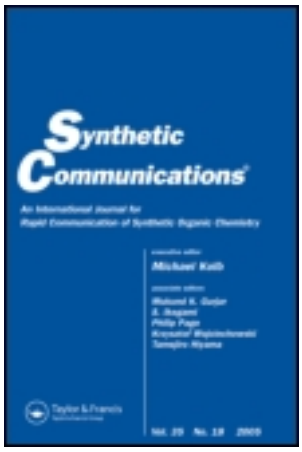

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SYNTHESIS AND REACTIONS OF SOME 1,2,4-TRIAZOLO-[4,3-b]-1,2,4-TRIAZOLES

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SYNTHESIS AND REACTIONS OF SOME 1,2,4-TRIAZOLO-[4,3-b]-1,2,4-TRIAZOLES

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ABSTRACT

Reaction of 5-aryl-3-chloro-4-phenyl-1,2,4-triazoles (**1** & **2**) with hydrazine hydrate afforded the hydrazino derivatives **3** and **4**. Reaction of **3** and **4** with carboxylic acids, CS_2 and phenylisothiocyanate gave the s-triazole-fused ring systems **5**–**12**. On the other hand, reaction of **3** and **4** with nitrous acid gave tetrazole-fused ring systems **13** and **14**, respectively. Mannich reaction on compounds **9** and **10** afforded Mannich bases **15** and **16**.

Certain compounds containing 1,2,4-triazole nucleus have been reported to possess fungicidal,[1,2] insecticidal,[3] antimicrobial,[4,5] bactericidal,[6] and antiviral[7] activity. Also, some triazole derivatives have been synthesized as anticonvulsants,[8,9] and plant growth regulators.[10] The present work aims to synthesize a special group of heterocyclic compounds in which the 1,2,4-triazole nucleus is fused with other heterocyclic rings, which may exert some reasonable biological activity.

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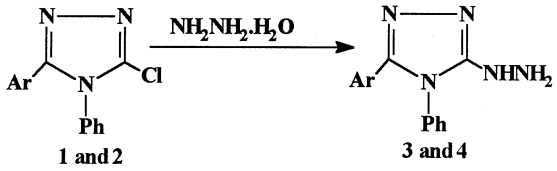
RESULTS AND DISCUSSION

The starting materials 4,5-diphenyl- and 4-phenyl-5-(4-chloro-phenyl)-3-chloro-1,2,4-triazoles (**1** & **2**) were prepared by oxidative chlorination of the corresponding 3-mercapto-derivatives using the method of Deshpande et al.[11]

The chemical structure of the new compound **2** was confirmed by elemental and spectral analyses.

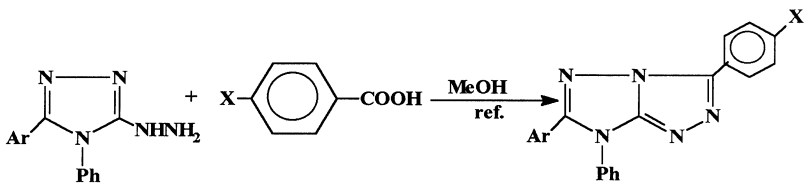
The starting triazoles **3** and **4** were prepared by refluxing 3-chloro-derivatives (**1** & **2**) with excess of hydrazine hydrate 98% in ethanol.



1 and 2 **3 and 4**

1 and 3; Ar = Ph
2 and 4; Ar = p-Cl-Ph

Treatment of compounds **3** and **4** with aromatic acids furnished the corresponding 1,2,4-triazolo[4,3-b]-1,2,4-triazoles **5** and **6**, respectively. The structure of these products was confirmed using elemental and spectral analyses (cf Table).



3 and 4 **5a-d and 6a-d**

3 and 5a-d; Ar = Ph
4 and 6a-d; Ar = p-Cl-Ph

Comp. No	X
5a, 6a	Cl
5b, 6b	OH
5c, 6c	NO_2
5d, 6d	CH_3

Compounds **3** and **4** underwent readily ring closure with formic acid and acetic acid to give 1,2,4-triazolo[4,3-b]-1,2,4-triazoles **7** and **8**.

