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5-Aryl-2-furaldehydes in the Synthesis of 2-Substituted 1,3-Benzazoles

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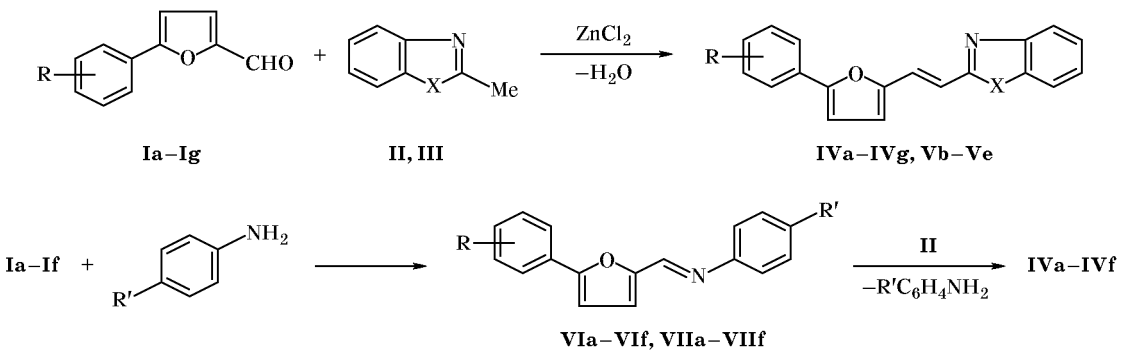
Abstract—2-[2-(5-Aryl-2-furyl)ethenyl]-1,3-benzazoles were synthesized by reaction of 5-aryl-2-furaldehydes with 2-methylbenzoxazole, 2-methylbenzothiazole, 2-methylbenzimidazole, and 2-cyanomethylbenzimidazole. The corresponding benzazoles were also obtained by reaction of *N*-(5-arylfurfurylidene)anilines with 2-methylbenzoxazoles and of 3-(5-aryl-2-furyl)-2-cyanopropenoyl chlorides with *o*-phenylenediamine. The acylation of *o*-aminophenol with 3-(5-aryl-2-furyl)-2-cyanopropenoyl chlorides occurs at the amino group without subsequent oxazole ring closure.

2-Arylethenyl-substituted benzazoles are used as organic luminophores and optical bleaching agents [1–4]. Luminescence properties of these compounds are determined by the length of the π-conjugation system. The conjugation system can be extended, e.g., via introduction of an arylfuryl group into the 2-position of benzazole ring. It is known that arylfurans also exhibit properties intrinsic to luminophores, bleaching agents, and scintillators [1–3]. Therefore, a combination of two fluorophoric fragments in a single molecule may be interesting from the practical viewpoint.

In the present work we examined the reaction of 5-aryl-2-furaldehydes **Ia**–**Ig** with 2-methylbenzoxazole, 2-methylbenzothiazole, 2-methylbenzimidazole, and 2-cyanomethylbenzimidazole. Aldehydes **Ia**–**Ig** were found to react with 2-methylbenzoxazole (**II**) and 2-methylbenzothiazole (**III**) under conditions analogous to those described in [5], namely in the presence of anhydrous zinc(II) chloride on heating to 120–180°C (depending on the substituent R) without a solvent. As a result, compounds **IVa**–**IVg** and **Vb**–**Ve** were formed (Scheme 1, Table 1).

The reaction is favored by the presence of electron-acceptor substituents R in the aromatic ring of aldehydes **Ia**–**Ig**. By contrast, in the reaction of 2-methylbenzoxazole (**II**) with 5-(4-methoxyphenyl)-2-furaldehyde we failed to isolate the corresponding condensation product, while the yield of benzoxazole **IVa** from

Scheme 1.



II, **IV**, X = O; **III**, **V**, X = S; **I**, **IV**–**VII**, R = H (**a**), 4-Cl (**b**), 4-Br (**c**), 2,5-Cl_2 (**d**), 3-NO_2 (**e**), 4-NO_2 (**f**), 2-Cl (**g**); **VI**, R′ = H; **VII**, R′ = OMe.

