Selective *N*(1)-arylation of benzotriazole with activated aryl halides under conditions of phase transfer catalysis

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Arylation of 1*H*-1,2,3-benzotriazole with activated aryl halides in a medium of aromatic hydrocarbons under conditions of phase transfer catalysis with the use of inorganic bases and cetyltrimethylammonium bromide as a phase-transfer catalyst was studied. Arylation affords both *N*(1)- and *N*(2)-arylation products. Their ratio depends on the nature of the base and the reactivity of the arylating agent. In the presence of catalytic amounts of copper phenylcyclopropanecarboxylate, arylation proceeds regioselectively at the N(1) atom.

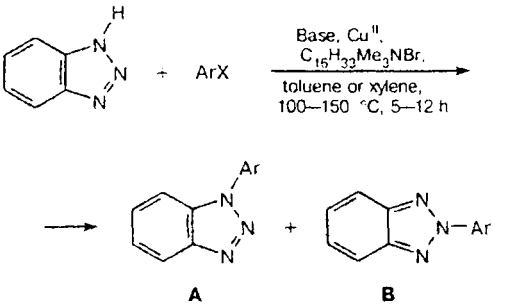
Key words: arylation, benzotriazole, phase transfer catalysis, regioselectivity.

Recently, *N*(1)-substituted derivatives of 1*H*-1,2,3-benzotriazole (BTA) have assumed significance as useful building blocks in organic synthesis, primarily, following the publications by Katritzky and coworkers.[1] However, traditional applications of *N*(1)-aryl derivatives of BTA for performing the Graebe—Ullmann rearrangement[2] with the aim of preparing biologically active compounds of the carbazole, γ-carboline, and acridine series,[3—6] for synthesizing tetraazapentalenes,[7] for producing explosives,[8] *etc.* are still topical problems in modern organic synthesis. The standard procedure for the preparation of the desired *N*(1)-arylbenzotriazoles, which involves arylation of *o*-nitroanilines according to Golberg followed by reduction of the nitro group and diazotization of the resulting amine,[4] is rather laborious and rarely affords the final products in yields higher than 70%. Therefore, only direct *N*(1)-arylation is a promising procedure for the synthesis of this class of compounds. A number of examples of direct arylation of BTA in dipolar solvents were reported.[3,5,6,8,9] However, the dual reactivity of the BTA anion was not taken into account and only in rare instances were *N*(2)-arylation products isolated.[10,11] Fortunately, BTA *N*(2)-arylation products differ substantially in solubility and chromatographic mobility from the *N*(1)-arylation products and can be readily separated from the latter. However, in this case the yields of the target products decrease substantially.

Only one procedure was developed for the quantitative and selective *N*(1)-arylation of BTA in toluene, with the use of an excess of BTA as a base. However, this method is applicable only in the case of sufficiently reactive aryl halides and it takes several days to complete the reaction. In this case, one equivalent of BTA is lost.[12] Therefore, the development of a general, fast, and simple procedure for selective *N*(1)-arylation of BTA is a topical problem.

Results and Discussion

We studied the possibility of selective *N*(1)-arylation of BTA under conditions of phase transfer catalysis in a medium of aromatic solvents with the use of inorganic bases of different strength in the presence of additives of copper salts. The general scheme of the process can be represented as follows:



Cu^{II} is copper 2-phenylcyclopropanecarboxylate.

To optimize *N*(1)-arylation of BTA, we used standard arylating agents, *viz.*, picryl chloride (2,4,6-trinitrochlorobenzene, TNCB) and 2,4-dinitrochlorobenzene (DNCB). The results obtained are listed in Table 1. It can be seen that the ratio between the *N*(1)- and *N*(2)-arylation products increases regularly as the strength of the inorganic base decreases and the reaction time increases (runs *1—8*). However, neither of these two arylating agents allowed us to achieve selective *N*(1)-arylation. KOAc cannot be used as a base because it is involved in the aromatic nucleophilic substitution to give the corresponding polynitrophenyl acetates, which, in turn, react with a hard BTA anion as acylating agents

