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Sulfonylamidation of alkylbenzenes at benzylic position with *p*-toluenesulfonamide and 1,3-diiodo-5,5-dimethylhydantoin

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ABSTRACT

Treatment of alkylbenzenes with *p*-toluenesulfonamide and 1,3-diiodo-5,5-dimethylhydantoin (DIH) in a small amount of carbon tetrachloride at 60 °C gave the corresponding α-(*p*-toluenesulfonylamido)alkylbenzenes in good to moderate yields. The present reaction is a simple method for the α-sulfonylamidation of the benzylic position in alkylbenzenes.

The use of trivalent iodines in organic synthesis has been studied widely.[1] In particular, (diacetoxyiodo)benzene (DIB) is the most popular and useful trivalent iodine reagent for organic synthesis as an alternative to toxic heavy-metal reagents.[2] The advantages of DIB are that it is a non-metal oxidant and can be used for not only polar reactions but also radical reactions to generate oxygen-centered radicals, nitrogen-centered radicals, and carbon-centered radicals.[3] For the radical reactions, a DIB–iodine system is used and the initial formation of acetyl hypoiodite (CH_3CO_2I) from the reaction of DIB and iodine is the key step.[4] The synthetic reactions of alkoxyl radicals derived from substrates, such as steroidal alcohols and sugars, with DIB and iodine have been well studied by Suarez et al.[3] We have also examined the utility of nitrogen-centered radicals for the construction of tetrahydroquinolines, benzosultams, and saccharins from sulfonamides with DIB and iodine under irradiation with a tungsten lamp,[5] and oxygen-centered radicals for the construction of chromans from 3-arylpropanols with DIB and iodine under irradiation with a tungsten lamp.[6] The formation of tetrahydroquinolines, benzosultams, and chromans proceeds through the intramolecular cyclization of the formed sulfonamidyl radicals and alkoxyl radicals onto their aromatic rings. Recently, the α-sulfonylamidation of alkylbenzenes with DIB and iodine at 50 °C without solvent was reported to provide α-(*p*-toluenesulfonylamido)alkylbenzenes in moderate to good yields.[7] This reaction proceeds through an intermolecular pathway via the hydrogen atom abstraction from alkylbenzenes at the benzylic position by the formed *p*-toluenesulfonamidyl radical and shows promise for the direct preparation of α-(*p*-toluenesulfonylamido)alkylbenzenes from alkylbenzenes. On the other hand, although 1,3-diiodo-5,5-dimethylhydantoin (DIH) is not a hypervalent iodine compound, it may work as a synthon of unstable acetyl hypoiodite derived from the reaction of DIB and iodine. Here, as part of our synthetic study of DIH,[8] which may work as a synthon in the reaction of DIB and iodine, we would like to report a simple method for the preparation of α-(*p*-toluenesulfonylamido)alkylbenzenes, which involves the reaction of alkylbenzenes with *p*-toluenesulfonamide and DIH alone under warming conditions. DIH is not a hypervalent iodine compound and its structure is similar to that of *N*-iodosuccinimide (NIS). However, as DIH has two N–I bonds, it is expected to be more effective and efficient than NIS. Although synthetic studies of DIH are extremely limited, it is used for the iodination of aromatic compounds.[9]

First, ethylbenzene was treated with *p*-toluenesulfonamide in the presence of DIH alone, similar to the DIB–iodine system, in a small amount of carbon tetrachloride (0.5 mL), as shown in Table 1, and the corresponding α-(*p*-toluenesulfonylamido)ethylbenzene was obtained in moderate to good yields, depending on the amounts of DIH and ethylbenzene used (entries 1–7). α-(*p*-Toluenesulfonylamido)ethylbenzene was obtained in good yield when the reaction was carried out with DIH (1.8 mmol), ethylbenzene (5 mmol), and *p*-toluenesulfonamide (1.0 mmol) at 60 °C (entry 6).[10] Here, an excess amount of ethylbenzene was required to furnish α-(*p*-toluenesulfonylamido)ethylbenzene in good yield, and non-reacted ethylbenzene was recovered in high yield (~90%). When the same reaction was carried out in the presence of galvin-