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Table 1. Yields, melting points, and elemental analyses of 2-substituted benzazoles **IVa**–**IVg**, **Vb**–**Ve**, and **IXa**–**IXc**

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
IVa	36	97–98	79.27	4.43	4.97	$C_{19}H_{13}NO_2$	79.42	4.56	4.88
IVb	63	176–177	70.80	3.63	4.32	$C_{19}H_{12}ClNO_2$	70.92	3.76	4.35
IVc	79	161–162	62.18	3.34	3.71	$C_{19}H_{12}BrNO_2$	62.32	3.30	3.82
IVd	64	165–166	64.15	3.04	3.69	$C_{19}H_{11}Cl_2NO_2$	64.07	3.11	3.93
IVe	54	195–196	68.41	3.52	8.50	$C_{19}H_{12}N_2O_4$	68.67	3.64	8.43
IVf	51	236–237	68.57	3.65	8.32	$C_{19}H_{12}N_2O_4$	68.67	3.64	8.43
IVg	75	130–131	70.59	3.73	4.44	$C_{19}H_{12}ClNO_2$	70.92	3.76	4.35
Vb	67	150–151	67.28	3.46	4.13	$C_{19}H_{12}ClNOS$	67.55	3.58	4.15
Vc	68	164–165	59.82	3.20	3.54	$C_{19}H_{12}BrNOS$	59.70	3.16	3.66
Vd	71	171–172	61.17	2.88	3.63	$C_{19}H_{11}Cl_2NOS$	61.30	2.98	3.76
Ve	58	176–177	65.40	3.35	8.11	$C_{19}H_{12}N_2O_3S$	65.51	3.47	8.04
IXa	36	152–153	64.05	3.41	7.69	$C_{19}H_{12}Cl_2N_2O$	64.24	3.40	7.89
IXb	44	235–237	68.58	3.87	12.75	$C_{19}H_{13}N_3O_3$	68.88	3.95	12.68
IXc	51	272–273	68.76	3.82	12.56	$C_{19}H_{13}N_3O_3$	68.88	3.95	12.68

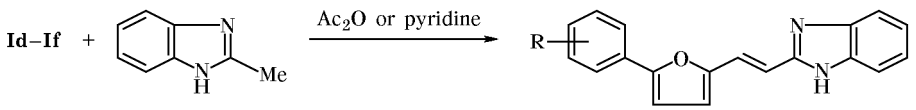
5-phenyl-2-furaldehyde (**Ia**) was poor. We also tried to synthesize 2-substituted benzazoles **IV** and **V** from Schiff bases which were prepared by reaction of aldehydes **Ia**–**If** with aniline and *p*-methoxyaniline. Compounds **VIa**–**VIf** and **VIIa**–**VIIf** thus obtained (Table 2) were brought into reaction with 2-methylbenzoxazole under the conditions reported in [6] (DMF, KOH). The products were benzoxazole derivatives **IVa**–**IVf** (Scheme 1). The reaction occurred under milder conditions (110–130°C) than the condensation of aldehydes **Ia**–**Ig** with 2-methylbenzoxazole, and in both cases (methods *a* and *b*) the yields of **IVa**–**IVf** were comparable. We also found that, from the preparative viewpoint, it is more convenient to use Schiff bases **VI** which are more reactive than their *p*-methoxy-substituted analogs **VII**. Benzazoles **IV** and **V** are bright yellow or orange substances, and most of them show fluorescence in benzene solution.

As might be expected [5], 2-methylbenzimidazole (**VIII**) turned out to be less reactive toward aldehydes **I** than azoles **II** and **III**. Compound **VIII** failed to react with aldehydes **I** under the given conditions, while under more severe conditions only tars were obtained. Condensation products **IXa**–**IXc** were isolated when the the reaction was carried out in acetic anhydride (method *c*) or pyridine (method *d*) (Scheme 2).