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Table. Analytical and Spectral Data of the Prepared Compounds

Comp. No.	M.p. Cryst. Solv	Yield (%)	M_F (M_W)	Analytical data Calc. (Found) (%)		
				C	H	N
2	196 Ethanol	80	$C_{14}H_9N_3Cl_2$ (290)	57.93 (58.29)	3.10 (2.84)	14.48 (14.17)
3	167 Ethanol	70	$C_{14}H_{13}N_5$ (251)	66.93 (66.65)	5.18 (4.89)	27.89 (27.60)
4	207 Ethanol	72	$C_{14}H_{12}N_5Cl$ (285.5)	58.84 (59.51)	4.20 (3.90)	24.52 (24.81)
5_a	119 Ethanol	59	$C_{21}H_{14}N_5Cl$ (371.5)	67.83 (67.49)	3.77 (4.06)	18.84 (18.52)
5_b	105 Ethanol	50	$C_{21}H_{15}N_5O$ (353)	71.39 (71.72)	4.25 (3.97)	19.83 (19.41)
5_c	173 Ethanol	48	$C_{21}H_{14}N_6O_2$ (382)	65.97 (65.64)	3.67 (3.29)	21.99 (21.68)
5_d	100 Ethanol	62	$C_{22}H_{17}N_5$ (351)	75.21 (75.70)	4.84 (5.07)	19.94 (19.44)
6_a	130 Ethanol	60	$C_{21}H_{13}N_5Cl_2$ (406)	62.07 (61.75)	3.20 (3.01)	17.24 (16.88)
6_b	126 Ethanol	47	$C_{21}H_{14}N_5OCl$ (387.5)	65.03 (64.87)	3.61 (3.34)	18.07 (17.59)
6_c	110 Ethanol	42	$C_{21}H_{13}N_6O_2Cl$ (416.5)	60.50 (60.82)	3.12 (2.87)	20.17 (19.89)
6_d	195 Ethanol	67	$C_{22}H_{16}N_5Cl$ (385.5)	68.48 (68.86)	4.15 (4.52)	18.16 (17.87)
7_a	155 Acetic acid	65	$C_{15}H_{11}N_5$ (261)	68.97 (68.54)	4.22 (4.42)	26.82 (26.51)
7_b	135 Ethanol	61	$C_{16}H_{13}N_5$ (275)	69.82 (69.51)	4.73 (5.04)	25.46 (25.19)
8_a	225 Acetic acid	64	$C_{15}H_{10}N_5Cl$ (295.5)	60.91 (60.58)	3.38 (3.11)	23.69 (23.25)
8_b	160 Ethanol	57	$C_{16}H_{12}N_5Cl$ (309.5)	62.04 (62.45)	3.88 (3.56)	22.62 (22.93)
9	259 Ethanol	65	$C_{15}H_{11}N_5S$ (293)	61.43 (61.83)	3.75 (3.40)	23.89 (23.47)
10	274 Ethanol	60	$C_{15}H_{10}N_5SCl$ (327.5)	54.96 (54.62)	3.05 (2.68)	21.37 (21.04)
11	175 Ethanol	43	$C_{21}H_{16}N_6$ (352)	71.59 (71.98)	4.55 (4.94)	23.86 (23.43)
12	98 Ethanol	49	$C_{21}H_{15}N_6Cl$ (386.5)	65.20 (65.61)	3.88 (3.59)	21.73 (22.11)
13	145 Ethanol	80	$C_{14}H_{10}N_6$ (262)	64.12 (64.51)	3.82 (3.46)	32.06 (31.74)
14	129 Ethanol	74	$C_{14}H_9N_6Cl$ (296.5)	56.66 (57.13)	3.04 (3.45)	28.33 (28.06)
15_a	236 Benzene	80	$C_{20}H_{20}N_6OS$ (392)	61.23 (61.58)	5.10 (4.80)	21.43 (21.07)
15_b	141 (Pet. Ether 60–80)	65	$C_{20}H_{22}N_6S$ (378)	63.49 (63.96)	5.82 (5.51)	22.22 (22.53)
15_c	124 Cyclohexan	64	$C_{23}H_{20}N_6S$ (412)	66.99 (66.49)	4.85 (4.56)	20.39 (20.07)
15_d	138 Cyclohexan	67	$C_{21}H_{17}N_7S$ (399)	63.16 (63.63)	4.26 (3.98)	24.56 (24.84)
15_e	120 Cyclohexan	60	$C_{24}H_{20}N_6OS$ (440)	65.46 (65.16)	4.55 (4.22)	19.09 (18.87)
16_a	138 Ethanol	74	$C_{20}H_{19}N_6OSCl$ (426.5)	56.27 (56.75)	4.46 (4.17)	19.70 (19.36)
16_b	110 Benzene	70	$C_{20}H_{21}NSCl$ (412.5)	58.18 (58.49)	5.09 (5.41)	20.36 (20.01)
16_c	97 Benzene	82	$C_{23}H_{19}N_6SCl$ (446.5)	61.81 (62.24)	4.26 (4.58)	18.81 (18.54)
16_d	93 Cyclohexan	62	$C_{21}H_{16}N_7SCl$ (433.5)	58.13 (58.64)	3.69 (3.38)	22.61 (22.47)
16_e	109 Cyclohexan	60	$C_{24}H_{19}N_6SOCl$ (474.5)	60.70 (60.31)	4.00 (4.32)	17.70 (17.35)