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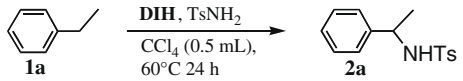
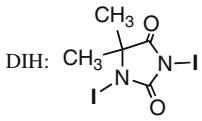
Table 1
Sulfonylamidation of ethylbenzene with $TsNH_2$ and DIH



1a 2a

Entry	$TsNH_2$ (mmol)	**1a** (mmol)	**DIH** (mmol)	**2a** Yield[a] (%)
1	1.1	1	1	25
2	1	3	1	45
3	1	3	0.6	31
4	1	5	1	55
5	1	5	1.5	82
6	1	5	1.8	90
7	1	5	2.0	89
8	1	5	1.8[b]	0
9	1	5	3.6[c]	57
10	1	5	3.6[d]	11
11	1	5	3.6[e]	0
12	1	5	1.8[f]	75
13	1	5	1.8[g]	0
14	1[h]	5	1.8	1
15	1[i]	5	1.8	23

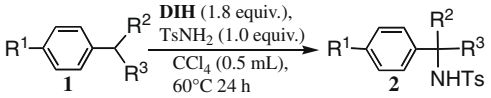
[a] Isolated yield based on $TsNH_2$.
[b] Galvinoxyl-free radical (1.0 equiv) was added.
[c] Instead of DIH, *N*-iodosuccinimide (3.6 mmol) was used.
[d] Instead of DIH, *N*-bromosuccinimide (3.6 mmol) was used.
[e] Instead of DIH, I_2 (3.6 mmol) was used.
[f] Irradiated with a tungsten lamp at 50 °C for 6 h.
[g] Under ultrasonic irradiation at 40 °C for 6 h.
[h] Instead of $TsNH_2$, *p*-$CH_3C_6H_4CONH_2$ was used.
[i] Instead of $TsNH_2$, $CH_3SO_2NH_2$ was used.



oxyl-free radical (1.0 equiv), α-(*p*-toluenesulfonylamido)ethylbenzene was not obtained at all (entry 8) and ethylbenzene was recovered quantitatively. Thus, the results indicate that the present reaction proceeds through the formation of radical species. NIS also worked to provide α-(*p*-toluenesulfonylamido)ethylbenzene in moderate yield (entry 9), although 3.6 equiv of NIS was required. Meanwhile, *N*-bromosuccinimide (NBS) did not work well and the starting material was recovered (entry 10), and molecular iodine did not work at all (entry 11). When the present reaction was carried out under irradiation with a tungsten lamp, α-(*p*-toluenesulfonylamido)ethylbenzene was obtained in good yield (entry 12). In contrast, the product was not obtained at all under ultrasonic irradiation (entry 13). The same treatment with *p*-toluamide and methanesulfonamide, instead of *p*-toluenesulfonamide, did not work well, providing the corresponding α-(*p*-toluamido)ethylbenzene and α-(methanesulfonylamido)ethylbenzene in poor yields, respectively (entries 14 and 15).

Based on these results, propylbenzene, butylbenzene, and 3-acetoxypropylbenzene were treated with *p*-toluenesulfonamide in the presence of DIH to give the corresponding α-(*p*-toluenesulfonylamido)alkylbenzenes in good yields, as shown in Table 2 (entries 2–4). The same treatment of *p*-(bromo)ethylbenzene, *p*-(*t*-butyl)ethylbenzene, and *p*-diethylbenzene with *p*-toluenesulfonamide and DIH provided the corresponding sulfonylamidated compounds at the benzylic position in good yields (entries 5–7), although the reaction of *p*-diethylbenzene was carried out under dark conditions. In these reactions, non-reacted alkylbenzenes were recovered in approximately 80–92% yields. When *p*-(methoxy)ethylbenzene, an electron-rich aromatic compound, was used as a substrate, iodination on the aromatic ring occurred at first by DIH. Therefore, the

