Highly Efficient One-Pot Synthesis of *N*-Acylsulfonamides Using Cyanuric Chloride at Room Temperature

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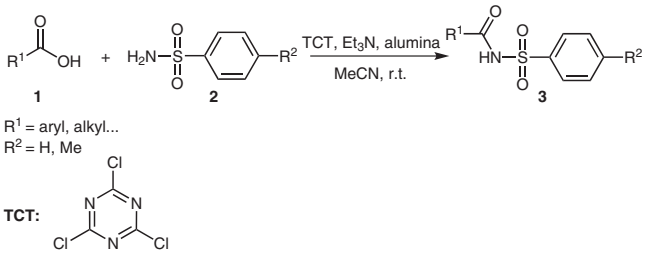
Abstract: A highly efficient and very mild one-pot synthesis of *N*-acylsulfonamides is described using cyanuric chloride. In this synthesis, structurally diverse carboxylic acids and sulfonamides are reacted in the presence of cyanuric chloride, triethylamine, and alumina in anhydrous acetonitrile at room temperature to afford various *N*-acylsulfonamides in excellent yields.

Key words: one-pot, *N*-acylsulfonamide, sulfonamide, carboxylic acid, cyanuric chloride

Cyanuric chloride (2,4,6-trichloro-1,3,5-triazine, TCT) is a stable, nonvolatile, inexpensive, and safe reagent that has long been used in various organic transformations.[1] Cyanuric chloride and its derivatives have been widely employed for the dehydration of amides to nitriles,[2] deoxygenation of sulfoxides,[3] Swern-type oxidation,[4] lactonization of hydroxy-carboxylic acids,[5] Friedel–Crafts acylation,[6] conversion of carboxylic acids into acyl chlorides,[7a] acyl azides,[7b] amides,[7c–e] ketones,[7f] Weinreb amides, hydroxamates,[7g] diazo ketones,[7h] and alcohols,[7i] sulfonic acids to sulfonyl chloride,[8] formamides to isonitriles,[9] ketoximes to amides (Beckmann rearrangement),[10] preparation of β-chlorohydrins from epoxides in water,[11] conversion of alcohols into the alkyl chloride and formate esters[12] and preparation of sulfonamides from sulfonic acid.[13] Furthermore, cyanuric chloride has been used as an in situ source of HCl to catalyze various organic reactions and synthesis.[14]

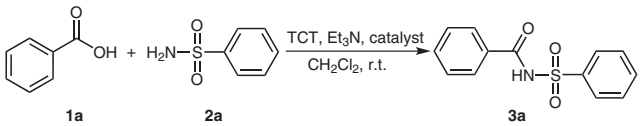
Recently, we reported that cyanuric chloride is a useful reagent for the synthesis of sulfonamides from amine-derived sulfonate salts in this journal.[13a] We now report a highly efficient one-pot synthesis of *N*-acylsulfonamides **3** from sulfonamides **2** and carboxylic acids **1** under very mild reaction conditions using cyanuric chloride in the presence of triethylamine as the base, alumina as a reusable heterogeneous catalyst, and acetonitrile as the solvent (Scheme 1).

In order to establish the optimized reaction conditions, we chose the reaction of benzenesulfonamide (**2a**) and readily available benzoic acid as a model reaction. Thus, benzoic acid (3 equiv) was treated with triethylamine (3 equiv) and cyanuric chloride (1 equiv) in anhydrous dichloromethane and then a solution was added at room temperature containing benzenesulfonamide (**2a**, 3 equiv) and triethylamine (3 equiv) in dichloromethane. The reaction was terminated after 12 hours and *N*-benzoylben-



Scheme 1 Preparation of *N*-acylsulfonamides from carboxylic acids and sulfonamides using cyanuric chloride

Table 1 Effect of Various Metal Oxide Catalysts on the Reaction of Benzoic Acid and Benzenesulfonamide Using Cyanuric Chloride



Entry	Catalyst	Time (h)	Yield[a] (%)
1	–	12	45
2	alumina[b]	1	90
3	alumina[c]	2	80
4	alumina[d]	5	73
5	MgO	2	63
6	montmorillonite K-10	5	70
7	ZnO	3	60
8	silica gel	2	65
9	SnO_2	4	59
10	TiO_2	7	63
11	ZrO_2	5	55
12	CdO	6	60

[a] Isolated yield.
[b] 1 equiv used.
[c] 0.5 equiv used.
[d] 0.25 equiv used.