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Table 1. Arylation of benzotriazole with TNCB and DNCB in the presence of $C_{16}H_{33}Me_3NBr$

Run	ArX	Base	Time /h	Yields of products (%)		Ratio
				A	B	A : B[a]
1	TNCB	K_2CO_3	4	70	28	2.5/1
2	TNCB	$KHCO_3$	6	79	17.5	4.5/1
3	TNCB	K_3PO_4	9	84	12.5	6.7/1
4	TNCB	K_3BO_3	12	85	10.5	8/1
5	DNCB	K_2CO_3	8	72	26	2.7/1
6	DNCB	$KHCO_3$	10	74	21.5	3.5/1
7	DNCB	K_3PO_4	12	76	19	4/1
8	DNCB	K_3BO_3	15	76	17.5	4.3/1
9	TNCB	K_2CO_3[b]	3	97	Traces	—
10	DNCB	K_2CO_3[b]	5	98	Traces	—
11	TNCB	KOAc	6	—[c]	—[c]	—
12	DNCB	KOAc	10	—[c]	—[c]	—

[a] The yields and the ratios of the *N*(1)- and *N*(2)-arylation products were determined spectrophotometrically after chromatographic separation of the reaction products using calibration curves.[13]

[b] In the presence of copper 2-phenylcyclopropanecarboxylate (5 mol.%).

[c] 1-Acetylbenzotriazole and the corresponding trinitro- and dinitrophenols, which were identified by NMR spectroscopy and TLC, were obtained as the major products.

to form 1-acetylbenzotriazole and the corresponding phenols[13] (runs *11* and *12*).

Previously, we have demonstrated that the addition of copper *trans*-2-phenylcyclopropanecarboxylate (PCPC) under conditions of Pd-catalyzed arylation of BTA made it possible to perform selective *N*(1)-arylation of the substrate both in DMF [14] and H_2O.[15] Therefore, we studied the effect of this additive on the ratio between the *N*(1)- and *N*(2)-arylation products using K_2CO_3 as the base. From the data in Table 1 it follows that the addition of Cu PCPC (5 mol.%) resulted in selective *N*(1)-arylation of BTA with both arylating agents (runs *9* and *10*). In addition, the reaction time decreased substantially. The conditions found were extended to a series of arylating agents possessing different reactivities. The data in Table 2 show that *N*(1)-arylation products can be obtained in high yields virtually in all cases. The reactions proceeded most readily in the case of analogs of TNCB (runs *1*—*4*) in toluene. In these cases, the corresponding phenols were always formed in insignificant amounts (apparently, due to hydrolysis) The reactions of perfluoroarenes (runs *9* and *10*) in toluene also proceeded rather rapidly. However, in the case of weakly activated aryl halides, such as 2-bromopyridine, xylene should be used to decrease the reaction time (runs *5*—*8* and *12*).

The structures of the resulting compounds were unambiguously established by NMR spectroscopy and mass spectrometry. In the ^1H NMR spectra of the *N*(1)-isomers, the protons of the benzotriazole fragment are nonequivalent, which is manifested in the presence of three or four multiplets, while the *N*(2)-isomers are characterized by the presence of two multiplets with integral intensities equal to two protons. The structures of the resulting isomers can be also unambiguously established by mass spectrometry because the mass spectra of the *N*(1)-isomers always have peaks of the $[M - N_2]^+$ and $[M + H - N_2]^+$ fragments along with the molecular ions $[M]^+$ and $[M + H]^+$, while this fragmentation is quite untypical of the *N*(2)-isomers. In the latter case, the cleavage of the BTA—Ar bond is predominantly observed.