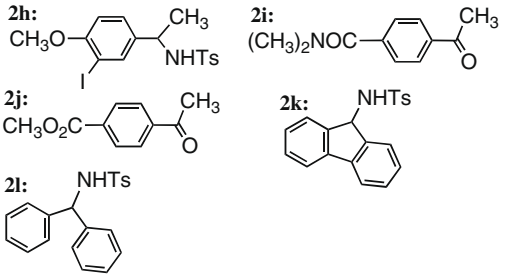
Table 2
Sulfonylamidation of alkylbenzenes with $TsNH_2$ and DIH



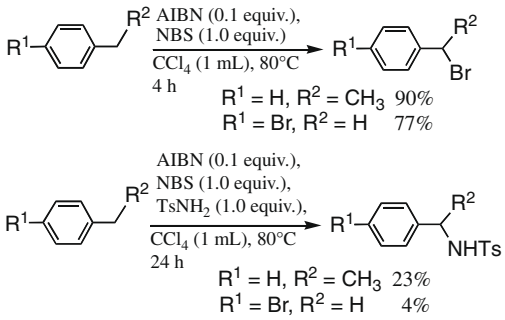
1 2

Entry	R^1–	R^2–	R^3–	**2** Yield[a] (%)
1	H	CH_3	H	90 (**2a**)
2	H	CH_2CH_3	H	58 (**2b**)
3	H	$(CH_2)_2CH_3$	H	60 (**2c**)
4	H	$(CH_2)_2OAC$	H	65 (**2d**)
5	Br	CH_3	H	63 (**2e**)
6	$C(CH_3)_3$	CH_3	H	83 (**2f**)
7[b]	C_2H_5	CH_3	H	79 (**2g**)
8[c]	OCH_3	CH_3	H	84 (**2h**)
9	$CON(CH_3)_2$	CH_3	H	15 (**2i**)
10	CO_2CH_3	CH_3	H	40 (**2j**)
11[d]	Fluorene			53 (**2k**)
12[d]	Diphenylmethane			89 (**2l**)
13[b]	H	CH_3	CH_3	40 (**2m**)
14	H	CH_3	C_2H_5	37 (**2n**)
15[e]	$C(CH_3)_3$	H	H	30 (**2o**)
16[e]	H	H	H	37 (**2p**)
17[e]	Br	H	H	44 (**2q**)

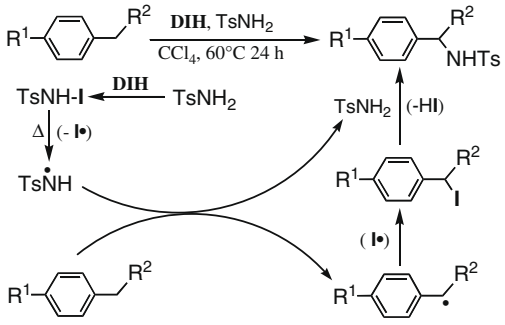
[a] Isolated yield based on $TsNH_2$.
[b] Reaction was carried out under dark conditions.
[c] DIH (6.7 equiv) was used.
[d] K_2CO_3 (1.0 equiv) was added.
[e] Substrate (10 equiv) was used.



