Cycloaddition of Aroyl/Acylketene *S,N*-Acetals with Tosyl Azide: Synthesis of Novel 4-Aroyl/Acyl-5-amino-1*H*-1,2,3-triazoles and 3,4-Annulated 1,2,3-Triazoles[1]

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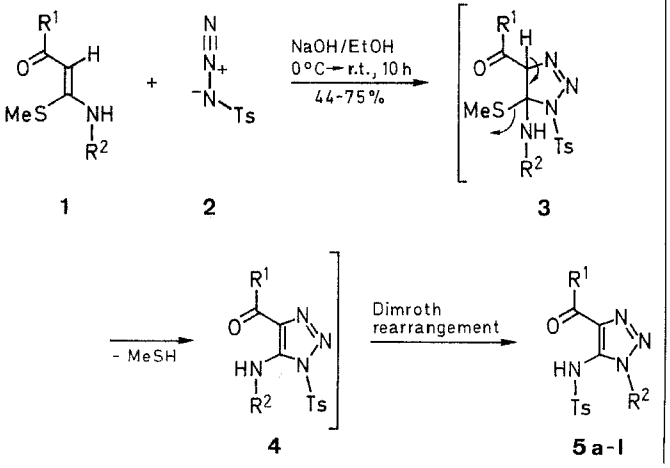
Cycloaddition of aroyl- and acylketene *S,N*-acetals **1a–l** with tosyl azide **2** under alkaline conditions affords novel regiospecifically substituted 4-aroyl/acyl-1-phenyl/alkyl-5-tosylamino-1*H*-1,2,3-triazoles **5a–l**. Some of them **(5a–e, g, h)** are shown to undergo facile detosylation in the presence of concentrated sulfuric acid to give the corresponding 5-aminotriazoles **6a–e, g, h** in excellent yields. The reaction of cyclic *S,N*-acetals **8a–c** with **2** in dioxane at higher temperature yields the corresponding bicyclic 3-aroyl-5,6-dihydrothiazolo[3,2-*c*][1,2,3]-triazoles **10a–c** in good yields.

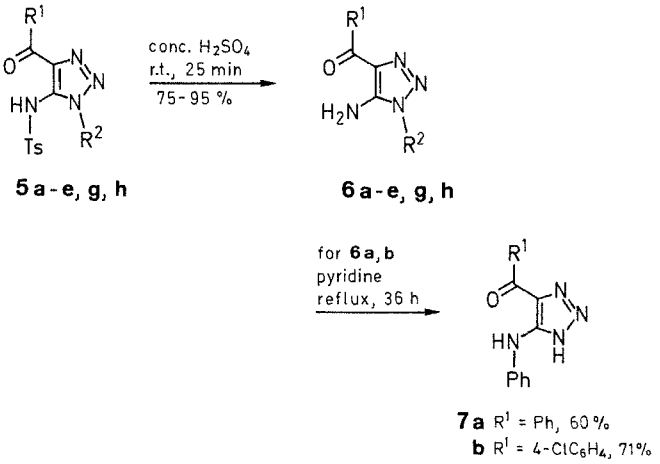
Cycloadditions of tosyl azide with enamines, ynamines and cyanomethylene compounds have been reported to yield different products depending on the structures of the starting materials and the reaction conditions.[2] The reaction of activated malonodinitrile, α-cyanoacetophenone, and β-anilinocrotonate with tosyl azide under basic conditions yields the corresponding rearranged triazoles,[3–6] while under neutral conditions, diazo transfer and other cleavage products are formed.[3] We have recently reported[7] the reaction of sodium azide with polarized ketene *S,N*-acetals derived from primary amines to give substituted tetrazoles through cyclization of initially formed azidoimine intermediates. However, when tosyl azide is reacted with aroyl/acylketene *S,N*-acetals **1**, the course of reaction leads to the corresponding triazoles **5**; we report our results in this paper.

