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5-Benzyl-1*H*-tetrazols from the reaction of 1-aryl-5-methyl-1*H*-tetrazoles with 1,2-dehydrobenzene

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Abstract—A series of 1-aryl-5-benzyl-1*H*-tetrazoles has been obtained during the reaction of 1-aryl-5-methyl-1*H*-tetrazoles with 1,2-dehydrobenzene. The mechanism of product formation was investigated.

Tetrazoles are an increasingly popular functionality[1] with wide ranging applications. They have found use in pharmaceuticals as lipophilic spacers and carboxylic acid surrogates,[2] in speciality explosives,[3] photography, and information recording systems,[4] not to mention as precursors to a variety of nitrogen containing heterocycles.[5] Tetrazole analogues of fatty acid esters are known as substrates for *N*-myristoyl transferase and its respective coenzyme, and show fungicidal and antiviral (including HIV) activity.[6] A classical synthesis of tetrazoles[7] involves the reaction of amides with phosphorus(V) chloride to form imidoyl chlorides as intermediates. Thereafter, the reaction of imidoyl chlorides with sodium azide or hydrazoic acid afforded the corresponding tetrazoles.[7b] In the field of heterocyclic synthesis, the chemistry of tetrazoles in cycloaddition reactions has been extensively investigated.[8–10] Huisgen et al. reported that tetrazoles undergo ring cleavage, in high temperature boiling solvents, to afford the corresponding nitrile imines through extrusion of a nitrogen molecule.[9] The nitrile imines react with dipolarophiles by [2π+3π]cycloaddition to produce pyrazole derivatives.[9] 1,2-Dehydrobenzene (*o*-benzyne) is an example of the numerous dienophilic arynes which have been added to a variety of dienes.[10,11] Sometime ago, we examined the reaction of 1,2-dehydrobenzene with aromatic diimines,[12] azomethine compounds with [2.2]paracyclophane moieties[13] and ethenyl[2.2]paracyclophanes.[14] Recently, we demonstrated a general methodology for the construction of a variety of pleiadene and perimidin derivatives by donor-acceptor interactions.[15] Our synthetic program uses efficient and facile methods for the preparation of novel heterocyclic compounds,[12,15,16a–f] heterophanes[16g,h] and polycyclic cyclophanes.[14,16h,i] Due to the course of studies on the synthetic applications of the reaction of 1,2-dehydrobenzene (*o*-benzyne),[17] we wish to report on the reactivity of 1-aryl-5-methyl-1*H*-tetrazoles towards 1,2-dehydrobenzene.

We chose the tetrazoles **3a–d**, which contain electron donating and withdrawing substituents on the benzene ring in order to examine their reactivity during the reactions with 1,2-dehydrobenzene.

Scheme 1 outlines the sequence of the synthesis of tetrazoles **3a–d** from the reaction of imidoyl chloride with hydrazoic acid using the methodology established in reference [7b]. With the aim to synthesize the amide **1d**,[18] we reacted 4-amino[2.2]paracyclophane (**4**)[19] with acetyl chloride in the presence of sodium hydroxide. Thus, **1d** was subjected to hydrazoic acid,[7b] after formation of its imidoyl chloride **2d**, to form **3d**[20] (Scheme 1). Thereafter, we carried out the reaction[21] between 1-aryl-5-methyl-1*H*-tetrazoles **3a–d** and 1,2-dehydrobenzene **5**, which was generated by diazotization of 1,2-anthranilic acid.[22] To our surprise, the reaction gave, after chromatographic purification and recrystallization, the 5-benzyltetrazoles **6a–d** (Scheme 1). The main structural feature of tetrazoles **3a–d** is the amidine group $R^1N–C(CH_3)=N–$. A pioneering study showed that **5** reacted with formamidines via a [2π+2π]cycloaddition reaction