Table 2. Arylation of benzotriazole with ArX in the presence of K_2CO_3, $C_{16}H_{33}Me_3NBr$, and Cu PCPC

Run	ArX	Solvent	Time /h	Yield* (%)
1	2-Chloro-1,3-dinitro-5-trifluoromethylbenzene	Toluene	4	93
2	2-Chloro-1,5-dinitro-3-trifluoromethylbenzene	Toluene	6	89
3	4-Chloro-3,5-dinitrobenzonitrile	Toluene	9	95
4	Ethyl-4-chloro-3,5-dinitrobenzoate	Toluene	12	95
5	1-Chloro-2-nitro-4-trifluoromethylbenzene	Xylene	8	89
6	1-Chloro-4-nitro-2-trifluoromethylbenzene	Xylene	10	90
7	2-Bromopyridine	Xylene	12	92
8	Chloropentafluorobenzene	Xylene	15	94
9	Octafluorotoluene	Toluene	3—4	96
10	Pentafluoropyridine	Toluene	5—6	98
11	2-Chloro-3-nitropyridine	Toluene	5—6	90
12	1-Fluoro-4-nitrobenzene	Xylene	12	85

* The yields of *N*(1)-arylation products determined by HPLC. In some cases, the corresponding phenols were obtained in yields of up to several percent.

To summarize, the proposed procedure is a simple and convenient selective method for the synthesis of *N*(1)-arylbenzotriazoles.

Experimental

The UV spectra were recorded in EtOH on a Hewlett—Packard 8452A spectrophotometer equipped with a diode matrix. The NMR spectra were measured on a Varian VXR-400 spectrometer in $CDCl_3$ (Me_4Si and C_6F_6 were used as the internal standards in the case of ^1H and ^{19}F NMR, respectively; for the latter, δ −162.8). HPLC was performed on a Gilson chromatograph (the GME-714 program) equipped with a pump (model 303) and a UV--VIZ Holochrome detector (4 : 1 methanol—water system; C18 column; the flow rate was 1.1 mL min^{-1}). The mass spectra were recorded on a Finnigan MAT-113 instrument (direct sample inlet; EI, 70 eV; for *m/z*, only characteristic signals are given and integrated intensities are omitted). The melting points were determined on an Electrothermal block (model IA9100).

Copper PCPC was prepared by the reaction of sodium PCPC with $CuSO_4$ in water.

Preparation of BTA *N*(1)-arylation products (general procedure). A mixture of BTA (1.1 mmol), K_2CO_3 (1 mmol), $C_{16}H_{33}Me_3NBr$ (5 mol.%), copper 2-phenylcyclopropanecarboxylate (2 mol.%), and ArX (1 mmol) was refluxed in toluene (xylene) (20 mL) under N_2 for 4—12 h. The course of the reaction was monitored by TLC. The reaction mixture was passed through a layer of silica gel and the product was eluted with toluene or chloroform depending on the initial arylating agent (TLC control). The eluate was concentrated *in vacuo* and the residue was crystallized from the suitable solvent (CCl_4 for fluorine-containing compounds and $CHCl_3$ or a $CHCl_3$—CCl_4 mixture for TNCB analogs).

1-(2,4-Dinitrophenyl)-1*H*-1,2,3-benzotriazole: m.p. 232 °C. ^1H NMR, δ: 7.48, 7.55, and 7.66 (all m, 1 H, BTA); 8.05 (d, 1 H, Ar, J = 9.1 Hz); 8.22 (m, 1 H, BTA); 8.73 (dd, 1 H, Ar, J_o = 8.9 Hz, J_m = 2.6 Hz); 9.02 (d, 1 H, Ar, J = 2.7 Hz). UV: λ_{max} = 322 nm, ε = $8.60 \cdot 10^3$. MS: 285 $[M]^+$, 286 $[M + H]^+$, 257 $[M - N_2]^+$, 258 $[M + H - N_2]^+$, 239 $[M - NO_2]^+$, 240 $[M + H - NO_2]^+$, 211 $[M - N_2 - NO_2]^+$, 212 $[M + H - N_2 - NO_2]^+$.

2-(2,4-Dinitrophenyl)-1*H*-1,2,3-benzotriazole: m.p. 192 °C. ^1H NMR, δ: 7.45 and 7.89 (both m, 2 H, BTA); 8.51 (d, 1 H, Ar, J_o = 10 Hz); 8.64 (dd, 1 H, Ar, J_o = 9.0 Hz, J_m = 2.4 Hz); 8.74 (d, 1 H, Ar, J_m = 2.4 Hz). UV: λ_{max} = 318 nm, ε = $1.95 \cdot 10^4$. MS: 285 $[M]^+$, 286 $[M + H]^+$, 239 $[M - NO_2]^+$, 240 $[M + H - NO_2]^+$, 167 $[M - BTA]^+$, 168 $[M + H - BTA]^+$.