treatment of *p*-(methoxy)ethylbenzene with *p*-toluenesulfonamide and an excess amount of DIH generated 3-iodo-4-methoxy-1-α-(*p*-toluenesulfonamido)ethylbenzene in good yield (entry 8). However, the treatment of *N,N*-dimethyl *p*-ethylbenzamide and methyl *p*-ethylbenzoate with *p*-toluenesulfonamide and DIH gave *N,N*-dimethyl *p*-acetylbenzamide and methyl *p*-acetylbenzoate in moderate to low yields, without the formation of *N,N*-dimethyl *p*-α-(toluenesulfonamido)ethylbenzamide and methyl *p*-α-(toluenesulfonamido)ethylbenzoate (entries 9 and 10). Recently, the Wohl–Ziegler reaction of 3,4-dibromo-6-methylbenzyl ethyl ether with NBS in CCl_4 was reported to give unexpected ethyl 3,4-dibromo-6-methylbenzoate instead of 3,4-dibromo-6-(bromomethyl)benzyl ethyl ether.[11] Thus, in the present reactions, ethylbenzene derivatives might be converted into α-(*p*-toluenesulfonylimino)ethylbenzene derivatives through the formation of *N*-iodo-α-(*p*-toluenesulfonylamido)ethylbenzene derivatives and the subsequent elimination of HI, followed by the hydrolysis to acetylbenzene derivatives. The same treatment of fluorine and diphenylmethane provided the corresponding sulfonylamidated products in good to moderate yields (entries 11 and 12). The same treatment of tertiary alkyl benzenes, such as cumene and *sec*-butylbenzene, with *p*-toluenesulfonamide and DIH gave the corresponding α-toluenesulfonylamide derivatives in moderate yields (entries 13 and 14). The same treatment of toluene derivatives with DIH and *p*-toluenesulfonamide provided the corresponding α-toluenesulfonylamide derivatives in moderate yields (entries 15–17). When the Wohl–Ziegler reaction of ethylbenzene and *p*-bromotoluene with NBS and AIBN



Scheme 1.



Scheme 2. Plausible reaction mechanism.

in CCl_4 was carried out, α-bromoethylbenzene and *p*-bromobenzylbromide were obtained in good yields, respectively, as shown in Scheme 1. However, when the same reactions of ethylbenzene and *p*-bromotoluene with NBS and AIBN in CCl_4 in the presence of *p*-toluenesulfonamide were carried out, α-(*p*-toluenesulfonamido)ethylbenzene and *N*-(*p*-bromobenzyl)-*p*-toluenesulfonamide were obtained in low yields even if the reactions were carried out for 24 h at 80 °C. Therefore, the present reactions might be a good method for the preparation of α-(*p*-toluenesulfonamido)alkylbenzenes from alkylbenzenes directly with DIH alone.

The plausible reaction mechanism is shown in Scheme 2. Thus, based on the previous synthetic study of DIH,[8] the initial formation of *N*-iodo-*p*-toluenesulfonamide from the reaction of *p*-toluenesulfonamide and DIH occurs, followed by homolytic cleavage to generate a sulfonamidyl radical. The sulfonamidyl radical abstracts a benzylic hydrogen atom from alkylbenzene to provide a benzyl radical that may react with an iodine atom. Once a benzylic iodide is formed, it smoothly reacts with *p*-toluenesulfonamide to give α-(*p*-toluenesulfonamido)alkylbenzene. Therefore, galvinoxyl-free radical completely retards the present reaction.

In conclusion, treatment of alkylbenzenes with *p*-toluenesulfonamide and 1,3-diiodo-5,5-dimethylhydantoin (DIH) alone at 60 °C gave the corresponding α-(*p*-toluenesulfonylamido)alkylbenzenes in good to moderate yields. The present reaction is a simple tool for the α-sulfonylamidation at the benzylic position of alkylbenzenes. The advantages of DIH are that it is pale yellow solid and does not sublimate, and can be used alone for the present radical reactions, instead of the combination of (diacetoxyiodo)benzene and molecular iodine (it sublimates). Thus, the present method is much more effective and simpler than the previous DIB and molecular iodine system.