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zenesulfonamide (**3a**) was isolated in 45% yield (Table 1). Subsequently, we attempted to shorten the reaction time and increase the chemical yield. Thus, we studied the influence of metal oxides as heterogeneous catalysts (Table 1).

As the data in Table 1 indicate, applying a metal oxide as a heterogeneous catalyst dramatically increases the yield as well as shortening the reaction time for the formation of *N*-benzoylbenzenesulfonamide (**3a**). Among these catalysts, one equivalent of neutral alumina (entry 2) gave the best result. Furthermore, using smaller amounts of alumina provided good yields of **3a** but in longer reaction times (entries 3 and 4). Applying other metal oxides also afforded reasonable to good yields of *N*-acylsulfonamide, but the reactions required longer times.

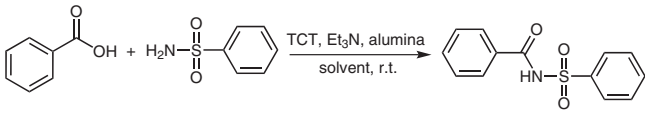
We also investigated the effect of various aprotic solvents on the model reaction (Table 2). Anhydrous acetonitrile and dichloromethane gave the best result (entries 1 and 2); however, acetonitrile was preferred over dichloromethane because it provided better solubility for polar reactants. Acetone and chloroform (entries 3 and 4) also afforded good yields of **3a**, but other solvents were not as efficient for this purpose (entries 5–8).

We then evaluated the effect of various organic and inorganic bases on the reaction model (Table 3).

The results in Table 3 demonstrate that, of the bases examined, triethylamine (entry 1) was the most appropriate base in terms of yield. Potassium and cesium carbonates (entries 2 and 3) as well as DABCO (entry 4) also afforded **3a** in low yield; however, other bases were ineffective (entries 5–8).

Having obtained the optimized reaction condition, it was of interest to determine whether other carboxylic acids and sulfonamides were capable of undergoing acylation to give the corresponding *N*-acylsulfonamides using cyanuric chloride. Hence, we studied the N-acylation reactions of diverse carboxylic acids **1** and sulfonamides **2a–c**.

Table 2 Effect of Various Aprotic Solvents on Reaction of Benzoic Acid and Benzenesulfonamide Using Cyanuric Chloride

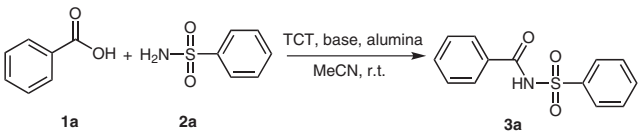


1a 2a 3a

Entry	Solvent[a]	Time (h)	Yield[b] (%)
1	CH_2Cl_2	1	90
2	MeCN	1	92
3	acetone	4	60
4	$CHCl_3$	3	50
5	DMF	12	–[c]
6	DMSO	12	–[c]
7	1,4-dioxane	12	–[c]
8	HMPA	12	–[c]

[a] Anhydrous solvent.
[b] Isolated yield.
[c] No reaction.