(*continued*)

The elemental and spectral analyses of these compounds were in agreement with their structures (cf Table).

Reaction of compounds **3** and **4** with CS_2 in ethanolic potassium hydroxide afforded 3-mercapto-1,2,4-triazolo[4,3-b]-1,2,4-triazoles **9** and **10**, respectively.

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Table. Continued

Comp. No.	IR (KBr) ν (cm^{-1})	^{1}H-NMR δ (ppm) (a, $CDCl_3$; b, DMSO)
2	698 (C–Cl).	7.8–7.3 (m, 4H, arom.).[a]
3	3412, 3225, 3217.7 (NH_2+NH).	8.0–7.4 (br, 10H, arom. + NH), 3.6 (br, 2H, NH_2).[a]
4	3412.5, 3337, 325 (NH_2+NH).	10.8–10.6 (br, 1H, NH), 4.3 (br, 2H, NH_2).[a]
5_a	765 (C–Cl).	7.7–7.1 (m, 14H, arom.).[a]
5_b	3425–3209 (OH).	7.8–7.1 (m, 14H, arom.); 2.2 (s, 1H, OH).[a]
5_c	1523, 1334 (NO_2).	8.1–7.0 (m, 14H, arom.).[a]
5_d	2960 (CH, aliph.).	7.7–7.2 (m, 14H, arom.); 2.1 (s, 3H, CH_3).[a]
6_a	800 (C–Cl).	7.6–7.1 (m, 13H, arom.).[a]
6_b	3450–3300 (OH).	7.7–7.1 (m, 13H, arom.); 2.2 (s, 1H, OH).[a]
6_c	1520, 1300 (NO_2).	8.2–7.2 (m, 13H, arom.).[a]
6_d	2940 (CH, aliph.).	7.7–7.2 (m, 13H, arom.); 2.3 (s, 3H, CH_3).[a]
7_a	1603 (C = N).	8.9 (s, 1H, = CH); 7.7–7.2 (m, 10H, arom.).[a]
7_b	2913 (CH, aliph.), 1618 (C = N).	7.7–7.2 (m, 10H, arom.); 2.2 (s, 3H, CH_3).[a]
8_a	1611 (C = N).	8.9 (s, 1H, = CH); 7.7–7.2 (m, 9H, arom.).[a]
8_b	2909 (CH, aliph.), 1601 (C = N).	7.7–7.2 (m, 9H, arom.); 2.2 (s, 3H, CH_3).[a]
9	3420 (NH), 2764 (SH), 1186 (C = S)	11.5 (br, 0.6H, NH), 7.8–7.1 (m, 10H, arom), 3.1 (s, 0.4H, SH).[b]
10	3420 (NH), 2764 (SH), 1186 (C = S).	11.5 (br, 0.65H, NH), 7.8–7.1 (m, 9H, arom.), 3.2 (s, 0.35H, SH).[b]
11	3420 (NH).	8.0–7.1 (m, 16H, arom. + NH).[b]
12	3420 (NH).	8.0–7.1 (m, 15H, arom. + NH).[b]
13	1612 (C = N).	7.9–7.3 (m, 10H, arom.).[a]
14	1607 (C = N).	7.8–7.2 (m, 9H, arom.).[a]

