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SYNTHESIS OF NEW HETEROAROMATIC SYSTEMS: NAPHTH[2,1-*e*]IMIDAZO[5,1-*c*]-1,2,4-TRIAZINES AND BENZ[*e*]IMIDAZO-[5,1-*c*]-1,2,4-TRIAZINES

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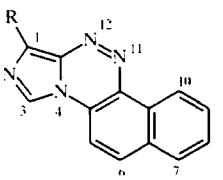
Cyclization of azo compounds, synthesized from 5-diazoimidazoles and 2-naphthol or p-substituted phenols, into naphth[2,1-e]imidazo[5,1-c]-1,2,4-triazines and benz[e]imidazo[5,1-c]-1,2,4-triazines occurs only in the presence of p-toluenesulfonic acid. Imidazo[4,5-d]-1,2,3-triazines are also formed in this reaction when an amide substituent is present in the imidazole ring.

Keywords: azo coupling, cyclization, *p*-toluenesulfonic acid, diazoimidazole, naphthimidazotriazine, benzimidazotriazine, imidazotriazine, phenols.

Diazoazoles couple with β-naphthol and *p*-substituted phenols to give the corresponding azo compounds. Some of these have been cyclized to naphthazolotriazines and benzazolotriazines [1-4]. The cyclization of analogous azo compounds obtained from derivatives of 5-diazoimidazoles has not been reported in the literature.

The objective of the present work was to synthesize new heteroaromatic systems: naphth[2,1-*e*]-imidazo[5,1-*c*]-1,2,4-triazines **1** and benz[*e*]imidazo[5,1-*c*]-1,2,4-triazines **2**, which are of interest as potential biologically active compounds.



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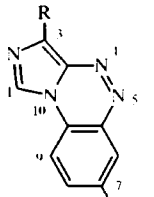

2

1 a R = COOEt, **b** R = NO_2, **c** R = $CONH_2$, **d** R = CONHMe, **e** R = CONHPh;
2 a-c R = COOEt, **a** R^2 = Me, **b** R^2 = NH_2, **c** R^2 = Cl, **d,e** R = NO_2, **d** R^2 = Me, **e** R^2 = Cl,
f,g R^2 = Me, **f** R = $CONH_2$, **g** R = CONHMe

To complete the aim the known 4-R-5-(2-hydroxynaphthylazo)imidazoles **4a,b** have been synthesized by the previously described method [5, 6]. In addition the new substituted hydroxynaphthylazoimidazoles **4b,d,e** and 4-R-5-(5-R^2-hydroxyphenylazo)imidazoles **7a-g** were prepared by the coupling of the 5-diazoimidazoles **3a-e** with β-naphthol **5** and the *p*-substituted phenols **6a-c** respectively in acetic acid.

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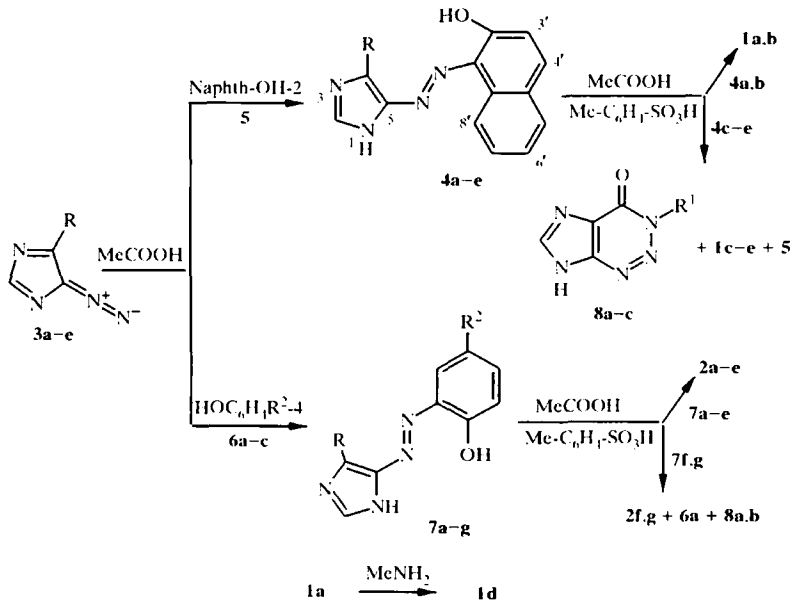
Azo derivatives of pyrazole cyclized into the corresponding naphthopyrazolotriazines on boiling in acetic acid, whereas the azo derivatives of triazoles and tetrazoles cyclized only in concentrated sulfuric acid or in its presence [4]. Under these conditions the imidazolylazo compounds **4** and **7** remained unchanged or were destroyed. Cyclization of these compounds was successfully achieved only with the addition of a catalytic amount of *p*-toluenesulfonic acid. It was found that substituents at position 4 of the imidazole ring affected the direction of the cyclization reaction. The azo compounds **4a,b** and **7a-e** containing an ester or nitro group in the imidazole moiety cyclized in adequately high yield to give only naphthimidazotriazines **1a,b** and benzimidazotriazines **2a-e** respectively. When cyclization of the azo compounds **4c-e** and **7f,g** was attempted the expected naphthimidazotriazines **1c-e** and benzimidazotriazines **2f,g** were obtained, but in addition the completely unexpected imidazo[4,5-*d*]-1,2,3-triazines **8** and β-naphthol **5** or the corresponding phenols **6** were isolated by chromatography. Imidazo[4,5-*d*]-1,2,3-triazines **8a,b,c** were identical to samples synthesized by an independent method [7, 8] (mp, R_f, IR and ^{1}H NMR spectra).



