Sulfenamide Catalyzed Oxidation of Alcohols to the Corresponding Carbonyl Compounds with Anhydrous Chloramine-T

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***N-tert*-Butylbenzenesulfenamide (1) catalyzed oxidation of various alcohols with stoichiometric amount of anhydrous chloramines-T (2) proceeded smoothly at room temperature to afford the corresponding carbonyl compounds in good yields.**

Key words catalytic oxidation; *N-tert*-butylbenzenesulfenamide; chloramine-T

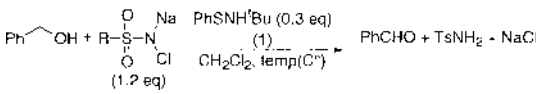
Oxidation of primary and secondary alcohols to the corresponding carbonyl compounds is one of the most important methods and to develop the catalytic oxidation is, therefore, quite a challenging. There are a few catalytic methods reported for the oxidation of alcohols including the reaction promoted by transition metal catalysts such as ruthenium and also by using oxidants such as *N*-(arylseleno)-4-chlorobenzenesulfonamide,[1] 2,2,6,6-tetramethylpiperidin-1-yloxyl (TEMPO)[2] or tetra-*n*-propylammonium perruthenate (TPAP).[3,4] However, the use of toxic oxidants and complicated operations in such oxidations were still disadvantageous.

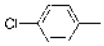
Recently, it was shown from our laboratory that various alcohols are oxidized smoothly by using a stoichiometric amount of *N-tert*-butylphenylsulfinimidoyl chloride (**3**) in the presence of DBU[5] or zinc oxide.[6] In the above oxidation, however, a problem in the separation of *N-tert*-butylbenzenesulfenamide (**1**), a reduced product of **3**, from the formed carbonyl compounds after the oxidation had to be solved. More recently, **1** catalyzed oxidation of alcohols by using a stoichiometric amount of *N*-chlorosuccinimide (NCS) in the presence of potassium carbonate as a base and molecular sieves 4A (MS4A) as a dehydrating agent was reported.[7] The facile *in situ* generation of **3**, a key oxidation, by chloronation of **1** with NCS enabled the above catalytic oxidation.

In this note, we would like to report an alternative method for catalytic oxidation of alcohols by using other chlorinating agent, chloramine-T, which behaves both as an oxidizing agent and a base, in place of NCS. Chloramine-T (**2**)[8] is known as inexpensive, commercially available reagent and it behaves as a source of positive halogen species. Moreover, it has an advantageous point that the catalytic oxidation could be performed by a simple operation without adding any bases and dehydrating agents. *p*-Toluenesulfonamide, formed by the oxidation, was easily separated after the reaction and the recovered amide was easily converted to chloramine-T on treatment with aqueous sodium hypochlorite.

First, catalytic oxidation of benzyl alcohol to benzaldehyde was examined using 0.3 eq of **1** and 1.2 eq of several anhydrous *N*-chloro-*N*-sodio arylsulfonamides as a model reaction (Table 1). Commercially sulfonamide hydrate was dehydrated by heating it up to 100 °C or by keeping it standing over phosphorous pentoxide under vacuum. When *N*-chloro-*N*-sodio-2-methylbenzenesulfonamide was used, the oxidation reaction proceeded slower than that of using **2** (Entry 2). The result suggested that it was the position of substitutent in benzene ring which influenced reaction velocity. It was expected that *N*-chloro-*N*-sodio-4-chlorobenzenesulfonamide[9] having electron-withdrawing substitutent (-Cl) at *para* position of the benzene ring to behave as a stronger oxidant than that of **2**. On the contrary, **2** was found to be the more effective chlorinating agent (Entry 1, 4). After screening the oxidation reaction using various sulfenamide derivatives[10] as catalysts, it was shown that the substitutent of benzene ring of the sulfenamide was not influential to this oxidation (Table 2, Entry 2—5), but the experimental procedures, that is, the way of adding the reagents were more influential to the yields of carbonyl compounds. For example, aldehyde was obtained in 69% when the mixture of benzyl alcohol and **2** in CH_2Cl_2 were added to a solution of **1** in CH_2Cl_2 while yield decreased down to 58% when benzyl alcohol and **1** in CH_2Cl_2 were added **2**. When a mixture of **1** and **2** in CH_2Cl_2 was added to a solution of benzyl alcohol in CH_2Cl_2, on the other hand, benzaldehyde was not obtained. Next, effects of **1** and solvents were examined (Table 3). When **1** was not used in this procedure, the desired product was not obtained at all (Entry 1). Among the solvents examined, CH_2Cl_2 gave the best result, though the oxidation stopped after 1 h and benzaldehyde was obtained in moderate yield. On the other hand, the yield of bezaldehyde was improved up to 84% after being kept stirring in toluene for a long time. The same