(*continued*)

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Table. Continued

15_a	2961, 2870 (CH, aliph.), 1179 (C = S).	7.7–7.3 (m, 10H, arom.); 5.2 (s, 2H, NCH_2N); 3.9–3.5 (m, 4H, CH_2OCH_2); 3.0–2.8 (m, 4H, CH_2NCH_2).[a]
15_b	2980, 2888 (CH, aliph.), 1170 (C = S).	7.7–7.2 (m, 10H, arom.); 5.2 (s, 2H, NCH_2N); 3.7–3.2 (q, 4H, $2CH_2$); 1.4–1.0 (t, 6H, $2CH_3$).[a]
15_c	2969 (CH_2 aliph.), 1190 (C = S).	7.8–7.0 (m, 16H, arom.+NH); 5.8 (s, 2H, NCH_2N); 4.0–3.8 (d, 2H, CH_2Ph).[a]
15_d	2988 (CH, aliph.), 1177 (C = S).	8.2 (S, 1H, NH); 7.7–7.2 (m, 14H, arom.); 5.8 (s, 2H, CH_2).[a]
15_e	2969 (CH, aliph.), 1690 (C = O), 1187 (C = S).	7.9–7.0 (m, 14H, arom.); 6.3 (S, 1H, NH); 5.8 (s, 2H, CH_2); 2.5 (S, 3H, CH_3).[a]
16_a	2961, 2869 (CH, aliph), 1183 (C = S).	7.7–7.3 (m, 9H, arom.); 5.2 (s, 2H, NCH_2N); 3.9–3.5 (m, 4H, CH_2OCH_2); 3.0–2.8 (m, 4H, CH_2NCH_2).[a]
16_b	2963, 2889 (CH, aliph.), 1176 (C = S).	7.9–7.2 (m, 9H, arom.); 5.3 (s, 2H, NCH_2N); 3.5–3.0 (q, 4H, $2CH_2$); 1.8–1.0 (t, 6H, $2CH_3$).[a]
16_c	2967 (CH, aliph.), 1181 (C = S).	7.8–7.1 (m, 15H, arom.+NH); 5.8 (s, 2H, NCH_2N); 3.8–3.4 (d, 2H, CH_2Ph).[a]
16_d	2987 (CH, aliph.), 1189 (C = S).	8.9 (S, 1H, NH); 8.5–7.2 (m, 13H, arom.); 5.2 (s. 2H, CH_2).[a]
16_e	2961 (CH, aliph.), 1687 (C = O), 1183 (C = S).	7.9–7.0 (m, 13H, arom.); 6.0 (S, 1H, NH); 5.3 (s, 2H, CH_2); 2.5 (S, 3H, CH_3).[a]

The elemental and spectral analyses of these compounds were in agreement with their structures.

Refluxing compound **3** with phenylisothiocyanate in dry pyridine for 20 h gave a mixture of triazolo[4,3-b]-1,2,4-triazoles **9** and **11**, which was separated by aq. sodium hydroxide solution. This reaction presumably

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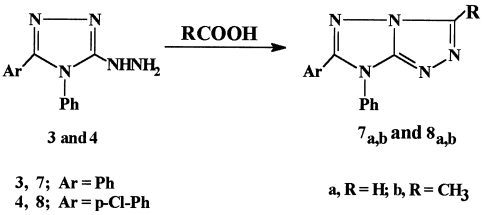
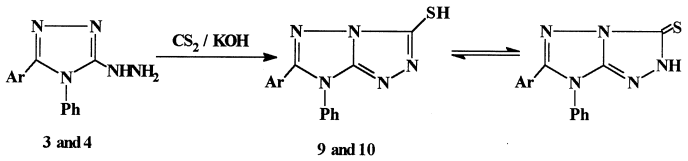
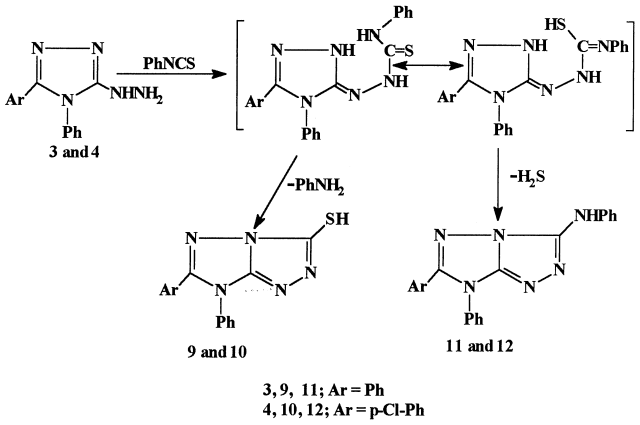