1-(2,4,6-Trinitrophenyl)-1*H*-1,2,3-benzotriazole: m.p. 222 °C. ^1H NMR, δ: 7.32, 7.57, 7.67, and 8.25 (all m, 1 H, BTA); 9.2 (s, 2 H, Ar). UV: λ_{max} = 354 nm, ε = $9.92 \cdot 10^3$. MS: 330 $[M]^+$, 331 $[M + H]^+$, 302 $[M - N_2]^+$, 303 $[M + H - N_2]^+$, 284 $[M - NO_2]^+$, 285 $[M + H - NO_2]^+$, 256 $[M - N_2 - NO_2]^+$, 257 $[M + H - N_2 - NO_2]^+$.

2-(2,4,6-Trinitrophenyl)-1*H*-1,2,3-benzotriazole: m.p. 183 °C. ^1H NMR, δ: 7.45 and 7.91 (both q, 2 H, BTA); 9.15 (s, 2 H, Ar). UV: λ_{max} = 358 nm, ε = $1.53 \cdot 10^4$. MS: 330 $[M]^+$, 331 $[M + H]^+$, 284 $[M - NO_2]^+$, 285 $[M + H - NO_2]^+$, 212 $[M - BTA]^+$, 213 $[M + H - BTA]^+$.

1-(4-Nitrophenyl)-1*H*-1,2,3-benzotriazole: m.p. 238 °C (*cf.* Ref. 13: m.p. 239 °C). ^1H NMR, δ: 7.50, 7.65, 7.85, and 8.20 (all m, 1 H, BTA); 8.38 and 8.52 (both m, 2 H, Ar, J_o = 9.0 Hz). MS: 240 $[M]^+$, 241 $[M + H]^+$, 212 $[M - N_2]^+$, 213 $[M + H - N_2]^+$, 194 $[M - NO_2]^+$, 195 $[M + H - NO_2]^+$, 166 $[M - N_2 - NO_2]^+$, 167 $[M + H - N_2 - NO_2]^+$.

1-(2-Pyridyl)-1*H*-1,2,3-benzotriazole: m.p. 110 °C (*cf.* Ref. 12: m.p. 108—110 °C). ^1H NMR, δ: 7.34, 7.47, 7.63, and 7.95 (all m, 1 H, BTA); 8.14 and 8.33 (both d, 1 H, Py, J_o = 8.3 Hz); 8.40 (m, 1 H, Py); 8.68 (d, 1 H, Py, J_o = 8.3 Hz). MS: 196 $[M]^+$, 197 $[M + H]^+$, 168 $[M - N_2]^+$, 169 $[M + H - N_2]^+$.

1-(3-Nitro-2-pyridyl)-1*H*-1,2,3-benzotriazole: m.p. 154—155 °C (*cf.* Ref. 7: m.p. 153—155 °C). ^1H NMR (DMSO-d_6), δ: 7.65 and 8.25 (both m, 2 H, BTA); 7.88 (dd, 1 H, Py, J_o = 8.0 Hz, J_o' = 4.5 Hz); 8.79 (dd, 1 H, Py, J_o = 8.0 Hz, J_m = 1.5 Hz); 9.0 (dd, 1 H, Py, J_o = 4.5 Hz, J_m = 1.5 Hz). MS: 241 $[M]^+$, 242 $[M + H]^+$, 213 $[M - N_2]^+$, 214 $[M + H - N_2]^+$, 167 $[M - N_2 - NO_2]^+$, 168 $[M + H - N_2 - NO_2]^+$.