Table 1. Effect of Various Substituted Chloramines



Entry	R	Temp (°C)	Time (h)	Yield (%)[a] benzaldehyde
1	 (2)	0	1.0	69
2		0 (rt)	1.0 (75.5)	22 (71)
3		0 (rt)	1.0 (69.5)	Trace (60)
4		0	1.0	3
5		0	1.0	Trace

a) Determined by GC-analysis. rt: room temperature.

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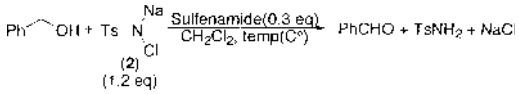
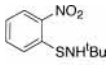
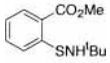
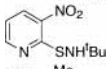
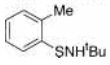
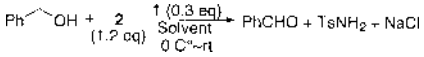
Table 2. Effect of Substituted Sulfenamides



Entry	Sulfenamide	Temp (°C)	Time (h)	Yield (%)[a] benzaldehyde
1	$PhSNH^tBu$ (1)	0–rt (0)	6.0 (1.0)	84[b] (69)
2		0–rt (0)	65.0 (3.0)	29 (5)
3		0	1.0	4
4		0	1.0	Trace
5		0–rt (0)	6.0 (1.0)	61[b] (29)

a) Determined by GC-analysis. *b*) Toluene was used as a solvent.

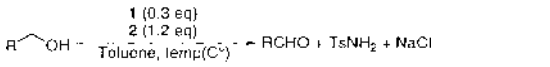
Table 3. Effect of Solvent and Amounts of Chloramine-T

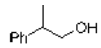
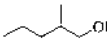
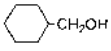


Entry	Solvent	Time (h)	Yield (%)[a] benzaldehyde
1[b]	CH_2Cl_2	1	N.R.
2	CH_2Cl_2	21	69 (69)
3	CH_2Cl_2	18	70 (68)
4	Toluene	6	84 (9)
5	Benzene	2	82 (19)
6	BTF[d]	5	84 (20)
7	MeCN	5	12 (12)
8	Et_2O	23	45 (N.R.)
9	THF	1	8

a) Determined by GC-analysis. *b*) No catalyst. *c*) Reaction condition is 0 °C, 1 h in parenthesis. *d*) Trifluoromethyl benzene. N.R.: No reaction.

Table 4. Catalytic Oxidation of Various Alcohols



Entry	Alcohol	Temp	Time (h)	Yield (%)[a] aldehyde
1	$PhCH_2OH$	0–rt	6.0	84
2	p-$MeOC_6H_4CH_2OH$	rt	6.0	97
3	p-$NO_2C_6H_4CH_2OH$	rt	20.0	80[b]
4	$Ph(CH_2)_3OH$	rt	7.5	84
5	Ph_2CHOH	rt	24.0	71[b,c]
6		0–rt	4.0	82
7		rt	3.5	83
8		rt	14.0	80
9		0–rt	17.0	49
10	$CH_3(CH_2)_7OH$	0–rt	17.0	92
11	$BzO(CH_2)_8OH$	rt	19.0	63[b]
12	$THPO(CH_2)_8OH$	0–rt	23.0	69[b]

a) Determined by GC-analysis. *b*) Isolated yield. *c*) **2** (2.0 eq) was used.