Table 3 Effect of Various Organic and Inorganic Bases on Reaction of Benzoic Acid and Benzene Sulfonamide Using Cyanuric Chloride



1a 2a 3a

Entry	Base	Time (h)	Yield[a] (%)
1	Et_3N	1	92
2	K_2CO_3	3	15
3	Cs_2CO_3	5	20
4	DABCO	6	32
5	DBN	12	–[b]
6	DBU	12	–[b]
7	pyridine	12	–[b]
8	DIPEA	12	–[b]

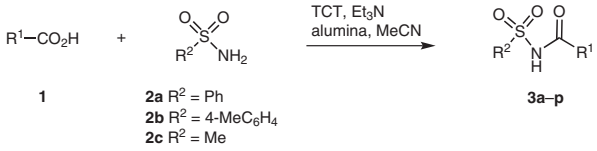
[a] Isolated yield.
[b] No reaction.

As shown by the data in Table 4, by employing different carboxylic acids **1** and sulfonamides **2a–c**, good to excellent yields as well as short reaction times were attained using this method. Both aromatic and aliphatic carboxylic acids and sulfonamides were successfully condensed to afford the corresponding *N*-acylsulfonamides **3a–p**. Electron-releasing or electron-withdrawing substituents on the arylcarboxylic acids had little effect on the efficiency of the reaction; however, longer reaction times were required for arylcarboxylic acids with electron-withdrawing groups (entries 3, 4, and 16). We studied the reaction of *N*-benzoylglycine with both aliphatic (entry 14) and aromatic sulfonamides (entries 6 and 11); interestingly, good yields of the corresponding *N*-acylsulfonamides **3f**,**k**,**n** were attained under mild reaction condition.

Mechanistically, we suggest that, this reaction proceeds via an initial S_nAr-type reaction between the carboxylate anion and cyanuric chloride which affords the active carboxylate-cyanuric ester (Scheme 2). Indeed, in agreement with the literature,[7a–e] the ready formation of the corresponding active ester was observed at early stage of the reaction. Interestingly, the sulfonamides were inert to cyanuric chloride, which reacted exclusively with the carboxylic acids as the carboxylic anions possess higher nucleophilic strength than the sulfonamides. The active carboxylate-cyanuric esters then react with the sulfonamides to provide the corresponding *N*-acylsulfonamides.

In summary, we have developed a mild, one-pot synthetic method for the preparation of *N*-acylsulfonamides from carboxylic acids and sulfonamides using readily available

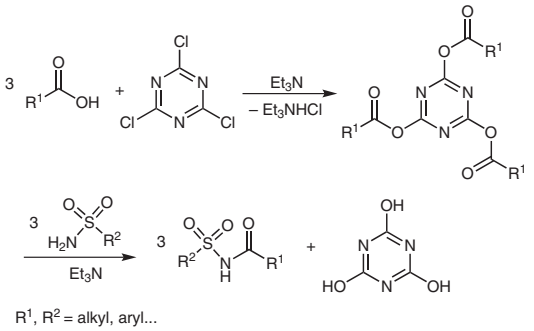
Table 4 One-Pot Synthesis of *N*-Acylsulfonamide from Carboxylic Acid and Sulfonamide Using Cyanuric Chloride at Room Temperature[a]



1 **2a** R^2 = Ph; **2b** R^2 = 4-MeC_6H_4; **2c** R^2 = Me; **3a–p**

Entry	R^1	R^2	Time (h)	Product[b]	Yield[c] (%)	Mp (°C)
1[16]	Ph	Ph	60	**3a**	92	149
2	4-ClC_6H_4	Ph	50	**3b**	85	164
3	4-$O_2NC_6H_4$	Ph	90	**3c**	75	143
4	3-pyridyl	Ph	120	**3d**	70	254
5	4-$MeOC_6H_4$	Ph	50	**3e**	86	145
6	CH_2NHBz	Ph	75	**3f**	60	191
7[17]	(*E*)-CH=CHPh	Ph	120	**3g**	62	157
8[16]	Me	Ph	80	**3h**	74	123
9	4-$MeOC_6H_4CH_2$	Ph	90	**3i**	65	foam
10[13]	Ph	4-MeC_6H_4	40	**3j**	85	149
11	CH_2NHBz	4-MeC_6H_4	70	**3k**	55	175
12[16]	Me	4-MeC_6H_4	75	**3l**	70	140
13[16]	Ph	Me	60	**3m**	65	75
14	CH_2NHBz	Me	70	**3n**	60	122
15[13]	Me	Me	75	**3o**	68	72
16	4-ClC_6H_4	Me	100	**3p**	76	116