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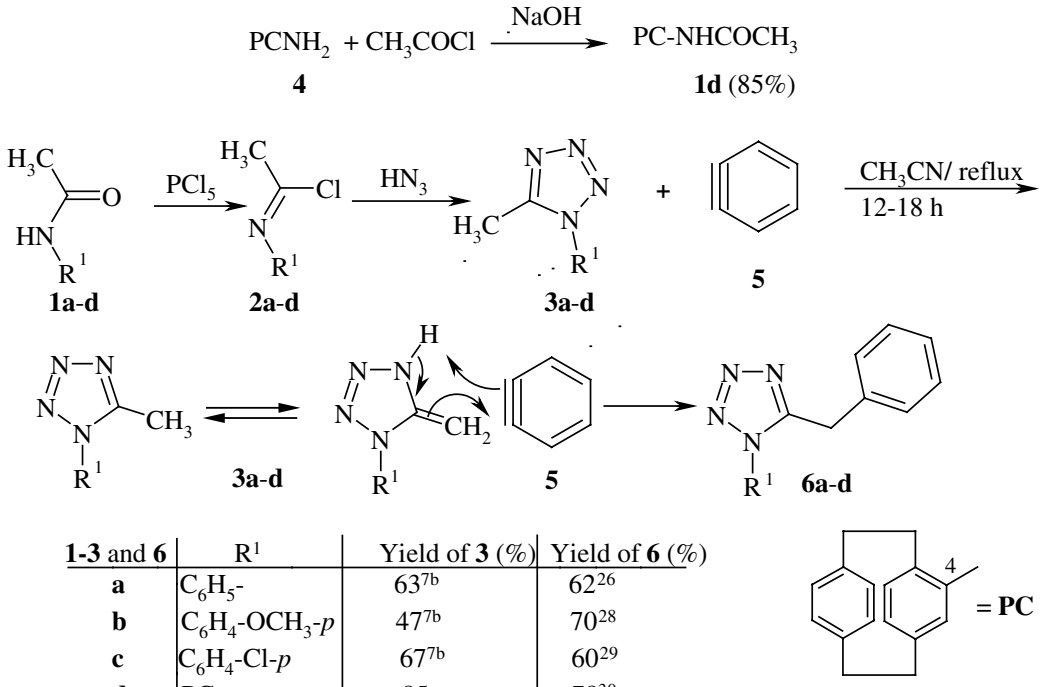

1-3 and 6	R^1	Yield of 3 (%)	Yield of 6 (%)
a	C_6H_5-	63[7b]	62[26]
b	C_6H_4-OCH_3-*p*	47[7b]	70[28]
c	C_6H_4-Cl-*p*	67[7b]	60[29]
d	PC-	85	78[30]

Scheme 1. Synthesis of 1-aryl-5-methyl-1*H*-tetrazoles and their reactions with 1,2-dehydrobenzene.

to give benzazetidines, which undergo spontaneous ring opening to a transient azaxylylenes followed by electrocyclization and aromatization to produce the corresponding acridines.[23] Conversely it was reported that acetamidines reacted with ylidenes by enamine-like reactions.[24] Moreover, the relevance of the *N*, C-tautomerism of cyclic *N*-methyl amidines in their reactions with electrophilic carbon skeletons has been firmly established.[25] Recently, we have noted similar behavior of the acetylnitrone derived by [2.2]paracyclophane towards dipolarophiles.[26] In our case, the formation of **6a–d** may be based on an analogous step involving an initial tautomerization of the R^1N–$C(CH_3)$=N– group in **3a–d** into an enamine R^1N–$C(=CH_2)$–NH– (Scheme 1). Thereafter the enamine can undergo an ene-addition with **5** to furnish **6a–d** (Scheme 1).

The structure of **6a**[27] was confirmed by detailed spectroscopic analysis and comparison of its spectroscopic data with those reported by Rappoport and Gazit.[28] The spectral data of the tetrazoles **6b**,[29] **6c**[30] and **6d**[31] confirmed the presence of benzyltetrazoles. The IR spectrum of **6b** showed strong sharp bands corresponding to the absorption of the azido-tetrazole groups. Mass spectroscopy indicated the molecular ion peak of **6b** at *m/z* 266 (4%) and a fragmentation peak at *m/z* 91 related to $PhCH_2^+$. Also, an ion peak at *m/z* 238 (60%) appeared in the mass spectrum of **6b** corresponding to the fragmentation pattern $[M^+-N_2]$. Both the mass spectrum and elemental analysis established the molecular formula of **6b** as $C_{15}H_{14}N_4O$. The characteristic features of the ^1H NMR spectrum of **6b**[29] as were two singlets at δ_H 3.90 and 4.60, the methoxy and the benzyl protons, respectively. The ^{13}C NMR spectrum supported the ^1H NMR spectroscopic data by the appearance of the methoxy, aliphatic-benzyl and *C*-5 carbons as three distinctive carbon signals at δ_C 50.90, 56.00 and 160.00, respectively. The aromatic carbon of the phenyl group attached directly to the *N*-1 (Ph-*C*-1) resonated in the ^{13}C NMR spectrum of **6b** as an aromatic carbon signal at δ_C 138.90, whereas the carbon of the same phenyl group attached directly to the methoxy group appeared at δ_C 148.60. The ^1H NMR spectrum of **6d** apparently showed the aliphatic-benzyl protons superimposed on the etheylenic-CH_2 of the paracyclophane protons. The H-5 of the paracyclophane-protons appeared as a doublet at δ_H 6.20 ($J_{H,H}$ = 1.4 Hz). The benzylic-CH_2 carbon in **6d** resonated in its ^{13}C NMR spectrum at δ_C 52.00, whereas four distinctive carbon signals of PC-*C* H-5, Ph-*C*-1, PC-*C*-4 and azomethine-*C*-5 appeared at δ_C 118.00, 138.00, 138.90 and 158.60, respectively.

