Convenient Synthesis of Sulfonamides from Amines and *p*-Toluene Sulfonyl Chloride Mediated by Crosslinked Poly(4-vinylpyridine)

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ABSTRACT: A general, mild, and convenient method has been developed for the synthesis of various *N*-substituted and *N, N*-disubstituted sulfonamides, as a class of sulfa drugs, from the corresponding amines and *p*-toluene sulfonyl chloride in the presence of readily available crosslinked poly-(4-vinylpyridine) as a catalyst, base or polymeric substrates. The use of polymeric catalyst simplifies routine sulfonylation of amines because it eliminates the traditional purification. The polymer can be removed quantitatively and it can be regenerated and reused for several cycles without losing its activity.
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Key words: sulfonamide; amine; *p*-toluene sulfonyl chloride; polymeric catalyst

INTRODUCTION

Sulfonamides are a class of combinatorial chemistry and medical imaging. Sulfonamide derivatives is an important class of pharmaceutical compounds exhibit a wide spectrum of biological activities.[1–3] The sulfa drugs have a veritable history of application for the treatment of bacterial infection. Over 30 drugs containing this functionality are in clinical use, including, antibacterial, diuretics, anticonvulsants, anticancer, anti-inflammatory, antiviral agent, hypoglycemic, and HIV protease inhibitors.[4] Some of these drugs have proved to be useful as herbicides[5] and plaguicides.[6] Arylsulfonyl substituent have been used as protecting groups for oxygen and nitrogen functionalities.[7] Sulfonamide derivatives of azo dyes have been reported to improve light stability and fiber fixation.[8] Sulfonylation is a significant reaction in the synthesis of naturally occurring bioactive molecules and is an important method for the protection of amines.[9,10]

Although many efforts have been made towards the preparation of novel sulfonamides,[11–20] the conventional synthesis involves the reaction of amino compounds with sulfonyl chlorides in the presence of a base or catalyst.[21–25] Alternatively sulfonamides can be obtained by reacting sulfinic acid salts with an electrophilic nitrogen source such as hydroxylamine-*O*-sulfonic acid,[26] or bis(2, 2, 2-trichloroethyl) azodicarboxylate,[27] by reacting sulfonic acid with isocyanides.[28] Also recently, Deng and Mani reported the synthesis of sulfonamides by reaction of amines with sulfonyl chloride in water.[29] However, these procedures involve the use of a base and elevated temperatures, especially for less reactive aniline substrates. For sterically hindered primary amines with electron withdrawing substituent, bis-sulfonylation is a common side reaction, which necessitates a further mono desulfonation step.[30]

Although the applications of polymeric reagent in organic synthesis have been rapidly developed,[31–54] but, there are a few reports on sulfonamide synthesis by polymeric reagents in literature.[15,54] Barrett et al. reported[54] only one example for synthesis of *N*-benzyl phenylsulfonamide by using polyvinylpyridine.

Previously we reported the synthesis of symmetrical carboxylic anhydride from carboxylic acids and *p*-toluene sulfonyl chloride (TsCl) mediated by crosslinked poly(4-vinylpyridine).[42]

In continuing of our studies on application of crosslinked poly(4-vinylpyridine)[37–53] in organic synthesis, herein we wish to report a green, clean, and simple method for synthesis of sulfonamides from amines and TsCl by using crosslinked poly(4-vinylpyridine) as a polymeric catalyst, base or substrate, under heterogeneous conditions.

EXPERIMENTAL

Chemical

Poly(4-vinylpyridine) crosslinked with 2% divinyl benzene (100–200 mesh), [P_4–VP], was purchased

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from Fluka (Milwaukee, WI) and other chemicals were purchased from Fluka (Milwaukee, WI), Aldrich (Buchs, Switzerland), and Merck chemical companies. Progress of the reaction was followed by thin layer chromatography (TLC) using silica gel Poly Gram SIL G/UV 254 plates. All sulfonamide products were known compounds and identified by comparison of their spectra and physical data with those of known samples and all yields refer to the isolated pure products. Melting points were determined with a Buchi melting point B-540 B.V. CHI apparatus. Fourier transform infrared (FTIR) spectra were obtained by using a Bruker, Equinox (model 55; Germany) and NMR spectra were recorded on a Bruker AC 500, Aveance DPX (Germany) spectrophotometer at 500 MHz for ^{1}H and at 125 MHz for ^{13}C NMR in $CDCl_3$ or D_2O solutions.