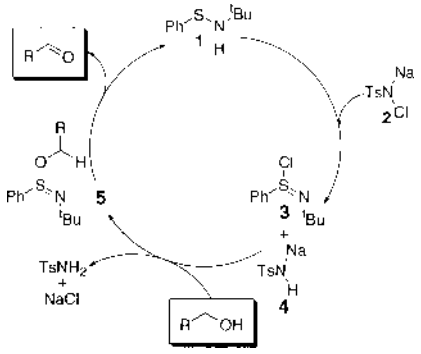


Fig. 1

results were obtained when aromatic solvents were used (Entry 5, 6) while acetonitrile, diethylether, and tetrahydrofuran (THF) were not suitable for this oxidation of alcohols. Further, it was observed that the yield of benzaldehyde was not influenced by the amount of **2**. Finally, the above mentioned oxidation system, **1** and **2**, was applied to the catalytic oxidation of various alcohols to the corresponding carbonyl compounds (Table 4). The desired aldehydes were obtained in good yields whithout any noticeable overoxidation to carboxylic acids under these conditions whereas secondary alcohols were oxidized to the corresponding ketones in moderate yields.

A proposed catalytic cycle is shown in Fig. 1. The catalyst **1** was oxidized with **2** to generate mainly **3** along with **4**. The formed oxidizing agent **3** successively reacted with alcohols in the presence of **4** to afford alkoxysulfilimine (**5**), a key intermediate of the oxidation, which decomposed to form carbonyl compounds along with regeneration of the catalyst **1** and thus a catalytic cycle was effectively established.

As reaction conditions presented here have not yet been sufficiently optimized, the continuous studies are now in progress.

Experimental

General All melting points were determined on a Yanagimoto micro melting point apparatus (Yanaco MP-S3) and are uncorrected. Infrared (IR) spectra were recorded on a Horiba FT300 FT-IR spectrometer. ^{1}H-NMR spectra were recorded on a JEOL EX270 (270 MHz) or a JNM-LA300 (300 MHz) spectrometer; chemical shifts (δ) are reported in parts per million relative to tetramethylsilane. Splitting patterns are designated as a s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. ^{13}C-NMR spectra were recorded on a JEOL EX270 (68 MHz) or a JNM-LA300 (75 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in parts per million relative to tetramethylsilane with the solvent resolution mass spectra (HR-MS) were recorded on a JEOL JMS-700 mass spectrometer. Analytical gasliquid chromatography (GLC) was performed on Shimadzu GC-17A instrument equipped with a flame ionizing detector and a capillary column of OV-101 (0.25 mm×50 m) or CBP10 (0.25 mm×25 m) using helium as carrier gas. Analytical TLC was performed on Merk precoated TLC plates (silica gel 60 GF254, 0.25 mm). Silica-gel column chromatography was carried out on Merk silica gel 60 (0.063—0.200 mm). Preparative thin-layer chromatography (PTLC) was carried out on silica gel Wakogel B-5F. Unless otherwise noted, commercially available reagents were used without purification. Dry solvents were prepared by distillation under appropriate drying agents. The oxidation products were identified by comparing those authentic samples with their gas chromatography (GC) retention times, spectroscopic data such as ^{1}H-, ^{13}C-NMR, and on analytical TLC. All reactions were carried out under an atmosphere of argon in oven-dried glassware with magnetic stirring.

