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Cyanoacetylene and Its Derivatives: XXVI.* Hydrohalogenation of 4-Alkyl-4-hydroxy-2-alkynenitriles**

A. G. Mal'kina, V. V. Nosyreva, R. N. Kudyakova, L. V. Sokolyanskaya, and B. A. Trofimov

Favorskii Irkutsk Institute of Chemistry, Siberian Division, Russian Academy of Sciences, ul. Favorskogo 1, Irkutsk, 664033 Russia

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Abstract—The reaction of 4-alkyl-4-hydroxy-2-alkynenitriles with hydrogen halides (HCl and HI) in dioxane is accompanied by intramolecular cyclization with formation of 5,5-dialkyl-4-halo-2-imino-2,5-dihydrofuran hydrohalides. Treatment of the latter with K_2CO_3 in ethanol yields 5,5-dialkyl-4-halo-2-imino-2,5-dihydrofurans.

The reactivity of nitriles derived from γ-hydroxy-α,β-acetylenic acids toward O-, N-, and S-nucleophiles was reviewed in [2]. General synthetic approaches to previously unknown polyfunctional heterocyclic compounds were developed, and a basis was created for a new promising field of fine organic synthesis oriented toward biologically active substances. However, available information on the reactions of γ-hydroxy-α,β-acetylenic nitriles with hydrogen halides is very poor. Skvortsov *et al.* [3, 4] reported on the addition of HBr to γ-hydroxy-α,β-acetylenic nitriles, which was accompanied by intramolecular cyclization to afford bromoiminodihydrofuran hydrobromides.

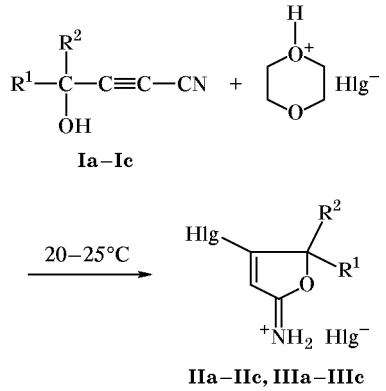
In the present work we continued studies on the reactivity of γ-hydroxy-α,β-acetylenic nitriles toward hydrogen halides with the goal of elucidating specific features of this process and obtaining haloiminodihydrofurans as potential biologically active compounds. The 2-oxo-2,5-dihydrofuran fragment related to the 2-imino-2,5-dihydrofuran structure constitutes a part of a number of naturally occurring molecules, in particular of ascorbic, penicillic, and tetronic acids.

We have found that dioxanium halides generated by passing gaseous hydrogen chloride (or hydrogen iodide) through anhydrous dioxane very readily add to 4-alkyl-4-hydroxy-2-alkynenitriles **Ia**–**Ic**, yielding 82–100% of the corresponding 5,5-dialkyl-4-halo-2-imino-2,5-dihydrofuran hydrohalides **IIa**–**IIc** and **IIIa**–**IIIc** (Scheme 1; Tables 1, 2).

Presumably, in the first stage halide ion adds at the triple bond of alkyne **Ia**–**Ic** with subsequent fast (or simultaneous) protonation to give intermediate adducts **IV** and **V**. The intramolecular ring closure is likely to be favored by electrophilic assistance by dioxanium ion (Scheme 2). This scheme is consistent with the fact that the most nucleophilic iodide ion (which does not require such assistance) gives up to 90% of linear primary adducts **V**.

The reaction of alkynes **Ia**–**Ic** with HCl is complete in 12 h (dioxane, 20°C); the same reaction with HI

Scheme 1.



I, $R^1 = R^2$ = Me (**a**); R^1 = Me, R^2 = Et (**b**); $R^1R^2 = (CH_2)_5$ (**c**); **II**, Hlg = Cl, $R^1 = R^2$ = Me (**a**); R^1 = Me, R^2 = Et (**b**); $R^1R^2 = (CH_2)_5$ (**c**); **III**, Hlg = I, $R^1 = R^2$ = Me (**a**); R^1 = Me, R^2 = Et (**b**); $R^1R^2 = (CH_2)_5$ (**c**).

* For communication XXV, see [1].