3, 4 a R = COOEt, **b** R = NO_2, **c** R = $CONH_2$, **d** R = CONHMe, **e** R = CONHPh;
6 a R^2 = Me, **b** R^2 = NH_2, **c** R^2 = Cl, **7 a-c** R = COOEt, **a** R^2 = Me, **b** R^2 = NH_2, **c** R^2 = Cl,
d,e R = NO_2, **d** R^2 = Me, **e** R^2 = Cl. **f-h** R^2 = Me, **f** R = $CONH_2$, **g** R = CONHMe;
8 a R^1 = H, **b** R^1 = Me, **c** R^1 = Ph

The formation of compounds **1** and **8** in one case and **2** and **8** in the other is the result of reversal of C-azo coupling. The yield of products **1c-e** and **2f,g** was only 14-17%, but of compounds **8** was 70-80%. Methylamide **1d** was obtained in 90% yield by treatment of naphthimidazotriazine ethyl ester **1a** with methylamine.

The structures of the synthesized naphthimidazotriazines **1**, benzimidazotriazines **2**, and the azo compounds **4b,d,e** and **7** were confirmed by IR and ^{1}H NMR spectroscopy.

It has therefore been established that, in contrast to *o*-hydroxyarylazoazoles, cyclization of azoimidazole derivatives occurs only on the addition of *p*-toluenesulfonic acid. When an amide group is present at position 4 of the imidazole ring two concurrent cyclization reactions of azo compounds **4** and **7** occur to give naphthimidazotriazines **1** and benzimidazotriazines **2** (but principally to imidazotriazines **8**). When ester or nitro groups are present cyclization of the azo compounds gives exclusively the heteroaromatic systems **1** and **2** under these conditions.

