 131.3, 133.9, 167.7.

(*E*)-*N*-(*4*-Chlorobenzylidene)-*4*-methylbenzenesulfonamide (7) [16] White needles; 1 H NMR (CDCl₃): δ 2.46 (s, 3H), 7.15 (d, J = 9.1 Hz, 2H), 7.38 (d, J = 9.0 Hz, 2H), 7.86 (dd, J = 8.9 Hz, 4H), 9.01 (s, 1H). 13 C NMR (CDCl₃): δ 22.0, 128.5, 129.9, 130.2, 131.2, 132.7, 135.3, 141.8, 145.2, 169.0.

(*E*)-*N*-(2-Chloro-benzylidene)-4-methylbenzenesulfonamide (8) [12] White needles; 1 H NMR (CDCl₃): δ 2.43 (s, 3H), 7.29-7.90 (m, 8H), 9.13 (s, 1H). 13 C NMR (CDCl₃): δ 22.6, 126.9, 128.5, 129.1, 129.5, 131.2, 131.9, 134.5, 137.1, 168.1.

(*E*)-*N*-(4-Cyanobenzylidene)-4-methylbenzenesulfonamide (9) [35] White needles; ${}^{1}\text{H NMR (CDCl}_{3})$: δ 2.35 (s, 3H), 7.28 (d, J = 10.2 Hz, 2H), 7.66–7.96 (m, 6H) 8.97 (s, 1H). ${}^{13}\text{C NMR (CDCl}_{3})$: δ 21.7, 117.7, 126.3, 128.3, 129.9, 130.0, 131.3, 132.8, 135.9, 145.3, 168.2.

(*E*)-*N*-(*Furan*-2-ylmethylene)-4-methylbenzenesulfonamide (*10*) [16] Brown powder; 1 H NMR (CDCl₃): δ 2.32 (s, 1H), 6.76–7.75 (m, 7H), 8.83 (s, 1H). 13 C NMR (CDCl₃): δ 22.5, 112.6, 115.4, 126.6, 128.6, 130.9, 148.4, 151.6, 154.4, 157.1.

(*E*)-4-Methyl-N-(thiophen-3-ylmethylene)benzenesulfonamide (*11*) [19] White needles; 1 H NMR (CDCl₃): δ 2.43 (s, 3H), 7.29–8.15 (m, 7H), 9.01 (s, 1H). 13 C NMR (CDCl₃): δ 22.0, 126.8, 128.2, 128.4, 130.2, 135.7, 137.4, 138.6, 144.9, 163.5.

(*E*)-*N*-benzylidenebenzenesulfonamide (12) [13] White solid; ¹H NMR (CDCl₃): 7.61 (m, 6H), 8.02 (m, 4H), 9.05 (s, 1H).

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