1-[4-Nitro-2-(trifluoromethyl)phenyl]-1*H*-1,2,3-benzotriazole: m.p. 131—132 °C. ^1H NMR, δ: 7.33 (d, 1 H, BTA, J_o = 8.4 Hz); 7.47 and 7.57 (both m, 1 H, BTA); 7.76 (d, 1 H, Ar, J_o = 8.4 Hz); 8.17 (d, 1 H, BTA, J_o = 8.4 Hz); 8.64 (dd, 1 H, Ar, J_o = 8.6 Hz, J_m = 2.4 Hz); 8.82 (d, 1 H, Ar, J_m = 2.4 Hz). ^{19}F NMR, δ: −54.0 (s, CF_3). MS: 308 $[M]^+$, 309 $[M + H]^+$, 280 $[M - N_2]^+$, 281 $[M + H - N_2]^+$, 262 $[M - NO_2]^+$, 263 $[M + H - NO_2]^+$. Found (%): C, 50.51; H, 2.22; N, 18.03. $C_{13}H_7F_3N_4O_2$. Calculated (%): C, 50.56; H, 2.29; N, 18.18.

1-[2-Nitro-4-(trifluoromethyl)phenyl]-1*H*-1,2,3-benzotriazole: m.p. 132—133 °C (*cf.* Ref. 9: m.p. 127—128 °C). ^1H NMR, δ: 7.31 (d, 1 H, BTA, J_o = 8.1 Hz); 7.42 (m, 1 H, BTA); 7.75 (d, 1 H, Ar, J_o = 8.6 Hz); 8.10 (d, 1 H, BTA, J_o = 8.1 Hz); 8.60 (dd, 1 H, Ar, J_o = 8.6 Hz, J_m = 2.4 Hz); 8.76 (d, 1 H, Ar, J_m = 2.5 Hz). ^{19}F NMR, δ: −54.5 (s, CF_3). MS: 308 $[M]^+$, 309 $[M + H]^+$, 280 $[M - N_2]^+$, 281 $[M + H - N_2]^+$, 262 $[M - NO_2]^+$, 263 $[M + H - NO_2]^+$. Found (%): C, 46.43; H, 1.14; N, 12.46. $C_{13}H_7F_3N_4O_2$. Calculated (%): C, 46.58; H, 1.20; N, 12.54.

1-[2,3,5,6-Tetrafluoro-4-(trifluoromethyl)phenyl]-1*H*-1,2,3-benzotriazole: m.p. 73—74 °C. ^1H NMR, δ: 7.34 (m, 2 H, BTA); 7.45 (m, 1 H, BTA); 7.95 (d, 1 H, BTA, J = 8.4 Hz). ^{19}F NMR, δ: −56.05 (m, 3 F, CF_3); −140.78 (m, 2 F, Ar); −143.16 (m, 2 F, Ar). MS: 335 $[M]^+$, 336 $[M + H]^+$, 307 $[M - N_2]^+$, 308 $[M + H - N_2]^+$. Found (%): C, 50.52; H, 2.24; N, 18.09. $C_{13}H_4F_7N_3$. Calculated (%): C, 50.56; H, 2.29; N, 18.18.

1-(4-Chloro-2,3,5,6-Tetrafluorophenyl)-1*H*-1,2,3-benzotriazole: m.p. 120—121 °C. ^1H NMR, δ: 7.32 (m, 2 H, BTA); 7.43 (m, 1 H, BTA); 7.92 (d, 1 H, BTA, J = 8.4 Hz). ^{19}F NMR, δ: −139.30 (m, 2 F, Ar); −144.97 (m, 2 F, Ar). MS: 301 $[M]^+$, 303 $[M + 2]^+$, 273 $[M - N_2]^+$, 275 $[M + 2 - N_2]^+$, 239 $[M + 2 - N_2 - Cl]^+$, 238 $[M - N_2 - Cl]^+$. Found (%): C, 47.81; H, 1.26; Cl, 11.64; N, 13.90. $C_{12}H_4ClF_4N_3$. Calculated (%): C, 47.78; H, 1.34; Cl, 11.75; N, 13.93.