When **1a** and tosyl azide **2** were refluxed in dioxane, the unreacted starting materials were recovered unchanged. However **1a** and **2** reacted smoothly in ethanolic sodium hydroxide to give a colorless product characterized as 4-benzoyl-1-phenyl-5-tosylamino-1*H*-1,2,3-triazole **(5a)** in 57% yield. The structural assignment of **5a** was established as follows. After detosylation[5] of **5a** in the presence of concentrated sulfuric acid, the corresponding 1-phenyl-4-benzoyl-5-amino-1*H*-1,2,3-triazole **(6a)** was formed as observed by its IR and ^{1}H-NMR spectra, which exhibited characteristic peaks at 3390, 3275 cm^{-1} and a broad signal at $\delta = 6.03$ (2H, exchangeable with D_2O), respectively, due to the amino group, thus confirming the exocyclic position of tosylamino functionality. The triazole **6a** further underwent Dimroth rearrangement in the presence of refluxing pyridine to give the corresponding 5-anilino-4-benzoyl-1*H*-1,2,3-triazole **(7a)**, which was distinguished by its ^{1}H-NMR spectrum. Thus the exocyclic aryl NH appeared at $\delta = 9.12$ (1H, exchangeable with D_2O), while the H-1 proton appeared in the region of aromatic protons ($\delta = 7.17–7.70$, exchangeable with D_2O), which apparently proves the rearrangement sequence through initially formed unstable triazole **4a** to **5a** and then **6a** to **7a**. The other *S,N*-acetals **1b–l** also reacted with **2** under similar reaction conditions to give the corresponding triazoles **5b–l** in 44–75% overall yields.

The earlier structural assignment remained consistent with all these triazoles, since the tosylamino NH signal in their ^{1}H-NMR spectra appears between $\delta = 7.80–8.15$. The structural assignment of **5** was further confirmed by detosylation of **5a–e, g, h** as described earlier, when the corresponding 5-aminotriazoles **6b–e, g, h**, respectively, were obtained in 75–95% overall yields. Only the triazole **6b** underwent Dimroth rearrangement under the described conditions to give **7b**, while **6c–e, g, h** remained unchanged under these conditions.[8]



1 2 3 4 5a-l



5a-e, g, h 6a-e, g, h

7a R^1 = Ph, 60%
b R^1 = 4-ClC$_6$H$_4$, 71%

1–6	R^1	R^2	1–6	R^1	R^2
a	Ph	Ph	g	Ph	Et
b	4-ClC$_6$H$_4$	Ph	h	4-ClC$_6$H$_4$	*n*-Pr
c	Ph	PhCH$_2$	i	Ph	*i*-Pr
d	CH$_3$	PhCH$_2$	j	4-CH$_3$C$_6$H$_4$	*n*-Bu
e	Ph	CH$_3$	k	4-ClC$_6$H$_4$	*c*-C$_6$H$_{11}$
f	4-CH$_3$C$_6$H$_4$	CH$_3$	l	Ph	CH$_2$CH(OEt)$_2$

The reaction of **2** with cyclic *S,N*-acetals **8a–c** was next examined. Thus, when **8a** was reacted with **2** in ethanolic sodium hydroxide as described above, the reaction mixture resulted in intractable tar, from which no well defined compound could be isolated. However, in dioxane, at higher temperature, the product isolated was identified as 3-benzoyl-5,6-dihydrothiazolo-[3,2-*c*][1,2,3]triazole **(9a)** (69%). The other substituted thiazolotriazoles **9b–c** were similarly obtained in good yields.

The reaction of acyclic *S,N*-acetals **1a–l** with **2** provides a facile entry to regiospecifically substituted 1-*N*-phenyl/alkyl-4-aroyl/acyl-5-tosylamino (or 5-amino after acidic hydrolysis)

Table 1. 1-Phenyl/benzyl/alkyl-4-aroyl/acyl-5-tosylamino-1*H*-1,2,3-triazole **5a–l** and 3-Aroyl-5,6-dihydrothiazolo[3,2-*c*][1,2,3]triazoles **10a–c** Prepared