TABLE 1. Characteristics of the Compounds Synthesized

Compound	Empirical formula	Found, % / Calculated, % *			R_f (eluent)	mp, °C	Yield, %
		C	H	N			
1a	$C_{16}H_{12}N_4O_2$	66.0 / 65.75	4.3 / 4.1	20.0 / 19.2	0.54 (a)	289-291	85
1b	$C_{13}H_8N_4O_2$	58.6 / 58.9	2.6 / 2.6	26.4 / 26.4	0.44 (b)	>300	91
1c	$C_{14}H_9N_4O$	64.1 / 63.9	3.4 / 3.4	26.6 / 26.6	0.44 (a)	295-297	15
1d	$C_{15}H_{11}N_4O$	65.1 / 65.0	4.05 / 4.0	25.5 / 25.3	0.49 (a)	286-289	16
1e	$C_{20}H_{13}N_4O$	70.95 / 70.8	4.0 / 3.8	20.7 / 20.6	0.33 (b)	276-277	17
2a	$C_{13}H_{12}N_5O_2$	61.15 / 60.9	4.8 / 4.7	21.3 / 21.9	0.56 (c)	208-211	65
2b	$C_{12}H_{11}N_6O_2$	56.13 / 56.0	4.4 / 4.3	27.6 / 27.2	0.60 (d)	249-250	45
2c	$C_{12}H_9N_5ClO_2$	52.3 / 52.1	3.5 / 3.3	21.0 / 20.25	0.43 (c)	217-219	68
2d	$C_{10}H_8N_6O_2$	52.0 / 52.40	3.05 / 3.1	30.57 / 30.60	0.6 (c)	275-278	75
2e	$C_9H_7N_6ClO_2$	43.7 / 43.3	1.7 / 1.6	28.5 / 28.1	0.7 (c)	247-249	70
2f	$C_{11}H_9N_6O$	58.6 / 58.15	4.0 / 4.0	30.6 / 30.8	0.67 (a)	295-300	20
2g	$C_{12}H_{11}N_6O$	60.2 / 59.75	4.4 / 4.6	29.6 / 29.05	0.77 (a)	225-226	18
4b	$C_{13}H_8N_4O_3$	55.5 / 55.1	3.1 / 3.1	23.9 / 23.7	0.75 (b)	>300	65
4d	$C_{15}H_{13}N_4O_2$	61.45 / 61.1	4.3 / 4.4	23.2 / 23.7	0.43 (b)	276-278	84
4e	$C_{20}H_{15}N_4O_2$	67.75 / 67.2	4.1 / 4.2	20.1 / 19.6	0.24 (b)	216-217	75
7a	$C_{13}H_{14}N_6O_3$	57.1 / 56.9	4.8 / 5.1	20.5 / 20.4	0.26 (c)	189-192	80
7b	$C_{12}H_{13}N_6O_3$	52.15 / 52.4	4.4 / 4.8	25.6 / 25.4	0.34 (d)	167-169	65
7c	$C_{12}H_{11}N_5ClO_3$	48.9 / 48.9	3.5 / 3.8	19.2 / 19.0	0.43 (c)	217-219	68
7d	$C_{10}H_8N_6O_3$	48.95 / 48.6	3.65 / 3.7	28.3 / 28.3	0.24 (c)	168-170	82
7e	$C_9H_6N_6ClO_3$	40.7 / 40.4	2.2 / 2.3	26.5 / 26.2	0.30 (c)	187-190	70
7f	$C_{11}H_{11}N_6O_2$	53.6 / 53.9	4.6 / 4.5	28.9 / 28.6	0.31 (a)	164-167	63
7g	$C_{12}H_{13}N_6O_2$	55.8 / 55.6	5.2 / 5.05	27.6 / 27.0	0.27 (a)	195-196	64

* Found/Calculated, Cl, %: 13.0/12.8 (**2c**), 14.4/14.2 (**2e**), 12.6/12.0 (**7c**), 13.3/13.25 (**7e**).

EXPERIMENTAL

IR spectra of the compounds synthesized were recorded on a Specord IR-75 spectrometer (KBr discs). ^{1}H NMR spectra of DMSO-d_6 solutions with TMS as internal standard were recorded on a Brucker WR-80 spectrometer (80 MHz). The course of reactions and the purity of compounds synthesized were monitored by TLC on Silufol UV-254 and Sorbfil UV-254 strips (silica gel CTX-1A) with the following eluents: (a) chloroform–ethanol, 10:1; (b) chloroform–ethanol, 5:1; (c) ethyl acetate, (d) butanol–acetic acid–water, 4:1:1. Column chromatography was carried out on a 700 × 12 column filled with silica gel L 40/100, eluent 10:1 chloroform–ethanol. 5-Diazoimidazoles **3a-e** were synthesized by methods [9-13]. Characteristics of the compounds synthesized are given in Table 1 and spectroscopic data (^{1}H NMR and IR spectra) in Table 2.