The yields of **IXa**–**IXc** were moderate (Table 1), and no corresponding products were isolated in the reaction with arylfuraldehydes **I** having electron-donor substituents in the aromatic ring. On the other hand, 2-cyanomethylbenzimidazole (**X**) in which the methyl group is activated by cyano group readily reacted with aldehydes **I** at room temperature to afford condensation products **XIa**–**XIf** in high yields (method *e*; Scheme 3, Table 3). Compounds **XIa**–**XIf** were also obtained by independent procedure based on the reaction of *o*-phenylenediamine with carboxylic acid chlorides to build up benzimidazole ring system [5–7] (method *f*, Scheme 3).

Initial carboxylic acid chlorides **XIIa**–**XIIf** were prepared from 3-(5-aryl-2-furyl)-2-cyanopropenoic acid esters which were in turn synthesized by condensation of 5-aryl-2-furaldehydes **I** with ethyl cyano-

Scheme 2.



VIII **IXa–IXc**

R = 2,5-Cl_2 (**a**), 3-NO_2 (**b**), 4-NO_2 (**c**).

Table 2. Yields, melting points, and elemental analyses of Schiff bases **VIa**–**VIf** and **VIIa**–**VIIf**

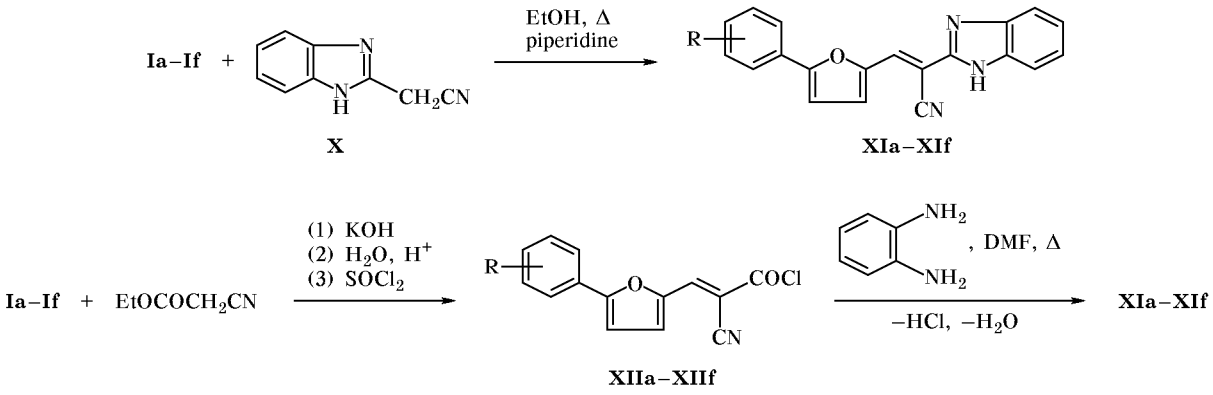
Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
VIa	60	71–72	82.43	5.22	5.74	$C_{17}H_{13}NO$	82.57	5.30	5.66
VIb	73	151–152	72.19	4.18	4.93	$C_{17}H_{12}ClNO$	72.47	4.29	4.97
VIc	91	103–104	62.29	3.70	4.35	$C_{17}H_{12}BrNO$	62.60	3.71	4.29
VId	86	93–94	64.41	3.47	4.29	$C_{17}H_{11}Cl_2NO$	64.58	3.51	4.43
VIe	65	133–134	69.58	4.09	9.37	$C_{17}H_{12}N_2O_3$	69.86	4.14	9.58
VIf	79	162–163	69.80	4.05	9.69	$C_{17}H_{12}N_2O_3$	69.86	4.14	9.58
VIIa	96	86–87	77.56	5.40	4.98	$C_{18}H_{15}NO_2$	77.96	5.45	5.05
VIIb	85	143–144	69.08	4.43	4.36	$C_{18}H_{14}ClNO_2$	69.35	4.53	4.49
VIIc	71	175–176	60.51	3.87	3.90	$C_{18}H_{14}BrNO_2$	60.69	3.96	3.93
VIId	83	89–90	62.27	3.64	4.11	$C_{18}H_{13}Cl_2NO_2$	62.45	3.78	4.05
VIIe	76	156–157	67.17	4.31	8.52	$C_{18}H_{14}N_2O_4$	67.08	4.38	8.69
VIIf	87	123–124	66.82	4.29	8.73	$C_{18}H_{14}N_2O_4$	67.08	4.38	8.69