Typical Experimental Procedure for Oxidation of Alcohols The experimental procedure (Table 4, Entry 1) is described: to a stirred suspension of **2** (0.36 mmol) and benzyl alcohol (0.3 mmol) in toluene (1.5 ml) were added successively a solution of **1** (0.09 mmol) in toluene (1 ml) at 0 °C. After the reaction mixture was kept stirring at room temperature for 6 h, it was quenched by adding H_2O and the resulting mixture was extracted with Et_2O. The yield of benzaldehyde was determined by GC-analysis using naphthalene as an internal standard.

8-Benzoyloxyoctanal (Table 4, Entry 11)[11,12] IR (neat) 709, 1103, 1712, 2738, 2823 cm^{-1}; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.38 (6H, m), 1.64 (2H, q, J=7.2 Hz), 1.78 (2H, q, J=6.6 Hz), 2.42 (2H, dt, J=1.8, 7.5 Hz), 4.31 (2H, t, J=6.6 Hz), 7.43 (2H, m), 7.55 (1H, m), 8.04 (2H, m), 9.75 (1H, t, J=1.8 Hz); ^{13}C-NMR (68 MHz, $CDCl_3$) δ: 21.82, 25.72, 28.51, 28.89, 28.89, 43.67, 64.84, 128.21, 129.38, 130.32, 132.71, 166.50, 202.59.

8-Tetrahydropyranyloxyoctanal (Table 4, Entry 12)[13,14] IR (neat) 1735, 1120 cm^{-1}; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.35—2.15 (16H, m), 2.43 (2H, td, J=1.7, 7.3 Hz), 3.37 (1H, td, J=6.6, 9.6 Hz), 3.50 (1H, m), 3.73 (1H, td, J=6.6, 9.6 Hz), 3.86 (1H, m), 4.57 (1H, m), 9.76 (1H, t, J=1.7 Hz); ^{13}C-NMR (68 MHz, $CDCl_3$) δ: 19.66, 21.96, 25.45, 26.00, 29.03, 29.13, 29.61, 30.73, 43.82, 62.32, 67.50, 98.83, 202.82.

***N-tert*-Butylbenzenesulfenamide (Table 2, Entry 1)**[15,16] To a stirred solution of *tert*-butylamine (13.6 g, 185 mmol) in dry ether (150 ml), solution of benzenesulfenyl chloride (12.2 g, 84.2 mmol) in dry ether (30 ml) was added dropwise during 30 min at 0 °C. After the reaction mixture was stirred for 2 h at room temperature, the resulting white suspension was filtered and the filtrate was concentrated. The crude product was distilled to give the desired product as colorless oil (9.53 g, 62%). bp 94—95 °C/933 Pa; IR (neat) 1095, 1203, 1365, 3286 cm^{-1}; ^{1}H-NMR (270 MHz, $CDCl_3$) δ: 1.18 (9H, s), 2.79 (1H, br s), 7.00—7.10 (1H, m), 7.20—7.30 (2H, m), 7.30—7.40 (2H, m); ^{13}C-NMR (68 MHz, $CDCl_3$) δ: 29.14, 54.71, 122.35, 124.40, 128.34, 144.35.

***N-tert*-Butyl-2-nitrobenzenesulfenamide (Table 2, Entry 2)** To a stirred solution of 2-nitrobenzenesulfenylchloride (9.48 g, 50.0 mmol) in dry dichloromethane (100.0 ml), solution of *tert*-butylamine (7.31 g, 100.0 mmol) in dry dichloromethane (40.0 ml) was added dropwise during 40 min at 0 °C. After the reaction mixture was stirred for 12 h at room temperature, the resulting orange suspension was added brine (50.0 ml), and the mixture was extracted with dichloromethane (50 ml×2). Combined organic extracts were washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated to give an orange oil. The crude product was purified by column chromatography (silica gel, hexanes–AcOEt) to give the desired products yellow crystal (9.00 g, 79.5 mmol, 80%). mp 38.0—39.0 °C; IR (KBr) 1200, 1295, 1333, 1504, 3325 cm^{-1}; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.23 (9H, s), 2.66 (1H, br s), 7.21 (1H, m), 7.61 (1H, m), 8.26 (2H, m); ^{13}C-NMR (68 MHz, $CDCl_3$) δ: 29.25, 54.87, 124.18, 125.36, 125.46, 133.32, 142.59, 147.17; high resolution (HR)-FAB-MS (positive-ion mode, *m*-nitrobenzyl alcohol as a matrix) *m/z*: 227.0848 $[M+H]^+$ (Calcd for $C_{10}H_{15}N_2O_2S$: 227.7500).