[a] Reaction conditions: TCT (1 equiv), Et_3N (6 equiv), alumina (1 equiv), MeCN (50 mL), r.t.
[b] All products were characterized by ^1H and ^{13}C NMR, IR, CHN, and MS analysis.
[c] Yield of isolated product.



R^1, R^2 = alkyl, aryl...

Scheme 2 A plausible mechanism for *N*-acylation of sulfonamide using cyanuric chloride

cyanuric chloride, triethylamine, and alumina in anhydrous acetonitrile. This method provided structurally diverse *N*-acylsulfonamides in good to excellent yields. Furthermore, the cyanuric acid byproduct can be removed from the reaction mixture using an aqueous workup.

All chemicals were purchased from commercial sources. Solvents were purified and dried using reported methods[15] and stored over 3 Å molecular sieves. The reaction progress was monitored by TLC using SILG/UV 254 silica gel plates. Column chromatography was carried out on silica gel 60, (0.063–0.200 mm, 70–230 mesh, ASTM). IR spectra were run on a Shimadzu FTIR-8300 spectrophotometer. ^1H (250 MHz) and ^{13}C NMR (62.5 MHz) were recorded using a Brüker Avance DPX-250, FT-NMR spectrometer. Mass spectra were recorded on a Shimadzu GC MS-QP 1000 EX apparatus. Microanalyses were performed on a Perkin-Elmer 240-B microanalyzer. Melting points were recorded on a Büchi 510 apparatus in open capillary tubes and are uncorrected. Full analytical data are provided for novel products and references are given for known compounds (see Table 4).

N-Acylsulfonamides 3; General Procedure

To a stirred soln of carboxylic acid (3 mmol) and Et_3N (3 mmol) in anhyd MeCN (40 mL) was added TCT (1 mmol) followed by the addition of alumina (1 mmol) and the mixture was stirred at r.t. for 5 min. A soln of sulfonamide (3 mmol) and Et_3N (3 mmol) in anhyd MeCN (10 mL) was added and the soln stirred for a further 40–120 min (TLC indicated completion of the reaction, see Table 4). The reaction was filtered and the filtrate was concentrated under vacuum and the residue was dissolved in $CHCl_3$ (150 mL). The organic layer

was washed with H_2O (2 × 100 mL), dried (anhyd Na_2SO_4), and evaporated in vacuo. The residue was purified by short column chromatography (silica gel, *n*-hexane–EtOAc).

***N*-(4-Chlorobenzoyl)benzenesulfonamide (3b)**
IR (KBr): 3247, 2945, 1753, 1458, 1365, 1146, 765, 539 cm^{-1}.

^1H NMR (250 MHz, DMSO-d_6): δ = 7.35–8.19 (m, 9 H, ArH), 11.85 (s, 1 H, NH, exchangeable with D_2O).

^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 125.51, 127.20, 127.65, 130.21, 132.54, 140.80, 144.05, 148.44, 166.79.

MS (EI): *m*/*z* (%) = 295.0 (32.8) [M^+].

Anal. Calcd for $C_{13}H_{10}ClNO_3S$: C, 52.80; H, 3.41; Cl, 11.99; N, 4.74; S, 10.84. Found: C, 52.83; H, 3.38; Cl, 12.03; N, 4.68; S, 10.80.

***N*-(4-Nitrobenzoyl)benzenesulfonamide (3c)**
IR (KBr): 3350, 3256, 3050, 1694, 1542, 1333, 1159, 1090, 755, 688, 536 cm^{-1}.