acetate. These reactions were considered in detail previously [8].

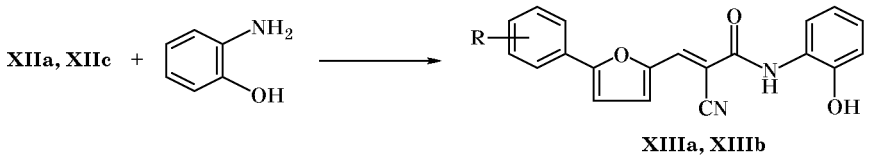
It is known that an analogous scheme is applicable to the synthesis of benzoxazoles and benzothiazoles (via reaction of carboxylic acid chlorides with *o*-aminophenol or *o*-aminobenzenethiol, respectively) [9–11]. We made an attempt to prepare 2-substituted benzoxazoles by reaction of acyl chlorides **XIIa** and **XIIc** with *o*-aminophenol; however, no oxazole ring closure occurred, and the products were the corresponding anilides **XIIIa** and **XIIIb** (Scheme 4). The yields, melting points, and analytical data of products **XIIIa** and **XIIIb** are given in Experimental, and their ^{1}H NMR spectra are presented in Table 4.

The electron absorption spectra of the condensation products (Table 5) are characterized by the presence of a strong *K*-band whose position is fairly sensitive to substituent in the aromatic ring of the arylfuryl

Scheme 3.



Scheme 4.



XIII, R = H (**a**), 4-Br (**b**).

Table 3. Yields, melting points, and elemental analyses of 2-substituted benzimidazoles **XIa**–**XIf**

Comp. no.	Yield, %	mp, °C	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
XIa	60	250–252	76.93	4.24	13.38	$C_{20}H_{13}N_3O$	77.16	4.21	13.50
XIb	73	292–294	69.17	3.42	12.20	$C_{20}H_{12}ClN_3O$	69.47	3.50	12.15
XIc	91	288–290	61.48	3.06	10.66	$C_{20}H_{12}BrN_3O$	61.56	3.10	10.77
XId	86	178–179	63.05	2.93	10.94	$C_{20}H_{11}Cl_2N_3O$	63.18	2.92	11.05
XIe	65	273–274	67.36	3.27	15.59	$C_{20}H_{12}N_4O_3$	67.41	3.39	15.72
XIf	79	324–326	67.19	3.35	15.81	$C_{20}H_{12}N_4O_3$	67.41	3.39	15.72

fragment. The absorption band shifts to longer wavelength in going from benzoxazole to benzimidazole derivatives [1]. Even stronger red shift is observed on introduction of a cyano group (compounds **XIa**–**XIf**).

Thus 5-aryl-2-furaldehydes are convenient reagents for the synthesis of 2-substituted benzazoles containing arylfuran fragments.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded from solutions in DMSO-d_6 on a Bruker AM-300 spectrometer (300 MHz); the spectra of compounds **XIb** and **XIe** were measured at 400 MHz. The signal from residual protons in the solvent (δ 2.50 ppm) was used as internal reference. The UV spectra were measured on a Specord M-40 spectrophotometer in toluene ($c = 5 \times 10^{-5}$ M, layer thickness 10.7 mm).

5-Aryl-2-furaldehydes **Ia**–**Ig** were synthesized by the procedure reported in [12], and acid chlorides **XIIa**–**XIIf** were prepared as described in [8].