Acknowledgments

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10. *Typical procedure for preparation of α-(p-toluenesulfonylamido)ethylbenzene with ethylbenzene, DIH, and p-toluenesulfonamide*: 1,3-Diiodo-5,5-dimethylhydantoin (1.8 mmol, 684 mg) was added to a solution of ethylbenzene (5.0 mmol, 531 mg) and *p*-toluenesulfonamide (1.0 mmol, 171 mg) in carbon tetrachloride (0.5 mL). The mixture was warmed at 60 °C for 24 h under an argon atmosphere. After the reaction, the mixture was poured into saturated aqueous sodium sulfite solution and extracted with $CHCl_3$ (3 × 20 mL). Then, the organic layer was dried over Na_2SO_4. After filtration and removal of the solvent under reduced pressure, the residue was subjected to preparative TLC on silica gel using a mixture of hexane, ethyl acetate, and chloroform (6:3:1) as an eluent to give α-(*p*-toluenesulfonylamido)ethylbenzene in 90% (254 mg) yield.
α-(p-Toluenesulfonylamido)ethylbenzene: Mp 83 °C; IR (Nujol): 1017, 1120, 1157, 1209, 1376, 3251 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.42 (d, J = 6.8 Hz, 3H), 2.39 (s, 3H), 4.46 (quintet, J = 6.8 Hz, 1H), 4.63 (br s, 1H), 7.10 (d, J = 8.5 Hz, 2H), 7.17–7.23 (m, 5H), 7.62 (d, J = 8.5 Hz, 2H); Elemental Anal. Calcd for

$C_{15}H_{17}NO_2S$: C, 65.43; H, 6.22, N, 5.09%. Found: C, 65.20; H, 6.05; N, 5.05.

α-(p-Toluenesulfonylamido)propylbenzene: Mp 102 °C; IR (Nujol): 1003, 1046, 1092, 1162, 1376, 3272 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.80 (t, J = 7.2 Hz, 3H), 1.67–1.86 (m, 2H), 2.35 (s, 3H), 4.19 (q, J = 7.2 Hz, 1H), 4.68 (d, J = 7.2 Hz, 1H), 6.99–7.02 (m, 2H), 7.12 (d, J = 8.4 Hz, 2H), 7.14–7.18 (m, 3H), 7.53 (d, J = 8.4 Hz, 2H); Elemental Anal. Calcd for $C_{16}H_{19}NO_2S$: C, 66.41; H, 6.62; N, 4.84. Found: C, 66.04; H, 6.57; N, 4.74.

α-(p-Toluenesulfonylamido)butylbenzene: Mp 83 °C; IR (Nujol): 1047, 1094, 1161, 1376, 3265 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.83 (t, J = 7.2 Hz, 3H), 1.10–1.32 (m, 2H), 1.62–1.79 (m, 2H), 2.35 (s, 3H), 4.27 (q, J = 7.2 Hz, 1H), 4.66 (d, J = 7.2 Hz, 1H), 6.98–7.02 (m, 2H), 7,10 (d, J = 8.2 Hz, 2H), 7.15–7.18 (m, 3H), 7.51(d, J = 8.2 Hz, 2H); Elemental Anal. Calcd for $C_{17}H_{21}NO_2S$: C, 67.29; H, 6.98; N, 4.62. Found: C, 67.13; H, 6.98; N, 4.58.

γ-Acetoxy-α-(p-toluenesulfonylamido)propylbenzene: Oil; IR (Nujol): 1042, 1159, 1376, 1737, 3268 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 2.00 (s, 3H), 2.00–2.18 (m, 2H), 2.36 (s, 3H), 3.87–4.05 (m, 2H), 4.45 (q, J = 7.2 Hz, 1H), 4.98 (br s. 1H), 7.01–7.04 (m, 2H), 7.16 (d, J = 8.2 Hz, 2H), 7.16–7.20 (m, 3H), 7.54 (d, J = 8.2 Hz, 2H); HRMS (ESI) Calcd for $C_{18}H_{21}NO_4SNa$: m/z 370.1084, Found m/z = 370.1077.

9-(p-Toluenesulfonylamido)fluorene: Mp 199 °C; IR (Nujol): 1064, 1154, 1182, 1376, 3309 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 2.51 (s, 3H), 4.76 (d, J = 9.6 Hz, 1H), 5.40 (d, J = 9.6 Hz, 1H), 7.19–7.23 (m, 4H), 7.32–7.40 (m, 2H), 7.42 (d, J = 8.5 Hz, 2H), 7.63 (d, J = 7.5 Hz, 2H), 7.97 (d, J = 8.5 Hz, 2H); Elemental Anal. Calcd for $C_{20}H_{17}NO_2S$: C, 71.62; H, 5.11; N, 4.18. Found: C, 71.28; H, 4.84; N, 4.11.