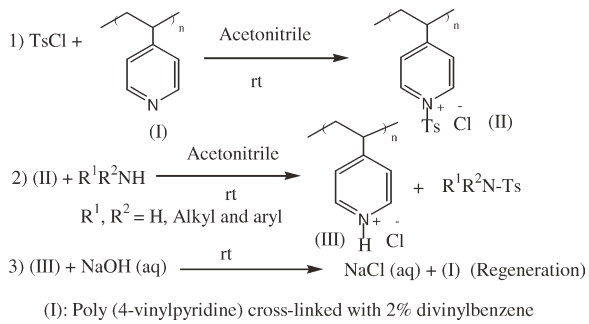
General procedure for the synthesis of sulfonamides from amines and TsCl mediated by [P_4–VP]

To a suspension of [P_4–VP] (1.75 g) in acetonitrile (5 mL), 0.190 g of TsCl, and 1 mmol of a primary or secondary amine were added, and were slowly stirred at room temperature for the appropriate reaction time (Table III). The progress of the reaction was monitored by TLC [eluent: *n*-hexane/ethyl acetate (2/1)]. When the reaction was completed, the mixture was filtered and was washed with acetonitrile (2 × 5 mL). The combined filtrates were evaporated to produce corresponding sulfonamides in good to excellent yields. If further purification was needed flash chromatography on silica gel [eluent: *n*-hexane/ethyl acetate (2/1)] provides highly pure products. The representative examples are given in Table III.

Sulfonylation of *p*-anisidine: A typical procedure

To a suspension of [P_4–VP] (1.75 g) in acetonitrile (5 mL), in round-bottomed flask (50 mL) *p*-methoxy aniline (123 mg; 1 mmol) was added and the mixture was slowly stirred at room temperature for 2.5 h. The progress of the reaction was monitored by TLC [eluent: *n*-hexane-ethyl acetate (2/1)]. When the reaction was completed, the mixture was filtered and was washed with acetonitrile (2 × 5 mL). The combined filtrates were evaporated and flash chromatography on silica gel [eluent: *n*-hexane/ethyl acetate (2/1)] provides highly pure, the cream solid *N*-(4-methoxyphenyl) *p*-toluene sulfonamide in 93% yield (257 mg).

m.p = 115–116°C (literature m.p = 114°C[51] and 104[21]); FTIR (KBr) ν_{max} (cm^{-1}) = 3267 (N—H), 1509,1466, and 1396 (C=C), 1329 (S=O, asymmetric stretching), 1250 and 1252 (C—O), 1218 (C—N), and



Scheme 1 Mechanism of the reaction and regeneration of the polymer.

1156 (S=O, symmetric stretching), 1090 (C—S), 1029, 1010, 811, and 772 (S—O—C), 675.

^{1}H-NMR, ($CDCl_3$, 500 MHz), δ (ppm) = 2.3 (3H, s, CH_3), 3.76 (3H, s, OCH_3), 4.06 (1H, b, N—H), 6.69 (2H, d, J = 8.78 Hz), 6.89 (2H, d, J = 8.86 Hz), 7.1 (2H, d, J = 7.7 Hz), 7.5 (2H, d, J = 8.1 Hz); ^{1}H-NMR, (D_2O, 500 MHz), δ (ppm) = 2.29 (3H, s, CH_3), 3.76 (3H, s, OCH_3), 6.99 (2H, d, J = 7.75 Hz), 7.25 (4H, t), 7.59 (2H, d, J = 7.7 Hz), 7.5 (2H, d, J = 7.17 Hz). In D_2O, the peak at 4.06 ppm (N—H) is disappeared and two doublet peaks at 6.89 and 7.1 are overlapped and a triplet at 7.25 ppm is observed; ^{13}C NMR ($CDCl_3$, 125 MHz), δ (ppm) = 21 (CH_3), 57 (OCH_3), 115, 116.3, 126, 129.6, 136.7, 139.2, 140.7, and 152.5 (aromatic).