***N*-(5-Arylfurfurylidene)anilines VIa–VIf and VIIa–VIIf.** A mixture of 20 mmol of 5-aryl-2-furaldehyde **Ia**–**If**, 23 mmol of aniline or *p*-anisidine, and 3.28 g of sodium acetate in 10 ml of anhydrous alcohol was heated for 6 h under reflux. The mixture was cooled, diluted with 30 ml of water, and extracted with ether. The extract was dried over $MgSO_4$ and evaporated, the residue was treated with ether, and the precipitate was filtered off and recrystallized from alcohol.

2-[2-(5-Aryl-2-furyl)ethenyl]benzoxazoles IVa–IVg. *a*. A mixture of 1 g (7.5 mmol) of freshly distilled 2-methyl-1,3-benzoxazole (**II**), 7.5 mmol of 5-aryl-2-furaldehyde **Ia**–**Ig**, and 0.5 g of $ZnCl_2$ (preliminarily dehydrated by melting) was stirred for 4 h at 150–160°C with protection from atmospheric moisture. The product was dissolved in DMF and precipitated with water. The precipitate was filtered off, washed with petroleum ether, and recrystallized from a 1:2 mixture of petroleum ether with toluene or from petroleum ether.

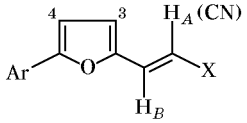
b. A mixture of 7.5 mmol of Schiff base **VIa**–**VIf** or **VIIa**–**VIIf**, 1 g (7.5 mmol) of 2-methylbenzoxazole (**II**), and 0.8 g of KOH in 15 ml of DMF was heated for 2.5–3 h at 120–140°C. The mixture was then poured into hot water, and the precipitate was treated as described above in *a*.

2-[2-(5-Aryl-2-furyl)ethenyl]benzothiazoles Vb–Ve were synthesized by the procedure described above in *a*, by heating of the reaction mixture containing the corresponding aldehyde **I** and 2-methylbenzothiazole for 0.5 h. Products **Vb**–**Ve** were recrystallized from hexane or hexane–benzene (1:1).

2-[2-(5-Aryl-2-furyl)ethenyl]benzimidazoles IXa–IXc. *c*. A solution of 10 mmol of 5-aryl-2-furaldehyde **Id**–**If** in 5–6 ml of acetic anhydride was mixed with a solution of 2 g (15 mmol) of 2-methylbenzimidazole (**VIII**) in 4 ml of acetic anhydride. Anhydrous $ZnCl_2$, 0.4 g, was added, and the mixture was heated for 3–3.5 h under reflux. It was then cooled to 90–95°C, 8 ml of 20% hydrochloric acid and 6 ml of water were added, and the mixture was refluxed for 0.5 h. The precipitate was filtered off, washed first with hot acetic acid and then with water (until neutral reaction), and dried. We thus isolated the corresponding hydrochloride of **IXa**–**IXc** which was dispersed in acetone or dioxane, and the suspension was treated with aqueous ammonia until the precipitate dissolved. The solution was diluted with water, and the precipitate was filtered off, washed with water and alcohol, and recrystallized from DMF–alcohol (1:2).

d. A solution of 5 mmol of 5-aryl-2-furaldehyde **Id**–**If** in 3–4 ml of pyridine was mixed with a solution of 1 g (7.5 mmol) of 2-methylbenzimidazole (**VIII**), 0.2 g of anhydrous $ZnCl_2$ was added, and the mixture was heated for 2.5–3 h under reflux. It was then

Table 4. ^{1}H NMR spectra (δ, ppm; *J*, Hz) of condensation products **IVb**–**IVg**, **Vb**–**Ve**, **XIb**, **XIe**, **XIIIa**, and **XIIIb**