We have now demonstrated a very convenient procedure to synthesize 1-aryl-5-benzyl-1*H*-tetrazoles from the reaction of 1-aryl-5-methyl-1*H*-tetrazoles with 1,2-dehydrobenzene. The advantages of this method are the reasonable yields, and the ease with which the reaction can be carried out as a one-pot procedure with readily available starting materials.

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18. *N*-(4′-[2.2]Paracyclophanyl)acetamide (**1d**): To a two-necked flask (250 mL) containing a cold (0 °C) stirred mixture of **4**[19] (1.12 g, 5 mmol) dissolved in methanol (150 mL) was added sodium hydroxide (0.20 g, 5 mmol) dissolved in water (10 mL). To the reaction mixture, acetyl chloride (0.37 g, 5 mmol) was added dropwise over 10 min. The mixture was stirred at −15 °C for 30 min. and at rt for 1 h. The precipitate obtained was collected by filtration and washed several times with water. Colorless crystals (R_f 0.3, CH_2Cl_2); yield 1.1 g (85%); mp 146 °C (cyclohexane); [Found: C, 81.65; H, 7.20; N, 5.20%. $C_{18}H_{19}NO$ (265.36) requires C, 81.47; H, 7.22; N, 5.28%]; ν_{max} (KBr) 3220 (NH) cm^{-1}, 3060–2990 (Ar-CH), 2890–2860 (Aliph.-CH), 1682 (CO-amide); δ_H (400 MHz, $CDCl_3$) 2.20 (s, 3H, CH_3), 2.30–2.70 (m, 7H, CH_2–CH_2), 3.20–3.35 (m, 1H, CH_2–CH_2), 4.10 (br, s, 1H, NH), 6.20 (d, 1 H, H-5, $J_{H,H}$ = 7.8 Hz), 6.40–6.74 (m, 6H, PC-H); δ_C (100.6 MHz, $CDCl_3$) 18.80 (*C*H_3), 31.60, 32.80, 33.60, 34.40 (*C*H_2-*C*H_2), 120.80 (PC-*C*H-5) 126.90, 130.10, 130.90, 132.00, 132.60 (PC-*C*H), 132.80 (PC-*C*), 134.00 (PC-*C*H), 134.80, 135.20, 135.90 (PC-*C*), 138.00 (PC-*C*-4), 166.00 (*C*O-amide); *m*/*z* (EI) 266 [M+1] (24), 265 [M^+] (100), 250 (40), 222 (24), 160 (40), 146 (28), 104 (65), 84 (22), 78 (18).
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20. 5-Methyl-1-(4′-[2.2]paracyclophanyl)-1*H*-tetrazole (**3d**): To a solution of **1d** (0.79 g, 3 mmol) in dry benzene (200 mL) under N_2 was added phosphorus(V) chloride in portions over 20 min., (0.63 g, 3 mmol). The reaction was followed by TLC analysis until the consumption of the starting materials was completed and a new product of imide chloride appeared. To the reaction mixture, a solution of hydrazoic acid (0.15 g, 3.5 mmol)[7b] in dry benzene (10 mL) was added over 10 min. The mixture was refluxed in a water bath for 8 h. The solvent was removed under reduced pressure and the residue was treated with ice and water to decompose any phosphorus oxychloride present. The organic layer was extracted with chloroform and washed several times with water. The solvent was removed in vacuo and the residue was treated with absolute ethanol (150 mL) to give **3d** which was collected by filtration. Colorless crystals (R_f 0.4, CH_2Cl_2); yield 0.74 g (85%); mp 180 °C (acetonitrile); [Found: C, 74.60; H, 6.20; N, 19.20%. $C_{18}H_{18}N_4$ (290.37) requires C, 74.46; H, 6.25; N, 19.29%]; ν_{max} (KBr) 3021–2962 cm^{-1} (Ar-CH), 2930–2852 (Aliph.-CH), 1597, 1519, 1494, 1451, 1428, 1387, 1380, 1286, 1270, 1113, 1077, 1039, 983, 959; λ_{max} (CH_3CN, log ε) 260 nm (3.40); δ_H (400 MHz, $CDCl_3$) 2.38 (s, 3H, CH_3), 2.50–2.56 (m, 1H, CH_2–CH_2), 2.80–3.25 (m, 7H, CH_2–CH_2), 6.55-6.68 (m, 6H, PC-H), 6.87 (dd, 1H, PC-H, $J_{H,H}$ = 8.0, 1.4 Hz); δ_C (100.6 MHz, $CDCl_3$) 10.80 (*C*H_3), 32.00, 35.00, 35.40, 35.80 (*C*H_2-*C*H_2), 126.40, 131.80, 132.70, 133.60, 134.40, 134.90, 135.20 (PC-*C*H), 135.60, 136.20, 138.30, 139.10 (PC-*C*), 142.40 (PC-*C*-4), 154.40 (azomethine-*C*-5); *m*/*z* (EI) 290 [M^+] (20), 262 (8), 221 (20), 208 (10), 158 (76), 130 (44), 105 (26), 104 (100), 84 (12), 78 (16), 56 (18).
21. Reaction of **3a**–**d** with **5**; General Procedure: Benzenediazonium carboxylate was prepared by the same procedure as described in reference.[22] Under nitrogen atmosphere, **5** (4 mmol) was added slowly to the stirred and refluxed solutions of **3a**–**d** (2 mmol) in dry acetonitrile (250 mL) over 1 h. The reaction mixture was refluxed until the consumption of the starting materials was completed (reaction progress monitored by TLC analysis) in 12–18 h. The solvent was concentrated and the residue was filtered off. The precipitate was washed with dichloromethane (200 mL). The filtrate was concentrated under vacuum and the residue was applied to PLC on silica gel using toluene: ethyl acetate (10:1) as eluent. The main zones containing compounds **6a**–**d** were extracted with acetone and the recovered products were recrystallized from the stated solvents.
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27. 5-Benzyl-1-phenyl-1*H*-tetrazole (**6a**): Yellow crystals (R_f 0.5, CH_2Cl_2); yield 0.29 g (62%); mp 125–126 °C (ethanol)