Regeneration of [P_4–VP]

The spent cream-colored polymer (1 g), was added to an aqueous solution of sodium hydroxide (10%), and was slowly stirred for 24 h. The mixture was filtered and was washed with distilled water and then was dried under vacuum at 40°C overnight. The regenerated polymer was reused several cycles without losing its activity (Entries 4–7 in Table III). The [P_4–VP] that was recycled for four cycles was used again and the results are given in Table III (Entries 3–7), the regenerated polymer was also used for sulfonylation of other aromatic amines.

RESULTS AND DISCUSSION

During our investigation of multiple phase techniques in organic synthesis, we observed that crosslinked poly(4-vinylpyridine) can catalyze sulfonamide formation from amines and TsCl. For this new system the *in situ* generation of [P_4–VP]TsCl mechanism can be proposed. The plausible mechanism is given in Scheme 1.

Probably crosslinked poly(4-vinylpyridine) supported tosyl chloride, [P_4–VP]TsCl, (II), as an intermediate was prepared by treatment of [P_4–VP], with TsCl in acetonitrile at room temperature (Scheme 1;

TABLE I
Sulfonylation of *p*-Anisidine with TsCl[a] in the Presence of [P_4–VP], in Different Solvents at Room Temperature

Entry	Solvent	Time (h)	Yield (%)[b]
1	*n*-Hexane	3	15
2	Petroleum benzene	3	17
3	Carbon tetrachloride	3	21
4	1,4-Dioxane	3	32
5	Dichloromethane	3	40
6	Ethanol	3	57
7	Diethyl ether	3	67
8	Acetone	3	86
9	Acetonitrile	2.5	93

[a] The amine and TsCl were used equimolar in the presence of 2 g [P_4–VP].
[b] Isolated yield.

Step 1). By nucleophilic displacement of an amine with a good leaving group {pyridinium pendent group of [P_4–VP]TsCl}, corresponding sulfonamide can be obtained (Scheme 1; Step 2). However, pyridine pendent group of crosslinked poly(4-vinylpyridine) is a base so, the hydrochloric acid that is generated in the reaction is trapped by the excess of crosslinked poly (4-vinylpyridine) and is remained bounded to the polymer after work-up; hence regeneration of the polymer is easily accomplished by the treatment with an aqueous solution of sodium hydroxide (10%; Scheme 1; Step 3). This kind of mechanism has been proposed in the reaction of thionyl chloride and poly vinylpyrolidone,[55] carboxylate ions with tosyl chloride[56] and synthesis of symmetrical carboxylic anhydrides from carboxylic acids.[42]

To find out the best solvent for the sulfonylation of amines by [P_4–VP], *p*-anisidine was chosen as a model substrate. The reaction between *p*-anisidine (1 mmol) and TsCl (1 mmol) in the presence of 2.0 g [P_4–VP], in various solvents were studied, at room temperature and the results are summarized in Table I. The reaction in *n*-hexane, petroleum benzene, carbon tetrachloride, 1, 4-dioxane, dichloromethane, ethanol, diethyl ether, and acetone (Table I; Entries 1–8) were found to be less effective but, when the reaction was carried out in acetonitrile an isolated yield of 93% (Table I; Entry 9) was obtained. Inspection of Table I reveals that the model reaction take place faster in the aprotic polar solvents such as acetone and acetonitrile and the results are better (Table I; Entries 8 and 9) than protic polar solvent such as ethanol (Table I; Entry 6) and nonpolar solvents such as *n*-hexane, petroleum benzene, and carbon tetrachloride (Table I; Entries 1–3). This is probably due to, the feat that polar solvents can stabilized the ionic intermediate [Scheme 1, (II)] while, the polar protic solvent such as ethanol compete with the amine as nucleophile.