Product	Yield[a] (%)	mp[b] (°C)	Molecular Formula[c]	IR (KBr)[d] ν(cm^{-1})	^{1}H-NMR ($CDCl_3$)[e] δ, J(Hz)	MS (70 eV)[f] m/z (%)
5a	57	180–181	$C_{22}H_{18}N_4O_3S$ (418.5)	3185, 1639, 1595, 1398, 1162, 920	2.06 (s, 3H, CH_3); 6.98 (d, J = 8, 2H$_{arom}$); 7.21–7.82 (m, 10H$_{arom}$); 8.22 (dd, J = 8, 1, 2H$_{arom}$); 8.15 (s, 1H, NH, exchangeable with D_2O)	418 (M^+, 1); 353 (11); 325 (14)
5b	65	182–183	$C_{22}H_{17}ClN_4O_3S$ (452.9)	3158, 1625, 1575, 1400, 1160, 917	2.09 (s, 3H, CH_3); 6.93 (d, J = 8, 2H$_{arom}$); 7.21–8.82 (m, 9H$_{arom}$); 8.21 (d, J = 8, 2H$_{arom}$); 8.48 (s, 1H, NH, exchangeable with D_2O)	452 (M^+, 2); 389 (4); 387 (8); 361 (5); 359 (11)
5c	47	205–206	$C_{23}H_{20}N_4O_3S$ (432.5)	3175, 1640, 1595, 1408, 1162, 920	2.01 (s, 3H, CH_3); 5.72 (s, 2H, $C_6H_5C\underline{H}_2$); 6.93 (d, J = 8, 2H$_{arom}$); 7.20–7.55 (m, 10H$_{arom}$); 7.91 (dd, J = 8, 1, 2H$_{arom}$)[g]	
5d	54	143–144	$C_{18}H_{18}N_4O_3S$ (370.4)	3243, 1673, 1560, 1398, 1155, 952	2.26 (s, 3H, CH_3); 2.38 (s, 3H, CH_3); 5.82 (s, 2H, $C_6H_5C\underline{H}_2$); 7.10–7.62 (m, 9H$_{arom}$); 7.80 (s, 1H, NH, exchangeable with D_2O)	370 (M^+, 3); 277 (4)
5e	61	184–185	$C_{17}H_{16}N_4O_3S$ (356.4)	3160, 1643, 1595, 1397, 1172, 917	2.03 (s, 3H, CH_3); 4.25 (s, 3H, NCH_3); 6.96 (d, J = 8, 2H$_{arom}$); 7.25–7.73 (m, 5H$_{arom}$); 8.10 (dd, J = 8, 1, 2H$_{arom}$); 8.15 (s, 1H, NH, exchangeable with D_2O)	356 (M^+, 48); 263 (26)
5f	54	180–181	$C_{18}H_{18}N_4O_3S$ (370.4)	3160, 1638, 1598, 1384, 1165, 915	2.00 (s, 3H, CH_3); 2.38 (s, 3H, CH_3); 4.24 (s, 3H, NCH_3); 6.92 (d, J = 8, 2H$_{arom}$); 7.03–7.45 (dd, A_2B_2, 4H$_{arom}$); 7.98 (d, J = 8, 2H$_{arom}$); 8.12 (s, 1H, NH, exchangeable with D_2O)	370 (M^+, 44); 277 (18)
5g	68	160–161	$C_{18}H_{18}N_4O_3S$ (370.4)	3190, 1640, 1595, 1400, 1181, 920	1.68 (t, 3H, J = 7, $C\underline{H}_3CH_2$); 1.98 (s, 3H, CH_3); 4.65 (q, 2H, J = 7, $NC\underline{H}_2CH_3$); 6.91 (d, J = 8, 2H$_{arom}$); 7.25–7.61 (m, 5H$_{arom}$); 8.03 (dd, J = 8, 1, 2H$_{arom}$); 8.12 (s, 1H, NH, exchangeable with D_2O)	370 (M^+, 32); 277 (18)
5h	75	160	$C_{19}H_{19}ClN_4O_3S$ (418.9)	3195, 1640, 1585, 1405, 1162, 921	1.00 (t, 3H, J = 7, $C\underline{H}_3CH_2CH_2$); 2.05 (s, 3H, CH_3); 2.13 (sext, 2H, J = 7, $CH_3C\underline{H}_2CH_2$); 4.56 (t, 2H, J = 7, $NC\underline{H}_2CH_2CH_3$); 6.97 (d, J = 8, 2H$_{arom}$); 7.40 (d, J = 8, 4H$_{arom}$); 8.01 (s, 1H, NH, exchangeable with D_2O); 8.09 (d, J = 8, 2H$_{arom}$)	No M^+; 327 (5); 325 (100)