^1H NMR (250 MHz, DMSO-d_6): δ = 7.38–8.29 (m, 9 H, ArH), 11.45 (s, 1 H, NH, exchangeable with D_2O).

^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 123.42, 125.25, 128.87, 130.64, 131.34, 135.8, 144.05, 148.44, 165.78.

MS (EI): *m*/*z* (%) = 306.0 (28.6) [M^+].

Anal. Calcd for $C_{13}H_{10}N_2O_5S$: C, 50.98; H, 3.29; N, 9.15; S, 10.47. Found: C, 60.01; H, 3.34; N, 9.10; S, 10.51.

***N*-Nicotinoylbenzenesulfonamide (3d)**
IR (KBr): 3355, 3071, 2449, 1923, 1712, 1596, 1488, 1417, 1322, 1300, 1185, 1040, 811, 747, 693, 641, 537 cm^{-1}.

^1H NMR (250 MHz, DMSO-d_6): δ = 7.50–7.54 (m, 3 H, ArH), 8.22–8.26 (m, 2 H, ArH), 8.74–8.77 (m, 3 H, ArH), 9.04–9.41 (m, 1 H, ArH), 13.18 (s, 1 H, NH, exchangeable with D_2O).

^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 123.43, 123.71, 126.50, 128.83, 135.98, 136.86, 140.01, 150.14, 153.20, 166.21.

MS (EI): *m*/*z* (%) = 262.0 (24.2) [M^+].

Anal. Calcd for $C_{12}H_{10}N_2O_3S$: C, 54.95; H, 3.84; N, 10.68; S, 12.23. Found: C, 54.98; H, 3.79; N, 10.71; S, 12.25.

***N*-(4-Methoxybenzoyl)benzenesulfonamide (3e)**
IR (KBr): 3325, 2885, 1686, 1465, 1446, 1356, 1237, 1158, 1090, 756, 533 cm^{-1}.

^1H NMR (250 MHz, DMSO-d_6): δ = 3.79 (s, 3 H, OCH_3), 6.98–7.97 (m, 9 H, ArH), 12.60 (s, 1 H, NH, exchangeable with D_2O).

^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 55.36, 113.75, 125.51, 127.54, 128.88, 130.58, 131.74, 144.06, 162.78, 166.98.

MS (EI): *m*/*z* (%) = 291.1 (48.7) [M^+].

Anal. Calcd for $C_{14}H_{13}NO_4S$: C, 57.72; H, 4.50; N, 4.81; S, 11.01. Found: C, 57.70; H, 4.55; N, 4.79; S, 11.07.

***N*-[2-Oxo-2-(phenylsulfonamido)ethyl]benzamide (3f)**
IR (KBr): 3458, 3132, 2780, 1728, 1640, 1524, 1458, 1355, 1146, 1088, 861, 711, 662, 554 cm^{-1}.

^1H NMR (250 MHz, DMSO-d_6): δ = 4.02 (s, 2 H, CH_2), 7.25–7.49 (m, 5 H, ArH), 7.83–7.97 (m, 5 H, ArH), 11.37 (s, 1 H, NH, exchangeable with D_2O), 12.64 (s, 1 H, NH, exchangeable with D_2O).

^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 47.37, 129.33, 129.49, 130.22, 130.30, 130.41, 131.25, 133.62, 135.81, 168.57, 170.39.

MS (EI): *m*/*z* (%) = 318.1 (42.8) [M^+].

Anal. Calcd for $C_{15}H_{14}N_2O_4S$: C, 56.59; H, 4.43; N, 8.80; S, 10.07. Found: C, 56.63; H, 4.39; N, 8.82; S, 10.11.

2-(4-Methoxyphenyl)-*N*-(phenylsulfonyl)acetamide (3i)
IR (KBr): 3227, 1690, 1474, 1425, 1348, 1232, 1158, 1090, 776, 531 cm^{-1}.