proceeds through the initial formation of the corresponding phenylthiourea intermediate and subsequent elimination of hydrogen sulphide to give **11** as a major product or elimination of aniline to give **9** as a minor product. Likewise, when compound **4** was refluxed with phenylisothiocyanate, it gave a mixture of triazolo[4,3-b]-1,2,4-triazoles **10** and **12** which was separated by the same method.



The structure of the products **9** and **10** was confirmed by the direct comparison (m.p. and mixed m.p.) with those obtained by the reaction of the corresponding hydrazino derivatives **3** and **4** with CS_2/KOH. The elemental

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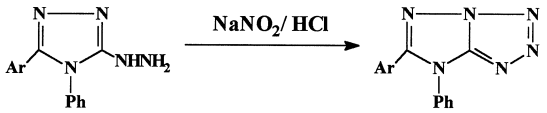
and spectral analyses of compounds **11** and **12** were in agreement with their structures (cf. Table).

Treatment of compounds **3** and **4** with nitrous acid gave 1,2,4-triazolo[1,5-d]tetrazoles **13** and **14**, respectively.

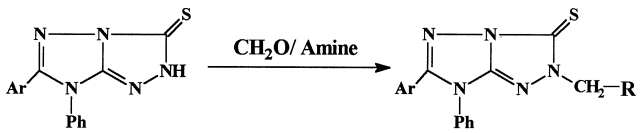


3 and 4 — 13 and 14

3 and 13; Ar = Ph
4 and 14; Ar = p-Cl-Ph

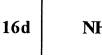
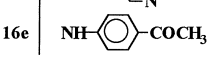
The structure of products **13** and **14** was confirmed using elemental and spectral analyses (cf Table). The mass spectrum of **13** showed the following fragmentation pattern m/e (rel. intensity %): 262 (M, 98.56), 235 (15.46), 220 (13.35), 207 (22.12), 180 (89.75), 103 (60.43), 77 (91).

When compound **9** and **10** were subjected to Mannich reaction using morpholine, diethylamine, benzylamine, 3-aminopyridine and p-aminoacetophenone, the corresponding Mannich bases **15a–e** and **16a–e** were obtained.



9 and 10 — 15a-e and 16a-e

9 and 15a-e; Ar = Ph
10 and 16a-e Ar = p-Cl-Ph

Comp. No.	R
15a, 16a	
15b, 16b	$N(C_2H_5)_2$
15c, 16c	$NHCH_2Ph$
15d, 16d	
15e, 16e	

Elemental and spectral analyses of products **15** and **16** are consistent with their structures (cf. Table). The mass spectrum of compound **15a** showed the following fragmentation pattern m/e (rel. intensity %) 392 (5.56), 293 (11.29), 180 (4.83), 100 (100), 77 (17.45), 70 (3.82).

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EXPERIMENTAL

Melting points are uncorrected and were determined on Kofler melting point apparatus. IR (cm^{-1}) spectra were obtained (KBr disc) on Nicolet 710 FT-IR Spectrophotometer. ^{1}H-NMR spectra were recorded at 60 MHz on Varian EM 360 L Spectrometer. The chemical shift are expressed in δ values (ppm) TMS was used as internal reference. The MS were recorded on a Micromass 7070 E Spectrometer operating at 70 ev, using direct inlet. Elemental analyses were carried out on an elemental analyzer 240 C.

Preparation of 3-chloro-5-(4-chlorophenyl)-4-phenyl-1, 2,4-triazole (2)

A slow stream of dry chlorine gas was passed through a suspension of 3-mercapto-5-(4-chlorophenyl)-4-phenyl-1,2,4-triazole (5.75 g, 0.02 mol) in dry chloroform (150 mL) within 15 min the whole material dissolved. Chlorine gas was passed for a further period of 40 min and the reaction mixture was washed thoroughly with dil. alkali (2% NaOH) and then with water (4 times). The chloroform layer was dried (Na_2SO_4) and evaporated to give compound **2** (cf. Table).