The reaction of *p*-anisidine (1 mmol), as a model substrate, and TsCl (1 mmol) in the absence and presence of different amounts of polymer, in acetonitrile at room temperature were investigated and the results are given in Table II. As shown in Table II, in the absence of polymer the model reaction, took place very slow and only small amount of sulfonylation was occurred (Table II; Entry 1) while, in the presence of 1.75 g of polymer the best result was observed (Table II; Entry 5). Probably this is due to the preparation of the crosslinked poly(4-vinylpyridine) supported tosyl chloride, [P_4–VP]TsCl, (II), as an intermediate that is a good leaving group {pyridinium pendent group of [P_4–VP]TsCl}.

A number of available amines such as ammonia solution, primary or secondary amines, aniline, and derivatives of aniline were used for the synthesis of the corresponding sulfonamides by using TsCl in the presence of [P_4–VP] in acetonitrile at room temperature and the representative results are given in Table III.

As can be seen in Table III, the reaction between TsCl, and *p*-anisidine, which is activated by an electron-donating group (methoxy group) in the presence of [P_4–VP], gives the best result with excellent isolated yield (93%) after 2.5 h, while *p*-nitroaniline that deactivated by an electron-withdrawing group (nitro group) was completely failed, and even after 5 h, no sulfonamide product was observed (Table III; Entry 10).

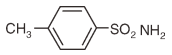
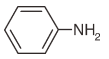
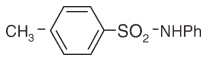
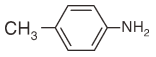
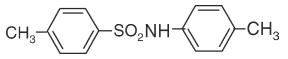
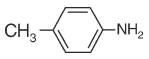
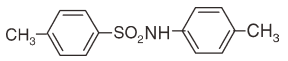
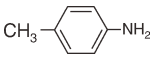
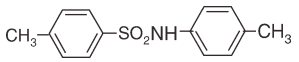
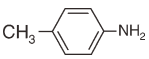
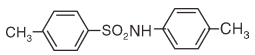
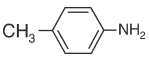
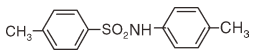
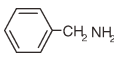
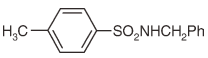
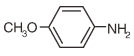
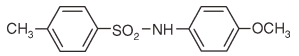
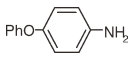
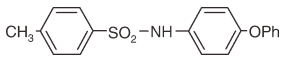
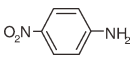
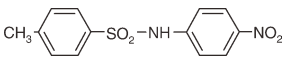
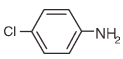
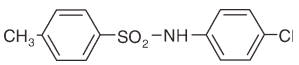
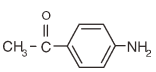
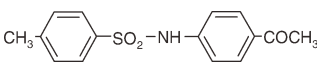
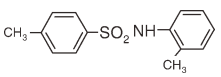
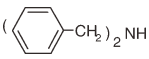
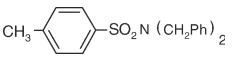
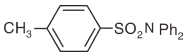
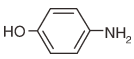
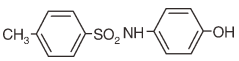
The chemoselectivity of the method is also noteworthy. Meshram and Patil reported the sulfonylation of amines, alcohols, and phenols with cupric oxide.[24] However, our effort for preparation of *p*-toluene sulfonate ester was not successful and even when the reaction was took place in ammonia solution or in ethanol, no *p*-toluenesulfonic acid or ethyl *p*-toluenesulfonate by-products were observed. Also, sulfonylation of molecule having both —NH_2 and —OH groups (Table III; Entry 17) afforded the corresponding sulfonamide in 86% yield, while the —OH group remained intact after 3 h and no corresponding tosylate ester was observed (Scheme 2).