^1H NMR (250 MHz, DMSO-d_6): δ = 3.26 (s, 2 H, CH_2), 3.48 (s, 3 H, CH_3), 6.58–6.97 (m, 4 H, ArH), 7.33–7.71 (m, 5 H, ArH), 12.08 (s, 1 H, NH, exchangeable with D_2O).

^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 40.23, 55.41, 116.87, 129.21, 129.86, 130.31, 130.3, 132.13, 144.21, 162.26, 170.43.

MS (EI): *m*/*z* (%) = 305.1 (22.4) [M^+].

Anal. Calcd for $C_{15}H_{15}NO_4S$: C, 59.00; H, 4.95; N, 4.59; S, 10.50. Found: C, 59.06; H, 4.98; N, 5.03; S, 10.48.

***N*-[2-(4-Methylphenylsulfonamido)-2-oxoethyl]benzamide (3k)**
IR (KBr): 3458, 3132, 2884, 2780, 1728, 1640, 1524, 1458, 1355, 1146, 1088, 861, 711, 662, 554 cm^{-1}.

^1H NMR (250 MHz, DMSO-d_6): δ = 2.36 (s, 3 H, CH_3), 3.98 (s, 2 H, CH_2), 7.38–7.53 (m, 5 H, ArH), 7.78–7.82 (m, 4 H, ArH), 11.17 (s, 1 H, NH, exchangeable with D_2O), 12.27 (s, 1 H, NH, exchangeable with D_2O).

^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 20.99, 48.28, 127.17, 128.28, 129.51, 131.46, 133.44, 136.34, 144.28, 149.88, 166.47, 168.14.

MS (EI): *m*/*z* (%) = 332.1 (43.4) [M^+].

Anal. Calcd for $C_{16}H_{16}N_2O_4S$: C, 57.82; H, 4.85; N, 8.43; S, 9.65. Found: C, 57.85; H, 4.80; N, 8.46; S, 9.60.

***N*-[2-(Methylsulfonamido)-2-oxoethyl]benzamide (3n)**
IR (KBr): 3340, 3211, 3084, 1718, 1638, 1600, 1555, 1463, 1415, 1400, 1317, 1144, 989, 779, 691, 533 cm^{-1}.

^1H NMR (250 MHz, DMSO-d_6): δ = 2.89 (s, 3 H, CH_3), 3.90 (s, 2 H, CH_2), 7.42–7.49 (m, 3 H, ArH), 7.83–7.86 (m, 2 H, ArH), 11.14 (s, 1 H, NH, exchangeable with D_2O), 12.61 (s, 1 H, NH, exchangeable with D_2O).

^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 41.03, 43.05, 127.16, 128.29, 131.37, 133.72, 166.44, 171.31.

MS (EI): *m*/*z* (%) = 256.1 (41.3) [M^+].

Anal. Calcd for $C_{10}H_{12}N_2O_4S$: C, 46.87; H, 4.72; N, 10.93; S, 12.51. Found: C, 46.81; H, 4.75; N, 10.99; S, 12.57.

***N*-(Methylsulfonyl)-4-nitrobenzamide (3p)**
IR (KBr): 3358, 3248, 1708, 1545, 1330, 1238, 1139, 775, 533 cm^{-1}.

^1H NMR (250 MHz, DMSO-d_6): δ = 2.96 (s, 3 H, CH_3), 7.90–8.51 (m, 4 H, ArH), 10.47 (s, 1 H, NH, exchangeable with D_2O).

^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 40.24, 122.79, 129.43, 144.48, 148.45, 168.46.

MS (EI): *m*/*z* (%) = 244.0 (26.7) [M^+].

Anal. Calcd for $C_8H_8N_2O_5S$: C, 39.34; H, 3.30; N, 11.47; S, 13.13. Found: C, 39.30; H, 3.36; N, 11.51; S, 13.17.

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