***N-tert*-Butyl-2-carbomethoxybenzenesulfenamide (Table 2, Entry 3)**[17,18] To a stirred solution of 2-methylatebenzenesulfenyl chloride (1.81 g, 10.8 mmol) in dry dichloromethane (20.0 ml), solution of *tert*-butylamine (1.58 g, 21.6 mmol) in dry dichloromethane (10.0 ml) was added dropwise during 5 min at 0 °C. After the reaction mixture was stirred for 2 h at room temperature, the resulting white suspension was added H_2O, brine, and the mixture was extracted with dichloromethane (50 ml×2). Combined organic extracts were washed with brine, dried over anhydrous Mg_2SO_4, filtered, and concentrated to give a white solid. The crude product was purified by column chromatography (silica gel, hexanes–AcOEt) to give the desired product as white solid (683.9 mg, 2.86 mmol, 26%). mp 83.0 °C; IR (KBr) 1100, 1268, 1310, 1700, 3289 cm^{-1}; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.22 (9H, s), 2.54 (1H, br s), 3.91 (3H, s), 7.10 (1H, ddd, J=0.6, 7.8, 15.6 Hz), 7.49 (1H, ddd, J=1.2, 8.1, 15.6 Hz), 7.97 (1H, dd, J=1.2, 7.8 Hz), 8.16 (1H, d, J=8.1 Hz); ^{13}C-NMR (68 MHz, $CDCl_3$) δ: 29.21, 51.96, 54.52, 123.14, 123.41, 123.47, 130.78, 132.18, 150.84, 166.94; HR-FAB-MS (positive-ion mode, glycerol+*m*-nitrobenzyl alcohol as a matrix) *m/z*: 239.0975 $[M+H]^+$ (Calcd for $C_{12}H_{17}NO_2S$: 239.0980).

***N-tert*-Butyl-3-nitro-2-pyridinesulfenamide (Table 2, Entry 4)** To a stirred solution of 3-nitro-2-pyridinesulfenyl chloride (161.2 mg, 8.46×10^{-1} mmol) in dry dichloromethane (2.0 ml), solution of *tert*-butylamine (123.7 mg, 1.69 mmol) in dry dichloromethane (1.0 ml) was added dropwise during 5 min at 0 °C. After the reaction mixture was stirred for 3 h at room temperature, the resulting orange suspension was added brine, and the mixture was extracted with dichloromethane (10 ml×2). Combined organic extracts were washed with H_2O and brine, dried over anhydrous Mg_2SO_4, filtered, and concentrated to give an orange oil. The crude product was purified by column chromatography (silica gel, hexanes–AcOEt) to give the desired product an orange oil (186.2 mg, 8.19×10^{-1} mmol, 97%). IR (neat) 1201, 1259, 1334, 1585, 2968 cm^{-1}; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.17 (9H, s), 3.98 (1H, br s), 7.20 (1H, dd, J=4.8, 8.1 Hz), 8.48 (1H, dd, J=1.5, 8.1 Hz), 8.73 (1H, dd, J=1.5, 4.8 Hz); ^{13}C-NMR (68 MHz, $CDCl_3$) δ: 29.01, 55.36, 118.93, 133.58, 140.00, 152.99, 164.64; HR-FAB-MS (positive-ion mode, glycerol+*m*-nitrobenzyl alcohol as a matrix) *m/z*: 228.0808 $[M+H]^+$ (Calcd for $C_9H_{14}N_3O_2S$: 228.0802).