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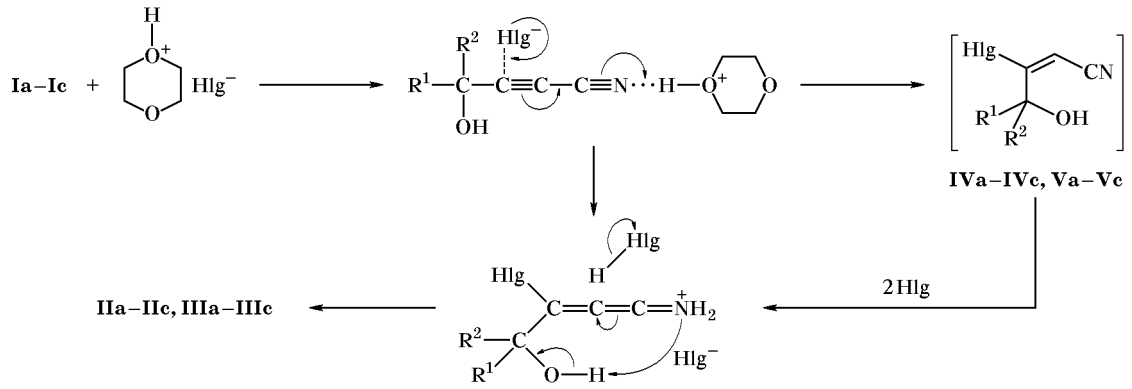
Scheme 2.



I, $R^1 = R^2$ = Me (**a**); R^1 = Me, R^2 = Et (**b**); $R^1R^2 = (CH_2)_5$ (**c**); **II**, **IV**, **VI**, Hlg = Cl; $R^1 = R^2$ = Me (**a**); R^1 = Me, R^2 = Et (**b**); $R^1R^2 = (CH_2)_5$ (**c**); **III**, **V**, **VII**, Hlg = I; $R^1 = R^2$ = Me (**a**); R^1 = Me, R^2 = Et (**b**); $R^1R^2 = (CH_2)_5$ (**c**).

takes 1 h, whereas compounds **Ia**–**Ic** failed to react with both gaseous HF and its 40% aqueous solution (dioxane, 20–25°C, 25 h). The observed reactivity series of hydrogen halides coincides with the nucleophilicity series of the corresponding anions $I^- > Cl^- \geq F^-$. This fact indicate that the driving force of the process is nucleophilicity of the anion.

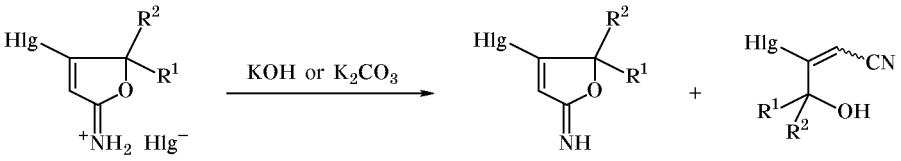
An analogous pattern was observed previously [5–14] while studying the kinetics of nucleophilic addition of HCl, HBr, and HI to 2-propynoic acid, acetylenedicarboxylic acid, and dimethyl acetylenedicarboxylate in aqueous [5] and alcoholic [6, 7] solutions, chloroform [8], acetonitrile and nitromethane [9], dimethylformamide [10, 11], toluene and hexane [12], and chlorobenzene [12, 13]. It was shown that the rate of hydrogen halide addition increases in the series HCl < HBr ≤ HI [5, 10].

Haloiminodihydrofurans hydrohalides **II** and **III** were also obtained from alkynes **I** using aqueous solutions of hydrogen halides. For example, the reaction of alkyne **Ia** with 33% hydrochloric acid under reflux (3 h) gave 44% of 4-chloro-2-imino-5,5-dimethyl-2,5-dihydrofuran hydrochloride (**IIa**). By reaction of **Ia** with iodide ion generated *in situ* from KI in the presence of acetic acid (20–60°C, 2–15 h; cf. [10]) we obtained a mixture containing, according to the IR and ^{1}H NMR spectra, initial alkyne **Ia** and 4-hydroxy-3-iodo-4-methyl-2-pentenenitrile (**Va**) (Table 3).

Our numerous attempts to direct the process toward quantitative formation of 4-hydroxy-3-iodo-4-methyl-2-pentenenitrile (**Va**) by varying the reaction conditions were unsuccessful. In all cases the reaction mixture contained unchanged alkyne **Ia**, though its conversion ranged from 52 to 81% (Table 3). In the presence of dibenzo-18-crown-6 (10 mol % relative to KI) up to 5% of iminodihydrofuran hydroiodide **IIIa** was formed in addition to product **Va**.