(lit.[28] mp 125 °C); [Found: C, 71.00; H, 5.10; N, 23.65%. $C_{14}H_{12}N_4$ (236.28) requires C, 71.17; H, 5.12; N, 23.71%]; ν_{max} (KBr) 3020–3000 cm^{-1} (Ar–CH), 2960–2940 (Aliph.-CH), 1590, 1525, 1494, 1460, 1392, 1380, 1290, 1012, 980, 960; δ_H (400 MHz, $CDCl_3$) 4.25 (s, 2H, CH_2-Ph), 7.30–7.60 (m, 10 H).

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29. 5-Benzyl-1-(4′-methoxyphenyl)-1*H*-tetrazole (**6b**): Pale yellow crystals (R_f 0.6, CH_2Cl_2); yield 0.37 g (70%); mp 170–172 °C (acetonitrile); [Found: C, 67.45; H, 5.20; N, 21.00%. $C_{15}H_{14}N_4O$ (266.31): C, 67.65; H, 5.30; N, 21.04%]; ν_{max} (KBr) 3040–3008 cm^{-1} (Ar-CH), 2970–2960 (Aliph.-CH), 1594, 1540, 1490, 1464, 1396, 1390, 1296, 1018, 990, 972; λ_{max} (CH_3CN, log ε) 340 nm (3.80); δ_H (400 MHz, $CDCl_3$) 3.90 (s, 3H, OCH_3), 4.60 (s, 2H, $PhCH_2$), 6.40 (dd, 2H, $J_{H,H}$ = 8.2, 1.4 Hz, Ph-H), 6.62 (dd, 2H, $J_{H,H}$ = 8.2, 1.6 Hz, Ph-H), 7.40–7.60 (m, 5H, Ph-H); δ_C (100.6 MHz, $CDCl_3$) 50.90 (O*C*H_3), 56.00 (*C*H_2-Ph), 126.80, 128.00, 128.60, 134.80, 136.40 (Ph-*C*H), 138.40 (Ph-*C*-CH_2), 138.90 (Ph-*C*-N), 148.60 (Ph-*C*-OCH_3), 160.00 (azomethine-*C*-5); *m*/*z* (EI) 266 [M^+] (4), 238 [M^+-N_2] (60), 224 (20), 195 (56), 174 (26), 146 (24), 116 (18), 91 (100), 78 (34).