5i	45	187–188	$C_{19}H_{20}N_4O_3S$ (384.4)	3173, 1639, 1593, 1400, 1162, 921	1.70 [d, 6H, J = 7, $(C\underline{H}_3)_2CH$)], 1.98 (s, 3H, CH_3); 5.25 [sept, 1H, J = 7, $(CH_3)_2C\underline{H}$]; 6.91 (d, J = 8, 2H$_{arom}$); 7.92 (s, 1H, NH, exchangeable with D_2O); 7.28–7.61 (m, 4H$_{arom}$); 8.04 (dd, J = 8, 1, 2H$_{arom}$); 7.92 (s, 1H, NH, exchangeable with D_2O)	384 (M^+, 16); 291 (4)
5j	70	148–149	$C_{21}H_{24}N_4O_3S$ (412.5)	3193, 1635, 1600, 1400, 1165, 919	0.98 [t, 3H, J = 7, $C\underline{H}_3(CH_2)_3$]; 1.38 (sext, 2H, J = 7, $CH_3C\underline{H}_2$=CH_2CH_2); 1.95 (quint, 2H, J = 7, $CH_3CH_2C\underline{H}_2CH_2$); 2.0 (s, 3H, CH_3); 2.39 (s, 3H, CH_3); 2.39 (s, 3H, CH_3); 4.60 [t, 2H, J = 7, $NC\underline{H}_2(CH_2)_2CH_3$]; 6.92 (d, J = 8, 2H$_{arom}$); 7.10–7.49 (dd, J = 8,4H$_{arom}$); 8.00 (d, J = 8, 2H$_{arom}$); 8.06 (s, 1H, NH, exchangeable with D_2O)	412 (M^+, 4); 357 (5); 319 (2)
5k	44	216–217	$C_{22}H_{23}ClN_4O_3S$ (458.9)	3200, 1640, 1583, 1410, 1166, 925	1.09–2.25 (br m, 10H, CH_2); 2.04 (s, 3H, CH_3); 4.72 (br m, 1H, –CHN); 6.93 (d, J = 8, 2H$_{arom}$); 7.21–7.43 (m, 4H$_{arom}$); 7.86 (br s, 1H, NH, exchangeable with D_2O); 8.10 (d, J = 8, 2H$_{arom}$)	460 (0.4); 458 (M^+, 1); 380 (11); 379 (17); 378 (24); 377 (38)
5l	56	118–119	$C_{22}H_{26}N_4O_5S$ (458.5)	3180, 1636, 1592, 1410, 1167, 921	1.15 (t, J = 7, 6H, $C\underline{H}_3CH_2O$); 2.00 (s, 3H, CH_3); 3.38–3.90 (m, 4H, $CH_3C\underline{H}_2O$); 4.70 (d, 2H, J = 6, NCH_2); 5.17 (t, 1H, J = 6, $NCH_2C\underline{H}$); 6.95 (d, J = 8, 2H$_{arom}$); 7.27–7.63 (m, 5H$_{arom}$); 8.06 (d, J = 8, 2H$_{arom}$); 8.10 (s, 1H, NH, exchangeable with D_2O)	No M^+; 413 ($M^+ - OC_2H_5$, 4)
9a	69	154–155	$C_{11}H_9N_3OS$ (231.3)	1632, 1500, 1482, 1240, 920	4.20 (distorted t, 2H, CH_2); 4.70 (distorted t, 2H, CH_2); 7.25–7.70 (m, 3H$_{arom}$); 8.20–8.53 (m, 2H$_{arom}$)[g]	231 (M^+, 36); 203 (33)
9b	60	175–176	$C_{11}H_8ClN_3OS$ (265.7)	1620, 1582, 1480, 1240, 918	4.20 (distorted t, 2H, CH_2); 4.68 (distorted t, 2H, CH_2); 7.50 (d, J = 9, 2H$_{arom}$); 8.40 (d, J = 9, 2H$_{arom}$)[g]	267 (13); 265 (M^+, 34); 239 (18); 238 (38); 237 (48); 236 (87)
9c	65	154–155	$C_{12}H_{11}N_3O_2S$ (261.3)	1608, 1595, 1488, 1232, 1222, 1172, 919	3.85 (s, 3H, CH_3O); 4.10 (distorted t, 2H, CH_2); 4.60 (distorted t, 2H, CH_2); 6.97 (d, J = 8, 2H$_{arom}$); 8.60 (d, J = 8, 2H$_{arom}$)	261 (M^+, 62); 233 (26); 232 (88)