TABLE 2. Spectral Characteristics of the Compounds Synthesized

Compound	IR spectrum, ν, cm^{-1}				^1H NMR spectrum, δ, ppm; coupling constant (J), Hz
	C=O	NO_2	C=N	NH	
1	2	3	4	5	6
1a	1725		1640		9.50 (1H, s, 3-H); 9.27 (1H, d, J = 7.8, 10-H); 8.17-7.54 (5H, m, 5-, 6-, 7-, 8-, 9-H); 4.27 (2H, q, J = 8.4, CH_2); 1.34 (3H, t, J = 8.4, CH_3)
1b		1380, 1540	1650		9.44 (1H, s, 3-H); 9.24 (1H, d, J = 7.8, 10-H); 8.24-7.62 (5H, m, 5-,6-,7-,8-,9-H)
1c	1660		1620	3340	9.42 (1H, d, J = 7.9, 10-H); 9.37 (1H, s, 3-H); 8.0 (2H, br. s, $CONH_2$); 8.64-7.62 (5H, m, 5-, 6-, 7-, 8-, 9-H)
1d	1660		1620	3350	9.43 (1H, s, 3-H); 9.38 (1H, d, J = 7.7, 10-H); 9.24 (1H, q, J = 4.9, NH); 8.40-7.78 (5H, m, 5-, 6-, 7-, 8-, 9-H); 3.87 (3H, d, J = 4.9, CH_3)
1e	1650		1620	3350	9.50 (1H, s, 3-H); 9.44 (1H, d, J = 7.9, 10-H); 8.47-7.91 (5H, m, 5-, 6-, 7-, 8-, 9-H); 7.98-7.26 (5H, m, C_6H_5)
2a	1725		1630		9.23 (1H, s, 1-H); 9.02 (1H, d, J = 7.0, 6-H); 8.17 (1H, dd, J = 2.4, 7.0, 8-H); 7.54 (1H, d, J = 2.4, 9-H); 4.34 (2H, q, J = 8.0, CH_2); 2.89 (3H, s, CH_3); 1.30 (3H, t, J = 8.0, CH_3)
2b	1725		1630	3325	9.12 (1H, d, J = 7.2, 6-H); 9.00 (1H, s, 1-H); 8.17 (1H, dd, J = 2.5, 7.2, 8-H); 7.67 (1H, d, J = 2.5, 9-H); 6.50 (2H, br. s, NH_2); 4.24 (2H, q, J = 7.8, CH_2); 1.31 (3H, t, J = 7.8, CH_3)
2c	1730		1640*		9.34 (1H, d, J = 7.0, 6-H); 9.05 (1H, s, 1-H); 8.27 (1H, dd, J = 2.6, 7.2, 8-H); 7.78 (1H, d, J = 2.5, 9-H); 4.22 (2H, q, J = 7.7, CH_2); 1.30 (3H, t, J = 7.7, CH_3)
2d		1380, 1540	1640		9.12 (1H, d, J = 7.2, 6-H); 9.00 (1H, s, 1-H); 8.17 (1H, dd, J = 2.5, 7.1, 8-H); 8.0 (1H, d, J = 2.6, 9-H); 2.74 (3H, s, CH_3)
2e		1370, 1540	1640*		9.34 (1H, d, J = 7.0, 6-H); 9.05 (1H, s, 1-H); 8.27 (1H, dd, J = 2.7, 7.0, 8-H); 7.74 (1H, d, J = 2.7, 9-H)
2f	1650		1640	3350	9.37 (1H, s, 1-H); 9.12 (1H, d, J = 7.0, 6-H); 8.24 (1H, dd, J = 2.8, 7.0, 8-H); 7.96 (2H, br. s, $CONH_2$); 7.17 (1H, d, J = 2.8, 9-H); 2.80 (3H, s, CH_3)

TABLE 2 (continued)