30. 5-Benzyl-1-(4′-chlorophenyl)-1*H*-tetrazole (**6c**): Pale yellow crystals (R_f 0.6, CH_2Cl_2); yield 0.32 g (60%); mp 130-132 °C (acetonitrile); [Found: C, 62.28; H, 4.10; N, 20.80%. $C_{14}H_{11}ClN_4$ (270.72) requires C, 62.11; H, 4.10; N, 20.70%]; ν_{max} (KBr) 3036–3012 cm^{-1} (Ar-CH), 1590, 1542, 1480, 1460, 1394, 1384, 1290, 1020, 990, 970; λ_{max} (CH_3CN, log ε) 330 nm (3.60); δ_H (400 MHz, $CDCl_3$) 4.50 (s, 2H, $PhCH_2$), 6.00 (dd, 2H, $J_{H,H}$ = 8.2, 1.2 Hz, Ph-H), 6.20 (dd, 2H, $J_{H,H}$ = 8.2, 1.4 Hz, Ph-H), 7.30–7.50 (m, 5H, Ph-H); δ_C (100.6 MHz, $CDCl_3$) 55.20 (*C*H_2-Ph), 125.60 (Ph-*C*-Cl), 126.80, 128.00, 130.00, 132.40, 134.20 (Ph-*C*H), 138.00 (Ph-*C*-CH_2), 136.40 (Ph-*C*-N), 158.80 (azomethine-*C*-5); *m*/*z* (EI) 272 [M+2] (14), 270 [M^+] (10), 242 [M^+-N_2] (46), 224 (20), 195 (56), 174 (26), 148 (22), 146 (24), 116 (20), 91 (100), 78 (30).

31. 5-Benzyl-1-(4′-[2.2]paracyclophanyl)-1*H*-tetrazole (**6d**): Colorless crystals (R_f 0.4, CH_2Cl_2); yield 0.57 g (78%); mp 200–202 °C (acetonitrile); [Found: C, 78.50; H, 6.00; N, 15.20%. $C_{24}H_{22}N_4$ (366.47) requires C, 78.66; H, 6.05; N, 15.29%]; ν_{max} (KBr) 3040–3010 cm^{-1} (Ar-CH); 2980–2960 (Aliph.-CH), 1600, 1520, 1490, 1450, 1390, 1375, 1286, 1080, 1040, 983, 970; λ_{max} (CH_3CN, log ε) 330 nm (3.40); δ_H (400 MHz, $CDCl_3$) 2.40–2.50 (m, 1H, CH_2-CH_2), 3.20–3.96 (m, 9H, $PhCH_2$, CH_2-CH_2), 6.20 (d, 1H, $J_{H,H}$ = 1.4 Hz, PC-CH-5), 6.50–6.72 (m, 6H, PC-H), 7.20–7.46 (m, 3H, Ph-H), 7.90–8.12 (m, 2H, Ph-H); δ_C (100.6 MHz, $CDCl_3$) 33.80, 34.00, 34.40, 34.80 (*C*H_2-CH_2), 52.00 (*C*H_2-Ph), 118.00 (PC-*C*H-5), 126.90, 128.60, 130.00, 132.00, 132.40, 132.60, (PC-*C*H, Ph-*C*H), 133.00, 133.30, 133.60 (PC-*C*, Ar-*C*), 134.20, 134.60, 134.80 (PC-CH), 135.10 (PC-*C*), 138.00 (Ph-*C*-1), 138.90 (PC-*C*-4), 158.60 (azomethine-*C*-5); *m*/*z* (EI) 366 [M^+] (18), 340 [M^+-N_2] (38), 264 (20), 234 (40), 220 (28), 104 (100), 92 (50), 78 (26).