N-Diphenylmethyl-p-toluenesulfonylamide: Mp 160 °C; IR (Nujol): 1027, 1058, 1086, 1160, 1376, 3244 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 2.37 (s, 3H), 4.98 (d, J = 7.0 Hz, 1H), 5.56 (d, J = 7.0 Hz, 1H), 7.06–7.11 (m, 4H), 7.15 (d, J = 8.0 Hz, 2H), 7.19–7.25 (m, 6H), 7.56 (d, J = 8.0 Hz, 2H); Elemental Anal. Calcd for $C_{20}H_{19}NO_2S$: C, 71.19; H, 5.68; N, 4.15. Found: C, 70.91; H, 5.53; N, 3.99.

3-Iodo-4-methoxy-1-α-(p-toluenesulfonylamido)ethylbenzene: Mp 115 °C; IR (Nujol): 1016, 1046, 1159, 1202, 1339, 3240 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.38 (d, J = 6.8 Hz, 3H), 2.40 (s, 3H), 3.82 (s, 3H), 4.38 (quintet, J = 6.8 Hz, 1H), 4.76 (d, J = 6.8 Hz, 1H), 6.63 (d, J = 8.3 Hz, 1H), 7.08 (d, J = 8.3 Hz, 1H), 7.17 (d, J = 8.1 Hz, 2H), 7.26 (s, 1H), 7.56 (d, J = 8.1 Hz, 2H); Elemental Analysis Calcd for $C_{16}H_{18}NO_3SI$: C, 44.56; H, 4.21; N, 3.25. Found: C, 44.98; H, 4.20; N, 3.22.

4-Bromo-α-(p-toluenesulfonylamido)ethylbenzene: Mp 141 °C; IR (Nujol): 1022, 1160, 1338, 3240 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.38 (d, J = 6.9 Hz, 3H), 2.40 (s, 3H), 4.38 (quintet, J = 6.9 Hz, 1H), 4.73 (br s, 1H), 6.96 (d, J = 8.5 Hz, 2H), 7.18 (d, J = 8.1 Hz, 2H), 7.30 (d, J = 8.5 Hz, 2H), 7.56 (d, J = 8.1 Hz, 2H); Elemental Anal. Calcd for $C_{15}H_{16}BrNO_2S$: C, 50.86; H, 4.55; N, 3.95. Found: C, 50.54; H, 4.34; N, 3.89.

N-(α,α-Dimethylbenzyl)-p-toluenesulfonamide: Mp 138–141 °C; IR (Nujol): 1076, 1154, 1376, 3364 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.65 (s, 6H), 2.38 (s, 3H), 4.97 (br s, 1H), 7.22–7.14 (m, 5H), 7.30 (d, J = 8.1 Hz, 2H), 7.57 (d, J = 8.1 Hz, 2H). Elemental Anal. Calcd for $C_{16}H_{19}NO_2S$: C, 66.41; H, 6.62; N, 4.84. Found: C, 66.25; H, 6.60; N, 4.80.

N-(α-Ethyl-α-methylbenzyl)-p-toluenesulfonamide: Mp 107–108 °C; IR (Nujol): 1033, 1096, 1150, 1376, 3275 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 0.70 (t, J = 7.5 Hz, 3H), 1.62 (s, 3H), 1.99–1.81 (m, 2H), 2.38 (s, 3H), 4.80 (s, 1H), 7.21–7.12 (m, 5H), 7.26–7.22 (m, 2H), 7.54 (d, J = 8.0 Hz, 2H). Elemental Anal. Calcd for $C_{17}H_{21}NO_2S$: C, 67.29; H, 6.98; N, 4.62. Found: C, 67.32; H, 6.92; N, 4.62.