TABLE II
Sulfonylation of *p*-Anisidine with TsCl[a] in the Presence of Different Amounts of [P_4–VP] in Acetonitrile at Room Temperature

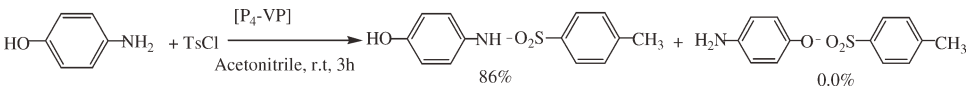
Entry	[P_4–VP] 2% DVB (g)	Time (h)	Yield (%)[b]
1	None	3	Trace
2	1.00	3	21
3	1.25	3	28
4	1.50	3	59
5	1.75	2.5	93
6	2.00	2.5	93

[a] The amine and TsCl were used equimolar.
[b] Isolated yield.

TABLE III
Preparation of Sulfonamides from Amines and Tosyl Chloride Mediated by [P_4–VP][a] in Acetonitrile at Room Temperature

Entry	Substrates	Products[b]	Time (h)	Yield (%)[c]
1	NH_3		2.5	85
2			3	88
3			2.5	91
4[d]			2.5	91
5[d]			2.5	91
6[d]			2.5	90
7[d]			2.5	90
8			2.5	83
9			2.5	93
10			2.5	77
11			5	0.0
12			5	65
13			5	63
14			3	72
15			4	73
16	PH_2NH		6	70
17			3	86

[a] The amine and TsCl were used equimolar in the presence of 1.75 g [P_4–VP].
[b] The structure of products were confirmed by comparison of the melting point, FTIR, ^{1}H-NMR, and ^{13}C-NMR spectral data with those of known compounds.
[c] Isolated yields.
[d] The Entries 4–7, refer to the use of regenerated polymer, that is recycled first, second, third, and fourth times, respectively, under identical conditions.



Scheme 2 Chemoselectivity of the method.

The sulfonamide products were characterized by FTIR, ^{1}H-NMR, and ^{13}C-NMR spectral data, and physical properties were compared to literature values.[24,25,57–59] In this respect, the disappearance of two peaks at 3180–3390 cm^{-1} (NH_2) and appearance of the single peak at 3188–3271 cm^{-1} (NH) and a strong peak at 1306–1338, (S=O; asymmetric stretching) and 1151–1159 cm^{-1} (S=O; symmetric stretching) for the primary amine and the disappearance of a single peak at 3250–3400 cm^{-1} (NH) and the appearance of the strong peaks at 1352–1354, (S=O, asymmetric stretching) and at 1150–1163 cm^{-1} (S=O; symmetric stretching) for secondary amines indicate the formation of the corresponding *N*-substituted and *N, N*-disubstituted sulfonamides.

Characteristic spectral data of some *N*-substituted and *N, N*-disubstituted sulfonamide products are given in Table IV and as an example the FTIR, ^{1}H-NMR, and ^{13}C-NMR spectra of N-(phenyl)-*p*-toluene sulfonamide are shown in Figures 1–3, respectively.

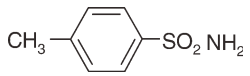
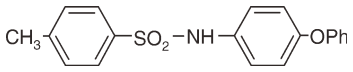
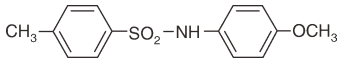
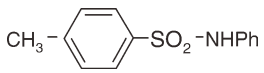
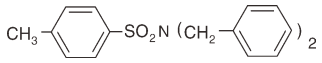
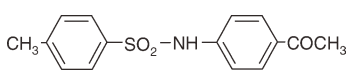
The reusability of the polymer was also investigated. It was observed that, the spent polymer can be easily regenerated by treatment with an aqueous solution of sodium hydroxide (10%; Scheme 1; Step 3). The polymer can be regenerated and reused several cycles without losing its activity (Table III; Entries 4–7). The use of the [P_4–VP] that is four times recycled are given in Table III (Entries 3–7), but also, the regenerated polymer can also be used for sulfonylation of other aromatic amines.

In summary, it was demonstrated that crosslinked poly(4-vinylpyridine) can be used as an efficient catalyst, base or substrate for the suspended solution phase synthesis of sulfonamides from amines and tosyl chloride. Sulfonamides were obtained in good yields. The main advantages of using this polymeric catalyst are simplification of the reaction work up, selectivity, regenerability, and mild reaction conditions.