Comp. no.	3-H, d (1H)	4-H, d (1H)	H_A (1H)	H_B, X	H_{arom}
IVb	6.96 (3.3)	7.07	7.04 d (16.2)	7.32–7.39 m (2H, 5-H, 6-H), 7.57–7.70 m (3H, H_B, 4-H, 7-H)	7.46 d (2H), 7.84 d (2H), *J* = 8.4
IVc	6.95 (3.0)	7.07	7.03 d (16.4)	7.32–7.39 m (2H, 5-H, 6-H), 7.60–7.70 m (3H, H_B, 4-H, 7-H)	7.59 d (2H), 7.78 d (2H), *J* = 7.5
IVd	7.02 (3.0)	7.34[a]	7.15 d (16.2)	7.63 d (1H, H_B), 7.59 d (1H) and 7.68 d (1H) (4-H, 7-H); 7.33–7.40 m (4H, 4-H, 5-H, 6-H, 3-H_{arom}), 7.53 m (1H, 4-H_{arom}), 8.03 m (1H, 6-H_{arom})	
IVe	7.00	7.33[a]	7.10 d (16.5)	7.27–7.42 m (3H, 4-H, 5-H, 6-H), 7.55–7.79 m (4H, H_B, 4-H, 7-H, 5-H_{arom}) 8.13 d (1H, 6-H_{arom}), *J* = 7.5; 8.24 d (1H, 4-H_{arom}), *J* = 6.9; 8.58 s (1H, 1-H_{arom})	
IVf	7.04 (2.4)	7.36[a]	7.16 d (16.2)	7.32–7.42 m (3H, 4-H, 5-H, 6-H), 7.58–7.72 m (3H, H_B, 4-H, 7-H)	8.10 d (2H), 8.28 d (2H), *J* = 9.0
IVg	7.00 (3.0)	7.26	7.06 d (16.2)	7.32–7.41 m (3H, 5-H, 6-H, 5-H_{arom}), 7.58–7.71 m (3H, H_B, 4-H, 7-H) 7.45 t (1H, 4-H_{arom}), 7.52 d (1H, 3-H_{arom}), *J* = 8.4; 8.04 d (1H, 6-H_{arom}), *J* = 7.2	
Vb	6.90 (4.2)	7.04	7.32–7.50 m (6H, H_A, H_B, 5-H, 6-H, 3-H_{arom}, 5-H_{arom}), 7.91 d (1H, 4-H), *J* = 7.2; 7.97 d (1H, 7-H), *J* = 8.4, 7.83 d (2H, 2-H_{arom}, 6-H_{arom}), *J* = 7.8		
Vc	6.90 (3.0)	7.06	7.33–7.50 m (4H, H_A, H_B, 5-H, 6-H), 7.92 d (1H, 4-H), *J* = 8.1; 7.97 d (1H, 7-H), *J* = 7.8		7.59 d (2H), 7.77 d (2H), *J* = 8.7
Vd	6.97 (3.3)	7.33	7.35–7.57 m (6H, H_A, H_B, 5-H, 6-H, 3-H_{arom}, 4-H_{arom}), 7.93 d (1H, 4-H), *J* = 7.8; 7.98 d (1H, 7-H), *J* = 7.8; 8.03 m (1H, 6-H_{arom})		
Ve	6.97 (3.9)	7.31	7.36–7.55 m (4H, H_A, H_B, 5-H, 7-H), 7.93 d (1H, 4-H), *J* = 7.8; 7.99 d (1H, 7-H), *J* = 7.2		7.74 t (1H, 5-H), *J* = 7.8; 8.15 d (1H, 6-H), 8.26 d (1H, 4-H), 8.60 s (1H, 2-H)
XIb	7.15 (3.0)	7.24	8.13 s	7.11 m (2H, 5-H, 6-H), 7.44 m (3H, 4-H, 3-H_{arom}, 5-H_{arom}), 7.56 d (1H, 7-H), 7.85 d (2H, 1-H_{arom}, 6-H_{arom}), *J* = 8.4; 12.80 br.s (1H, NH)	
XIe	7.31 (3.6)	7.39	8.18 s	7.10–7.17 m (2H, 5-H, 6-H), 7.45 m (1H, 4-H), 7.56 m (1H, 7-H), 12.85 br.s (1H, NH)	7.72 t (1H, 5-H), 8.15 m (1H, 6-H), 8.26 d (1H, 4-H), *J* = 7.2; 8.68 s (1H, 2-H)
XIIIa	7.23 (2.7)	7.45[a]	8.13 s	6.81 t (1H, 5-H), 6.93 m (2H, 3-H, 4-H), 8.09 d (1H, 6-H), *J* = 6.9; 8.97 br.s (1H, OH), 10.06 br.s (1H, NH)	7.37–7.55 m (4H, 4-H, 3-H, 4-H, 5-H), 7.91 d (2H, 2-H, 6-H), *J* = 8.1
XIIIb	7.31 (2.7)	7.47	8.14 s	6.80 t (1H, 5-H), 6.88–7.01 m (2H, 3-H, 4-H), 8.07 d (1H, 6-H), *J* = 8.4; 8.96 br.s (1H, OH), 10.06 br.s (1H, NH)	7.66 d (2H), 7.85 d (2H), *J* = 8.1