N-(α-Methyl-4-ethylbenzyl)-p-toluenesulfonamide: Mp 89–91 °C; IR (Nujol) 1019, 1079, 1158, 1324, 1460, 3250 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.18 (t, J = 7.6 Hz, 3H), 1.42 (d, J = 6.7 Hz, 3H), 2.37 (s, 3H), 2.57 (q, J = 7.6 Hz, 2H), 4.43 (quintet, J = 6.7 Hz, 1H), 4.78(d, J = 6.7 Hz, 1H), 7.01 (d, J = 8.2 Hz, 4H), 6.99 (d, J = 8.3 Hz, 2H), 7.02 (d, J = 8.3 Hz, 2H), 7.60 (d, J = 8.3 Hz, 2H); Elemental Anal. Calcd for $C_{17}H_{21}NO_2S$: C, 67.29; H, 6.98; N, 4.62. Found: C, 67.18; H, 6.98; N, 4.55.

N-(4-^{t}Butylbenzyl)-p-toluenesulfonamide: Mp 108–110 °C; IR (Nujol): 1056, 1093, 1150, 1462, 3270 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.17 (s, 9H), 2.34 (s, 3H), 4.00 (d, J = 6.1 Hz, 2H), 4.73 (br s, 1H), 7.03 (d, J = 8.4 Hz, 2H), 7.20 (d, J = 8.4 Hz, 4H), 7.66 (d, J = 8.4 Hz, 2H); Elemental Anal. Calcd for $C_{18}H_{23}NO_2S$: C, 68.10; H, 7.30; N, 4.41. Found: C, 67.76; H, 7.31; N, 4.33.

4-^{t}Butyl-α-(p-toluenesulfonamido)ethylbenzene: Oil; IR (Nujol): 1026, 1078, 1160, 1376, 3255 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 1.26 (s, 9H), 1.43 (d, J = 6.7 Hz, 3H), 2.36 (s, 3H), 4.45 (quintet, J = 6.7 Hz, 1H), 4.85 (d, J = 6.7 Hz, 1H), 7.00 (d, J = 8.2 Hz, 2H), 7.14 (d, J = 8.2 Hz, 2H), 7.19 (d, J = 8.2 Hz, 2H), 7.58 (d, J = 8.2 Hz, 2H); HRMS (ESI) Calcd for $C_{19}H_{25}NO_2SNa$: m/z 354.1498, Found m/z 354.1487.

N-(4-Bromobenzyl)-p-toluenesulfonamide: Mp 105 °C; IR (Nujol): 1064, 1156, 1376, 3263 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 2.44 (s, 3H), 4.09 (d, J = 6.1 Hz, 2H), 4.67 (t, J = 6.1 Hz, 1H), 7.08 (d, J = 8.2 Hz, 2H), 7.32 (d, J = 8.3 Hz, 2H), 7.40 (d, J = 8.2 Hz, 2H), 7.73 (d, J = 8.3 Hz, 2H); Elemental Anal. Calcd for $C_{14}H_{14}BrNO_2S$: C, 49.42; H, 4.15; N, 4.12. Found: C, 49.20; H, 3.92; N, 4.12.

N-Benzyl-p-toluenesulfonamide: Mp 107–109 °C; IR (Nujol): 1027, 1058, 1081, 1162, 1376, 3267 cm^{-1}; ^1H NMR ($CDCl_3$, TMS) δ = 2.33 (s, 3H), 4.13 (d, J = 6.4 Hz, 2H), 4.56 (br s, 1H), 7.19 (d, J = 7.8 Hz, 2H), 7.24–7.30 (m, 3H), 7.31 (d, J = 8.4 Hz, 2H), 7.77 (d, J = 8.4 Hz, 2H); Elemental Anal. Calcd for $C_{14}H_{15}NO_2S$: C, 64.34; H, 5.79; N, 5.36. Found: C, 64.13; H, 5.68; N, 5.30.

11. Gauna, G. A.; Cobice, D.; Awruch, J. *Tetrahedron* **2008**, *64*, 7242.