CONCLUSIONS

We have developed an efficient and general method for the synthesis of various *N*-substituted and *N, N*-disubstituted *p*-toluene sulfonamides mediated by [P_4–VP] as a heterogeneous catalyst, base, or polymer-supported substrate at room temperature. The reusability of polymer and its commercial availability accompanied with the chemoselectivity and

TABLE IV
Characteristic Spectral Data of Some N-aryl Sulfonamide Products

Entry	Product	m.p (°C)	ν_{max} (cm^{-1})	^{1}H- and ^{13}C-NMR δ (ppm)
1		136–137 (lit,[59] 137)	3327, 3240, 3126, 3049, 2926, 1599, 1574, 1497, 1402, 1389, 1307, 1289, 1184, 1172, 1121, 1095, 1017, 818, 797, 633	2.36 (3H, CH_3, s), 7.26 (2H, NH), 7.36, (2H, d), 7.72, (2H, d)
2		134–135	3188, 1590, 1504, 1488, 1328, 1249, 1219, 1151, 1090, 914, 809, 792, 745	2.43 (CH_3, s), 6.61 (N—H), 6.91, 6.98, 7.05, 7.13, 7.28, 7.35, and 7.67 (13H, ArH)
3		114–116 (lit,[59] 114)	3267, 1509, 1466, 1396, 1329, 1250, 1252, 1218, 1156, 1090, 1029, 1010, 811, 772, 675	2.29 (3H, CH_3, s), 3.76 (3H, OCH_3, s), 4.6 (D_2O) 6.99 (2H), 7.25 (4H), and 7.59 (2H)
4		104–106 (lit,[59] 103)	3235, 2950–3120, 1597, 1481, 1415, 1335, 1153, 1090, 908, 817, 753, 694	2.41 (3H, CH_3, s), 6.78 (1H, N-H), 7.13, 7.26, and 7.68 (9H, ArH)
5		231–233	3050, 2987, 2828, 1599, 1497, 1455, 1443, 1227, 1156, 1122, 1031, 1008, 890, 862, 811, 744, 722, 680	2.32 (3H, CH_3, s), 2.06 (4H, CH2, s), 7.29 (2H, d, $J = 7.91$), 7.39–7.43 (10H, Ar—H), 7.61 (2H, d, $J = 8.21$)
6		201–203 (lit,[59] 203)	3216, 2950–3100, 1668, 1597, 1407, 1338, 1276, 1155, 1088, 909, 822, 682, 629	2.42 (3H, CH_3, s), 2.55 (3H, CH_3—CO, s), 7.17 (2H, d), 7.29 (2H, d), 7.45(2H, d), 7.88 (2H, d)

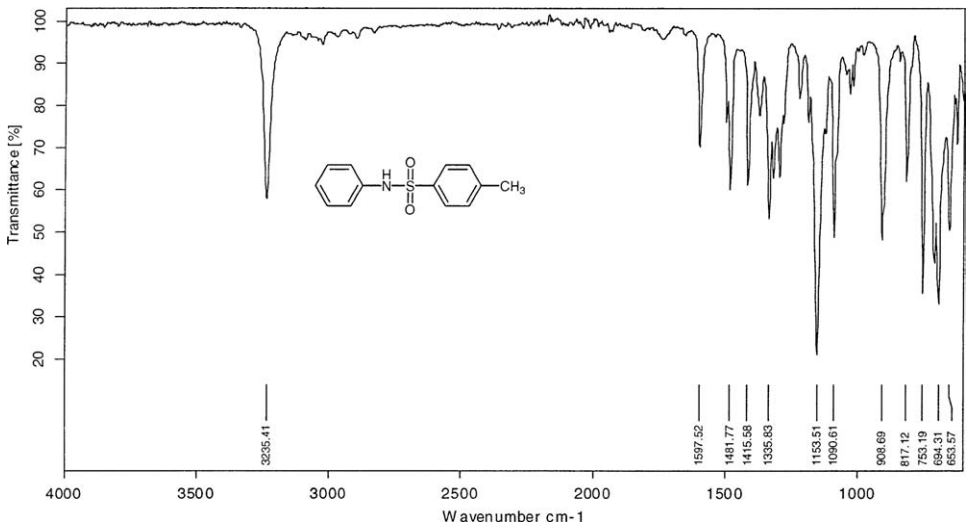


Figure 1 FTIR spectrum of *N*-(phenyl)-*p*-toluene sulfonamide.