[a] Overlapped by other proton signals.

Table 5. Electron absorption spectra of compounds **IVa**–**IVf**, **IXa**–**IXc**, and **XIa**–**XIf**

Compound no.	λ_{max}, nm	log ε	Compound no.	λ_{max}, nm	log ε
IVa	367	4.80	**IXc**	413	4.77
IVb	398	4.87	**XIa**	410	4.88
IVc	405	4.76	**XIb**	446	4.93
IVd	389	4.83	**XIc**	450	4.81
IVe	377	4.73	**XId**	433	4.88
IVf	414	4.69	**XIe**	440	4.79
IXa	392	4.84	**XIf**	431	4.77
IXb	386	4.79			

cooled to 80–85°C, 8 ml of hydrochloric acid and 6 ml of water were added, and the mixture was heated for 0.5–1 h at the boiling point and was left to stand until a solid precipitated. The precipitate was treated as described above in *c*.

3-(5-Aryl-2-furyl)-2-(2-Benzimidazolyl)acrylonitriles XIa–XIf. *e*. Piperidine, 1 ml, was added to a mixture of 1.57 g (10 mmol) of compound **X** and 10 mmol of aldehyde **Ia**–**If** in 15 ml of alcohol, and the mixture was heated for 0.5–1 h under reflux. It was then left to stand until a solid precipitated. The product was filtered off, washed with ether, and recrystallized from DMF–alcohol.

f. A mixture of 0.68 mmol of acyl chloride **XIIa**–**XIIf** and 0.19 g (1.76 mmol) of *o*-phenylenediamine in 5 ml of DMF was heated for 1 h under reflux. The mixture was cooled and diluted with 6 ml of water, and the precipitate was filtered off, washed in succession with water, alcohol, and ether, and recrystallized from appropriate solvent.

***N*-(2-Hydroxyphenyl)-3-(5-aryl-2-furyl)-2-cyanopropenamides XIIIa and XIIIb.** A mixture of 0.8 g (3.1 mmol) of compound **XIIa**, 0.34 g (3.1 mmol) of *o*-aminophenol, and 45 ml of triethylamine was stirred for 1 h on heating to the boiling point. The mixture was then cooled and acidified with dilute hydrochloric acid to pH 1. The precipitate was filtered off, washed with water and alcohol and recrystallized from alcohol–DMF (4:1). Yield of **XIIIa** 0.65 g (65%), mp 264–265°C. Found, %: C 72.45; H 4.16; N 8.40. $C_{20}H_{14}N_2O_3$. Calculated, %: C 72.72; H 4.27; N 8.48. Compound **XIIIb** was synthesized in a similar way. Yield 61%, mp 315–316°C. Found, %: C 58.52; H 3.13; N 6.91. $C_{20}H_{13}BrN_2O_3$. Calculated, %: C 58.70; H 3.20; N 6.85.

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