1-(2,3,5,6-Tetrafluoro-4-pyridyl)-1*H*-1,2,3-benzotriazole: m.p. 129—130 °C. ^1H NMR, δ: 7.35 (m, 2 H, BTA); 7.47 (m, 1 H, BTA); 8.00 (d, 1 H, BTA, J = 8.5 Hz). ^{19}F NMR, δ: −87.35 and −153.65 (both m, 2 F, Ar). MS: 268 $[M]^+$, 269 $[M + 1]^+$, 240 $[M - N_2]^+$, 241 $[M + 1 - N_2]^+$. Found (%): C, 49.24; H, 1.45; N, 20.81. $C_{11}H_4F_4N_4$. Calculated (%): C, 49.27; H, 1.50; N, 20.89.

1-[2,6-Dinitro-4-(trifluoromethyl)phenyl]-1*H*-1,2,3-benzotriazole: m.p. 215—216 °C (*cf.* Ref. 9: m.p. 216 °C). ^1H NMR, δ: 7.30, 7.55, 7.65, and 8.23 (all m, 1 H, BTA); 9.0 (s, 2 H, Ar). ^{19}F NMR, δ: −57.2 (s, CF_3). MS: 353 $[M]^+$, 354 $[M + H]^+$, 325 $[M - N_2]^+$, 326 $[M + H - N_2]^+$, 307 $[M - NO_2]^+$, 308 $[M + H - NO_2]^+$, 279 $[M - N_2 - NO_2]^+$, 280 $[M + H - N_2 - NO_2]^+$.

1-[2,4-Dinitro-6-(trifluoromethyl)phenyl]-1*H*-1,2,3-benzotriazole: m.p. 169 °C. ^{1}H NMR, δ: 7.24, 7.49, and 7.58 (all m, 1 H, BTA); 8.17 (d, 1 H, BTA, J = 8.4 Hz); 9.02 and 9.13 (both d, 1 H, Ar, J_m = 2.5 Hz). ^{19}F NMR, δ: −59.8 (s, CF_3). MS: 353 $[M]^+$, 354 $[M + H]^+$, 325 $[M - N_2]^+$, 326 $[M + H - N_2]^+$, 307 $[M - NO_2]^+$, 308 $[M + H - NO_2]^+$, 279 $[M - N_2 - NO_2]^+$, 280 $[M + H - N_2 - NO_2]^+$. Found (%): C, 44.20; H, 1.70; N, 19.91. $C_{13}H_6F_3N_5O_4$. Calculated (%): C, 44.21; H, 1.71; N, 19.83.

4-(1*H*-1,2,3-Benzotriazol-1-yl)-3,5-dinitrobenzonitrile: m.p. 201 °C. ^{1}H NMR, δ: 7.50 (m, 1 H, BTA); 7.60 (m, 2 H, BTA); 8.13 (m, 1 H, BTA); 9.12 (s, 1 H, Ar). MS: 310 $[M]^+$, 311 $[M + H]^+$, 282 $[M - N_2]^+$, 283 $[M + H - N_2]^+$, 264 $[M - NO_2]^+$, 265 $[M + H - NO_2]^+$, 236 $[M - N_2 - NO_2]^+$, 237 $[M + H - N_2 - NO_2]^+$. Found (%): C, 50.22; H, 1.87; N, 27.00. $C_{13}H_6N_6O_4$. Calculated (%): C, 50.33; H, 1.95; N, 19.09.

Ethyl 4-(1*H*-1,2,3-benzotriazol-1-yl)-3,5-dinitrobenzoate: m.p. 148–149 °C. ^{1}H NMR, δ: 1.50 (t, 3 H, CH_3); 4.56 (q, 2 H, CH_2); 7.28, 7.49, 7.59, and 8.17 (all m, 1 H, BTA); 8.93 (s, 1 H, Ar). MS: 357 $[M]^+$, 358 $[M + H]^+$, 329 $[M - N_2]^+$, 330 $[M + H - N_2]^+$, 311 $[M - NO_2]^+$, 312 $[M + H - NO_2]^+$, 301 $[M - N_2 - NO_2]^+$, 302 $[M + H - N_2 - NO_2]^+$. Found (%): C, 50.36; H, 3.04; N, 19.54. $C_{15}H_{11}N_5O_6$. Calculated (%): C, 50.43; H, 3.10; N, 19.60.

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