1	2	3	4	5	6
2g	1660		1640	3350	9.34 (1H, q, *J* = 4.9, NH.CH_3); 9.07 (1H, s, 1-H); 8.96 (1H, d, *J* = 7.3, 6-H); 8.54 (1H, dd *J* = 2.7, 7.3, 8-H); 7.67 (1H, d, *J* = 2.7, 9-H); 3.77 (3H, d, *J* = 4.9, NH$\underline{CH_3}$); 2.87 (3H, s, CH_3)
4b		1370, 1540	1630	3320*[2]	15.9 (1H, s, NH); 13.92 (1H, br. s, OH); 8.66 (1H, dd, *J* = 7.3, 8'-H); 7.88 (1H, s, 2-H); 7.94, 6.72 (2H, AB-system, 3'-, 4'-H); 7.8-7.36 (3H, m, 5'-, 6'-, 7'-H)
4d	1660		1640	3350, 3370*[2]	13.9 (1H, s, NH); 12.42 (1H, br. s, OH); 9.54 (1H, q, *J* = 4.9, NH); 8.85 (1H, dd, *J* = 7.4, 8'-H); 8.13 (1H, s, 2-H); 7.90, 6.95 (2H, AB-system, 3', 4'-H); 7.7-7.16 (3H, m, 5'-, 6'-, 7'-H); 3.93 (3H, d, *J* = 4.9, CH_3)
4e	1660		1640	3350, 3380*[2]	13.5 (1H, s, NH); 11.9 (1H, br. s, OH); 9.2 (1H, d, *J* = 7.7, 3'-H); 8.85 (1H, s, 2-H); 8.71-7.6 (5H, m, 4'-, 5'-, 6'-, 7'-, 8'-H); 7.56-7.25 (5H, m, C_6H_5)
7a	1720		1640	3340	13.56 (1H, br. s, NH); 10.90 (1H, s, OH); 8.86 (1H, s, 2-H); 9.0 (1H, d, *J* = 7.1, 3'-H); 8.47 (1H, dd, *J* = 2.4, 7.1, 4'-H); 7.14 (1H, d, *J* = 2.4, 6'-H); 4.44 (2H, q, *J* = 7.9, CH_2); 2.87 (3H, s, CH_3); 1.34 (3H, t, *J* = 8.0, CH_3)
7b	1725		1640	3325, 3350*[2]	13.46 (1H, br. s, NH); 10.67 (1H, s, OH); 9.12 (1H, d, *J* = 7.2, 3'-H); 8.70 (1H, s, 2-H); 8.17 (1H, dd, *J* = 2.4, 7.2, 4'-H); 7.67 (1H, d, *J* = 2.5, 6'-H); 6.45 (2H, br. s, NH_2); 4.28 (2H, q, *J* = 7.7, CH_2); 1.34 (3H, t, *J* = 7.7, CH_3)
7c					13.46 (1H, br. s, NH); 10.50 (1H, s, OH); 9.14 (1H, d, *J* = 7.2, 3'-H); 8.75 (1H, s, 2-H); 8.07 (1H, dd, *J* = 2.5, 7.2, 4'-H); 7.38 (1H, d, *J* = 2.5, 6'-H); 4.32 (2H, q, *J* = 7.8, CH_2); 1.30 (3H, t, *J* = 7.8, CH_3)
7d		1380, 1540	1640	3320*[2]	13.36 (1H, br. s, NH); 10.01 (1H, s, OH); 9.00 (1H, d, *J* = 7.4, 3'-H); 8.65 (1H, s, 2-H); 8.27 (1H, dd, *J* = 2.6, 7.4, 4'-H); 7.60 (1H, d, *J* = 2.6, 6'-H); 2.74 (3H, s, CH_3)
7e			1640	3320*, *[2]	14.06 (1H, br. s, NH); 10.50 (1H, s, OH); 9.04 (1H, s, *J* = 7.2, 3'-H); 8.65 (1H, s, 2-H); 8.17 (1H, dd, *J* = 2.5, 7.2, 4'-H); 7.74 (1H, d, *J* = 2.5, 6'-H)
7f	1650		1640	3320, 3350*[2]	13.93 (1H, br. s, NH); 10.50 (1H, s, OH); 9.10 (1H, d, *J* = 7.2, 3'-H); 8.67 (1H, s, 2-H); 8.44 (1H, dd, 7-2.6, 7.2, 4'-H); 7.66 (2H, br. s, $CONH_2$); 7.17 (1H, d, *J* = 2.6, 6'-H); 2.74 (3H, s, CH_3)
7g	1660		1640	3320, 3350*[2]	13.97 (1H, br. s, NH); 10.35 (1H, s, OH); 9.34 (1H, q, *J* = 4.9, NH); 8.96 (1H, d, *J* = 7.4, 3'-H); 8.67 (1H, s, 2-H); 8.44 (1H, dd, *J* = 2.7, 7.4, 4'-H); 7.70 (1H, d, *J* = 2.7, 6'-H); 3.85 (3H, d, *J* = 4.9, CH_3); 2.80 (3H, s, CH_3)

* C–Cl stretching vibrations were observed at 900 (**2d**), 900 (**2e**), 920 (**7c**), and 920 cm^{-1} (**7e**).

*[2] OH stretching vibrations were observed at 3420 (**4c,d,e**), 3450 (**7b**), 3450 (**7d**), 3450 (**7e**), 3460 (**7f**), and 3470 cm^{-1} (**7g**).