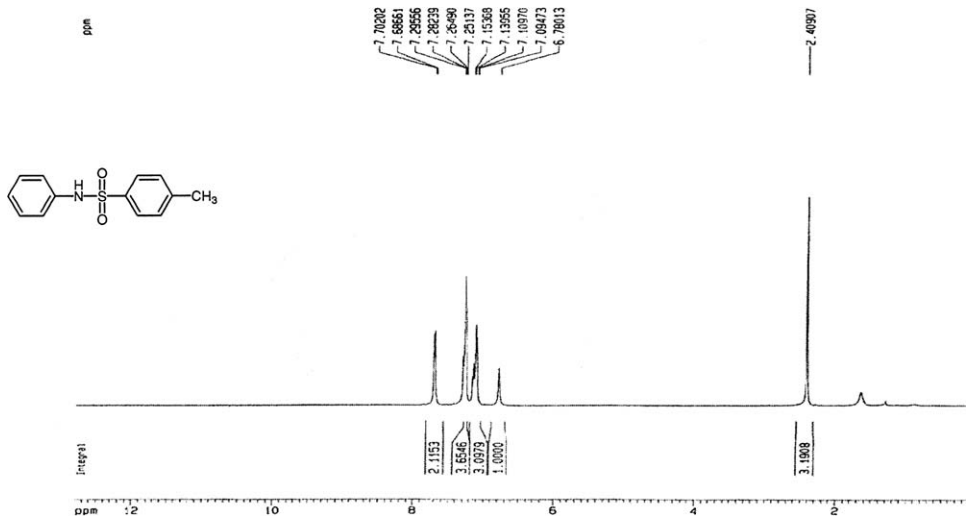


Figure 2 ^{1}H-NMR (500 MHz) spectra of *N*-(phenyl)-*p*-toluene sulfonamide.

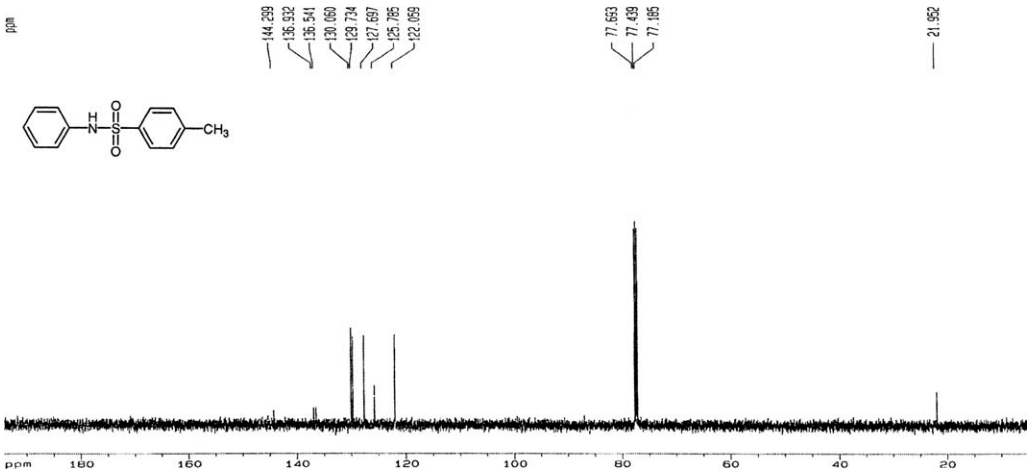


Figure 3 ^{13}C-NMR (125 MHz) spectrum of *N*-(phenyl)-*p*-toluene sulfonamide.

simple work-up procedure are the strong practical points of the presented method. We hope that this approach may be of value to others seeking novel synthetic fragments with unique properties for medical chemistry programs.

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