[a] Yield of pure isolated products based on **1**.
[b] Uncorrected, measured on Thomas Hoover Capillary apparatus.
[c] Satisfactory microanalyses obtained: C ±0.30, H ±0.31, N ±0.28.
[d] Recorded on Perkin Elmer 297 spectrophotometer.
[e] Recorded on Varian EM-390 spectrometer.
[f] Recorded on Jeol JMS-D 300 spectrometer.
[g] In $CDCl_3$/DMSO-d_6.

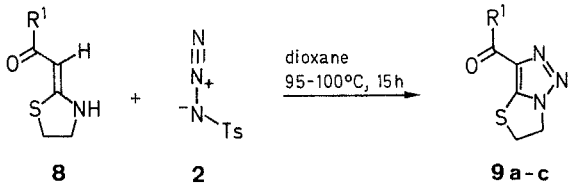
Table 2. 5-Amino-4-aroyl/acyl-1-phenyl/benzyl/alkyl-1*H*-1,2,3-triazoles **6a–e, g, h** and 4-Aroyl-5-anilino-1*H*-1,2,3-triazoles **7a–b** Prepared

Product	Yield[a] (%)	mp[b] (°C)	Molecular Formula[c]	IR (KBr)[d] ν (cm^{-1})	^{1}H-NMR ($CDCl_3$)[e] δ, J (Hz)	MS (70 eV)[f] *m/z* (%)
6a	95	140–141	$C_{15}H_{12}N_4O$ (264.3)	3390, 3275, 1625, 1600, 1510, 1390, 920	6.03 (br s, 2H, NH_2, exchangeable with D_2O); 7.31–7.77 (m, 8H_{arom}); 8.40–8.63 (m, 2H_{arom})	264 (M^+, 22); 236 (16); 235 (21); 208 (23)
6b	87	180–181	$C_{15}H_{11}ClN_4O$ (298.7)	3465, 3335, 1625, 1605, 1510, 928	5.98 (br s, 2H, NH_2, exchangeable with D_2O); 7.41–7.72 (m, 7H_{arom}); 8.45–8.68 (d, J = 9, 2H_{arom})	300 (12); 298 (M^+, 36); 270 (12); 272 (10); 269 (20); 271 (12)
6c	89	150–151	$C_{16}H_{14}N_4O$ (278.3)	3380, 3280, 1638, 1620, 1510, 938	5.40 (s, 2H, $C_6H_5C\underline{H}_2$); 6.31 (br s, 2H, NH_2, exchangeable with D_2O); 7.15–7.57 (m, 8H_{arom}); 8.30–8.54 (m, 2H_{arom})	278 (M^+, 2); 250 (21)
6d	84	185–186	$C_{11}H_{12}N_4O$ (216.2)	3385, 3280, 1658, 1639, 1508, 952	2.52 (s, 3H, CH_3); 5.40 (s, 2H, $C_6H_5C\underline{H}_2$); 6.49 (br s, 2H, NH_2, exchangeable with D_2O); 7.26 (s, 5H_{arom})[g]	–
6e	75	131–132	$C_{10}H_{10}N_4O$ (202.2)	3440, 3280, 1639, 1605, 1521, 916	3.75 (s, 3H, NCH_3); 6.92 (br s, 2H, NH_2, exchangeable with D_2O); 7.31–7.65 (m, 3H_{arom}); 8.30–8.49 (m, 2H_{arom})	–
6g	82	96–97	$C_{11}H_{12}N_4O$ (216.2)	3400, 3310, 1630, 1618, 1505, 921	1.27 (t, J = 7, 3H, $C\underline{H}_3CH_2$); 4.00 (q, 2H, J = 7, $NC\underline{H}_2CH_3$); 6.00 (br s, 2H, NH_2, exchangeable with D_2O); 7.32–7.57 (m, 3H_{arom}); 8.24–8.43 (m, 2H_{arom})	216 (M^+, 32); 187 (15); 160 (28)
6h	80	138–139	$C_{12}H_{13}ClN_4O$ (264.7)	3405, 3300, 1622, 1608, 1500, 920	1.00 (t, 3H, J = 7, $CH_3C\underline{H}_2CH_2$); 1.88 (sext, 2H, J = 7, $CH_3C\underline{H}_2CH_2$); 4.12 (t, 2H, J = 7, $NC\underline{H}_2CH_2CH_3$); 5.70 (br s, 2H, NH_2, exchangeable with D_2O); 7.43 (d, J = 8, 2H_{arom}); 8.50 (d, J = 8, 2H_{arom})	266 (13); 264 (M^+, 50)
7a	60	160–161	$C_{15}H_{12}N_4O$ (264.3)	3130, 1590, 1560, 931	6.81–7.08 (m, 2H_{arom}); 7.17–7.70 (m, 7H_{arom} + NH); 8.28–8.50 (m, 2H_{arom}); 9.12 (s, 1H, NH, exchangeable with D_2O)[g]	264 (M^+, 54); 235 (19); 208 (20)
7b	71	180–181	$C_{15}H_{11}ClN_4O$ (298.7)	3126, 1600, 1580, 928	6.18–7.10 (m, 2H_{arom}); 7.10–7.65 (m, 6H_{arom} + NH); 8.20–8.47 (m, 2H_{arom}); 8.98 (s, 1H, NH, exchangeable with D_2O)[g]	300 (12); 298 (M^+, 29); 272 (5); 270 (16)