1-R-Naphth[2,1-*e*]imidazo[5,1-*c*]-1,2,4-triazines (1a,b). 4-R-5-(2-hydroxynaphthylazo)imidazole **4a,b** (3.23 mmol) and *p*-toluenesulfonic acid (0.58 mmol) were added to concentrated acetic acid (25 ml), the mixture was boiled for 12 h, cooled, the precipitate was filtered off, crystallized from dimethylformamide and washed with a small amount of ether.

1-R-Naphth[2,1-*e*]imidazo[5,1-*c*]-1,2,4-triazines (1c-e) and 3-R^1-Imidazo[4,5*d*]-1,2,3-triazin-4-ones (8a-c). Hydroxynaphthylazoimidazole **4c,e** (3.6 mmol) and *p*-toluenesulfonic acid (0.29 mmol) were added to concentrated acetic acid (15 ml), the reaction mixture was boiled for 12 h, and the precipitate containing products **1c-e** and **8a-c** was filtered off. The filtrate was evaporated to dryness in vacuum and 2-naphthol was separated by column chromatography (70% yield). Compounds **1c-e** and **8a-c** were separated by column chromatography. The fraction containing compounds **1c-e** was evaporated to dryness in vacuum and the solid residue recrystallized from dimethylformamide. The fraction containing compounds **8a-c** was evaporated to dryness in vacuum and the solid residue was recrystallized from ethanol. The known imidazo[4,5-*d*]-1,2,3-triazin-4-ones **8a-c**, obtained in 74-80% yields, were identical to samples synthesized by methods [7, 8].

1-Methylcarbamoylnaphth[2,1-*e*]imidazo[5,1-*c*]-1,2,4-triazine (1d). A solution of naphthimidazotriazine **1a** (1 g, 3.43 mmol) in 30% methylamine (30 ml) was stirred for 6 h at 40°C, cooled to 5°C, the precipitate was filtered off and crystallized from ethanol. Yield 0.85 g (90%).

3-R-Benz[*e*]imidazo[5,1-*c*]-1,2,4-triazines (2a-e). 4-R-5-(5-R^2-hydroxyphenylazo)imidazole **7a-e** (3.65 mmol) and *p*-toluenesulfonic acid (0.58 mmol) were added to concentrated acetic acid (20 ml), the reaction mixture was boiled for 8 h and evaporated to dryness in vacuum. The residue was crystallized from ethanol, the crystals were filtered off and washed with ether.

3-R-Benz[*e*]imidazo[5,1-*c*]-1,2,4-triazines (2f,g). *p*-Cresol (6a). 3-R^1-Imidazo[4,5-*d*]-1,2,3-triazin-4-ones (8a,b). Hydroxyphenylazoimidazole **7f,g** (3.65 mmol) and *p*-toluenesulfonic acid (0.58 mmol) were added to concentrated acetic acid (15 ml), the reaction mixture was boiled for 6 h and then evaporated to dryness in vacuum. The dry residue, which contained compounds **2f,g**, **8a,b**, and **6a**, was dissolved in 10:1 chloroform–ethanol mixture and separated by column chromatography (see synthesis of compounds **1c-e**). The known imidazotriazines **8a,b** obtained were identical to samples synthesized by methods [7, 8] and also to compounds obtained at isolation of compounds **1c,d** (mp, R_f, IR and ^{1}H NMR spectra). Product **6a**, obtained in 70% yield, was identical to the sample of phenol **6a** used in the coupling reactions (R_f, mp, IR spectrum).

4-R-5-(2-Hydroxynaphthylazo)imidazoles (4b,d,e). β-Naphthol (5.16 mmol) was added to a solution of 5-diazoimidazole **3b,d,e** (4.6 mmol) in acetic acid (10 ml) with vigorous stirring at a temperature not exceeding 5°C. The stirred reaction mixture was kept at this temperature for 2 h. The excess of acetic acid was evaporated in vacuum and the residue was crystallized from 50% ethanol.

4-R-5-(5-R^2-Hydroxyphenylazo)imidazoles (7a-g). Phenol **6a-c** (6.6 mmol) was added with vigorous stirring at a temperature not exceeding 5°C to a solution of 5-diazoimidazole **3a-e** (6.0 mmol) in acetic acid (10 ml). The stirred reaction mixture was kept at this temperature for 2-3 h. The excess of acetic acid was evaporated in vacuum, the dry residue was crystallized from 50% ethanol and washed with ether.

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