***N-tert*-Butyl-2-methylbenzenesulfenamide (Table 2, Entry 5)** To a stirred solution of 2-methylbenzenesulfenyl chloride (4.27 g, 26.9 mmol) in dry dichloromethane (60.0 ml), solution of *tert*-butylamine (4.13 g, 56.5 mmol) in dry dichloromethane (40.0 ml) was added dropwise during 40 min at −78 °C. After the reaction mixture was stirred for 30 min at the same temperature, the mixture was added H_2O, and extracted with dichloromethane (50 ml×2). Combined organic extracts were washed with brine, dried over anhydrous Mg_2SO_4, filtered, and concentrated to give a colorless oil. The crude product was purified by bulb to bulb distillation under reduced pressure to give the desired product as colorless oil (1.21 g, 6.19 mmol, 23%). bp 160.0—170.0 °C/106.7 Pa (bulb to bulb distillation); IR (neat) 1203, 1361, 1462, 2969 cm^{-1}; ^{1}H-NMR (300 MHz, $CDCl_3$) δ: 1.19 (9H, s), 2.23 (3H, s), 2.60 (1H, br s), 6.98 (1H, ddd, J=1.1, 7.2, 14.9 Hz), 7.03 (1H, d, J=7.2 Hz), 7.19 (1H, ddd, J=1.2, 8.1, 14.9 Hz), 7.65 (1H, dd, J=1.1, 8.1 Hz); ^{13}C-NMR (68 MHz, $CDCl_3$) δ: 18.55, 29.17, 54.66, 122.35, 123.86, 125.87, 129.51, 131.47, 142.51; MS [EI$^+$] *m/z* 195 [M^+].

***N*-Chloro-*N*-sodio-4-chlorobenzenesulfonamide**[19,20] To a stirred solution of NaOH (4.0 g, 100.0 mmol) in H_2O (50.0 ml), 4-chlorobenzenesulfonamide (19.5 g, 100.0 mmol) was added at 0 °C. And the reaction mixture was added cold 12% aqueous sodium hypochlorite (105.0 mmol, 65 ml) at the same temperature. After the reaction mixture was stirred for 44 h at room temperature, the resulting white suspension was filtered and the filtered was washed with H_2O. The crude product was purified by recrystallization from H_2O to afford the desired product as white crystal, and dried at 80 °C under reduced pressure. mp 190.0 °C (decomp); IR (KBr) 1091, 1140, 1253 cm^{-1}; ^{1}H-NMR (300 MHz, DMSO-d_6) δ: 7.43 (2H, d, J=8.6 Hz), 7.62 (2H, d, J=8.6 Hz); ^{13}C-NMR (68 MHz, DMSO-d_6) δ: 127.87, 128.80, 133.95, 144.27.

***N*-Chloro-*N*-sodiomethanesulphonamide**[21] To a stirred suspension of *N*,*N*-dichloromethanesulphonamide (4.06 g, 42.7 mmol) and methanesulphonamide (7.00 g, 42.7 mmol) in H_2O (70.0 ml), aqueous NaOH (3.41 g, 85.4 mmol) in H_2O (85 ml) was added dropwise at 0 °C. After the reaction mixture was stirred for 25 h at room temperature, the resulting white suspension was filtered and the filtered was washed with acetone and dried (P_2O_5) at room temperature. The desired product as was obtained as a white powder (5.82 g, 38.4 mmol, 90%). mp 88.0—100.0 °C (decomp); IR (KBr) 1026, 1112, 1245 cm^{-1}; ^{1}H-NMR (300 MHz, DMSO-d_6) δ: 2.70 (3H, s); ^{13}C-NMR (68 MHz, DMSO-d_6) δ: 147.87.

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References and Notes

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