[a,b,d–f] As in Table 1.

[c] Satisfactory microanalyses obtained: C ±0.29, H ±0.30, N ±0.30.

[g] In $CDCl_3$/DMSO-d_6.



8 **2** **9a-c**

8, 9	R^1	8, 9	R_1
a	Ph	**c**	4-$CH_3OC_6H_4$
b	4-ClC_6H_4		

triazoles, which are suitably functionalized at 4,5-positions to facilitate further chemical elaboration for the construction of heteroannulated triazoles.[2b,5,9] It is pertinent to note that regiospecific introduction of a methyl group by direct methylation on the ring nitrogen in 5-aminotriazoles[9] is complicated by the formation of other regioisomers,[2c,5] whereas the higher 1-alkylated triazoles are not reported in the literature. Similarly the formation of dihydrothiazolotriazoles[10] **9a–c** from the corresponding cyclic *S,N*-acetals **8a–c** in a one-pot reaction makes the reaction of general application for the synthesis of substituted and annulated triazoles.

The starting acyclic **1a–l**, cyclic **8a–c** *S,N*-acetals and tosyl azide were prepared according to earlier reported procedures.[11–13]

1-Phenyl/alkyl-4-aroyl/acyl-5-tosylamino-1*H*-1,2,3-triazoles 5a–l; General Procedure:

A solution of NaOH (4.80 g, 0.12 mol) in EtOH (10 mL) is added slowly (5 min) to an ice-cooled and stirred suspension of **1** (0.01 mol) and **2** (2.36 g, 0.012 mol) in EtOH (10 mL), and the reaction mixture is further stirred at room temperature for 10 h. It is then poured over crushed ice (150 g), acidified with 20% acetic acid (30 mL), and extracted with $CHCl_3$ (3 × 50 mL). The organic extract is washed with water (3 × 50 mL), dried (Na_2SO_4) and evaporated to give crude triazoles **5a–l**, which are further purified by recrystallization from EtOH (Table 1).

1-Phenyl/alkyl-4-benzoyl-5-amino-1*H*-1,2,3-triazoles 6a–e, g, h; General Procedure:

A solution of appropriate triazole (**5a–e, g, h**, 3 mmol) in conc. H_2SO_4 (10 mL) is stirred at room temperature for 25 min. The reaction mixture is poured over crushed ice (150 g), and the triazoles **6** are separated as colorless solids, which are filtered and recrystallized from EtOH (Table 2).

5-Anilino-4-aroyl-1*H*-1,2,3-triazoles 7a–b; General Procedure:

A solution of triazoles **6a** or **6b** (2 mmol) in pyridine (5 mL) is refluxed for 36 h. The pyridine is removed under reduced pressure, and the residue is poured over crushed ice (100 g) to give triazoles **7a** or **7b** as bright yellow solids, which are filtered and crystallized from EtOH (Table 2).

3-Aroyl-5,6-dihydrothiazolo [3,2-*c*][1,2,3]triazoles 9a–c; General Procedure:

A solution of **8** (0.01 mol) and tosyl azide (**2**; 2.36 g, 0.012 mol) in dioxane (25 mL) is heated with stirring at 90–100 °C for 15 h monitored by TLC, (silica gel-G, 75 μ, Acmes). Dioxane is removed under reduced pressure, the residue is poured over crushed ice (100 g), and the products **9a–c** are separated as colorless solids, which are filtered and recrystallized from EtOH (Table 1).

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