Preparation of 4,5-diphenyl- and 5-(4-chlorophenyl)-4-phenyl-3-hydrazino-1,2,4-triazoles (3 & 4)

General Procedure

A solution of compound **1** or **2** (0.023 mol) and hydrazine hydrate 99% (10 mL) in ethanol (10 mL) was refluxed for 5 h. On cooling the solid product was filtered off and recrystallized from the proper solvent (cf. Table).

Synthesis of 6,7-diphenyl- and 6-(4-chlorophenyl)-7-phenyl-3-aryl-1,2,4-triazolo[4,3-b]-1,2,4-triazoles (5a–e & 6a–e)

General Procedure

A mixture of **3** and/or **4** (0.004 mol) and an aromatic acid: 4-hydroxybenzoic acid, p-toluic acid, 4-chlorobenzoic acid, and/or 4-nitrobenzoic acid (0.004 mol) was refluxed in methanol (10 mL) for 9 h. Excess solvent was

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removed and the residue poured onto crushed ice. The solid thus separated was washed with 10% sodium bicarbonate solution and recrystallized from the proper solvent (cf. Table).

Synthesis of 6,7-diphenyl- and 6-(4-chlorophenyl)-7-phenyl-1,2,4-triazolo-[4,3-b]-1,2,4-triazoles (7a & 8a)

General Procedure

A mixture of **3** and/or **4** (0.002 mol) and formic acid (10 mL) was refluxed for 5 h. After cooling the reaction mixture was poured onto crushed ice. The obtained solid was filtered off then recrystallized from the proper solvent (cf. Table).

Synthesis of 6,7-diphenyl- and 6-(4-chlorophenyl)-7-phenyl-3-methyl-1,2,4-triazolo[4,3-b]-1,2,4-triazoles (7b & 8b)

General Procedure

A mixture of **3** and/or **4** (0.004 mol) and acetic acid (15 mL) was refluxed for 6 h. After cooling the reaction mixture was poured onto crushed ice. The obtained solid was filtered off then recrystallized from the proper solvent (cf. Table).

Synthesis of 6,7-diphenyl- and 6-(4-chlorophenyl)-7-phenyl-3-mercapto-1,2,4-triazolo[4,3-b]-1,2,4-triazoles (9 & 10)

General Procedure

A mixture of **3** and/or **4** (0.02 mol), KOH (0.025 mol), ethanol (95%, 60 mL) and CS_2 (0.04 mol) was refluxed for 14 h and the alkaline solution was filtered. After precipitation with dilute hydrochloric acid, the crude product was filtered off then recrystallized from the proper solvent (cf. Table).

Synthesis of 6,7-diphenyl- and 6-(4-chlorophenyl)-7-phenyl-3-phenyl-amino-1,2,4-triazolo[4,3-b]-1,2,4-triazoles (11 & 12)

General Procedure

To a solution of **3** and/or **4** (0.007 mol) in pyridine was added phenylisothiocyanate (0.007 mol) and the solution was heated under reflux for

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20 h, concentrated and poured into water, the precipitate was filtered off and recrystallized from ethanol to give a mixture of **9** and **11** or **10** and **12**. The mixture was separated by stirring for 30 min with dil. alkali (2% NaOH) and filtered. The filtrate was acidified with dilute HCl to give **9** or **10** in 29% and 24% yield, respectively and the precipitate washed with water (5 times), filtered off and recrystallized from ethanol to give **11** or **12** in 43% and 49% yield, respectively.

Synthesis of 6,7-diphenyl- and 6-(4-chlorophenyl)-7-phenyl-1, 2,4-triazole-[1,5-d]tetrazoles (13 & 14)

General Procedure

To a solution of **3** and/or **4** (0.004 mol) in conc. Hydrochloric acid (20 mL), sodium nitrite solution (30 mL, 5%) was added dropwise at 0°C for 40 minutes with stirring. The solid product was filtered off and washed with water and recrystallized from the proper solvent (cf. Table).

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