Iminodihydrofuran hydrochlorides **IIa**–**IIc** are colorless crystalline substances, soluble in acetone, alcohol, and chloroform; they can be stored in a closed glass vessel without appreciable decomposition. Iminodihydrofuran hydroiodides **IIIa**–**IIIc** are crystalline substances, soluble in acetone, alcohol, DMSO, and chloroform. They turn yellow on storage at room temperature, and the iodine content decreases, presumably due to elimination of HI. The IR and ^{1}H NMR spectra of compounds **IIa**–**IIc** and **IIIa**–**IIIc** are given in Table 2.

Scheme 3.



II, **IV**, **VI**, Hlg = Cl; $R^1 = R^2$ = Me (**a**); R^1 = Me, R^2 = Et (**b**); **III**, **VII**, Hlg = I; $R^1 = R^2$ = Me (**a**); R^1 = Me, R^2 = Et (**b**); $R^1R^2 = (CH_2)_5$ (**c**).

Table 1. Yields, melting points, and elemental analyses of 5,5-dialkyl-4-halo-2-imino-2,5-dihydrofuran hydrohalides **IIa**–**IIc** and **IIIa**–**IIIc** and 5,5-dialkyl-2-imino-4-iodo-2,5-dihydrofurans **VIIa** and **VIIb**

Comp. no.	Yield, %	mp, °C	Found, %				Formula	Calculated, %			
			C	H	Hlg	N		C	H	Hlg	N
IIa	100	180–182	39.49	5.01	38.71	7.31	$C_6H_9Cl_2NO$	39.58	4.98	38.95	7.69
IIb	82	129–130	43.16	5.66	35.65	6.93	$C_7H_{11}Cl_2NO$	42.88	5.65	36.16	7.14
IIc	91	156–158	48.73	5.51	31.59	6.22	$C_9H_{13}Cl_2NO$	48.66	5.91	31.92	6.31
IIIa	98	188–190	19.75	2.34	69.54	3.81	$C_6H_9I_2NO$	19.75	2.49	69.55	3.84
IIIb	82	148–150	22.75	3.42	66.04	3.43	$C_7H_{11}I_2NO$	22.19	2.93	66.97	3.70
IIIc	100	170–175	27.34	4.43	62.08	3.04	$C_9H_{13}I_2NO$	26.69	3.24	62.67	3.45
VIIa	78	68–72	31.84	3.60	53.58	5.10	C_6H_8INO	30.40	3.40	53.54	5.91
VIIb	100	60	34.69	3.83	50.28	5.32	$C_7H_{10}INO$	33.49	4.01	50.55	5.58

Table 2. IR and ^{1}H NMR spectra of 5,5-dialkyl-4-halo-2-imino-2,5-dihydrofuran hydrohalides **IIa**–**IIc** and **IIIa**–**IIIc** and 5,5-dialkyl-2-imino-4-iodo-2,5-dihydrofurans **VIIa**–**VIIc**

Comp. no.	^{1}H NMR spectrum, δ, ppm		IR spectrum (KBr), ν, cm^{-1}
	Alk	=CH (s, 1H)	
IIa	1.69 s (6H, $2CH_3$)	7.11	2700–3050, 1650–1600, 1540, 1520, 1460–1405, 1370, 1295, 1195, 1140, 1100, 1020, 970, 860, 850, 830, 605, 520
IIb	2.08 q (2H, CH_2), 1.66 s (3H, CH_3), 0.83 t (3H, CH_3)	7.15	2700–2950, 1650, 1590, 1540, 1500, 1450, 1410, 1380, 1330, 1240, 1185, 1160, 1140, 1100, 1050, 1010, 1000, 970, 860, 830, 805, 700, 600, 510
IIc	1.81 br.s (10H, $5CH_2$)	7.09	2700–3050, 1670, 1590, 1440, 1420, 1360, 1270, 1260, 1240, 1200, 1140, 1100, 1070, 970, 940, 890, 860, 840, 600, 570, 560, 500
IIIa	1.69 s (3H, $2CH_3$)	6.99	2770–3210, 1670, 1570, 1530, 1410, 1370, 1300, 1270, 1260, 1170, 1100, 1040, 960, 940, 910, 850, 810, 790, 700, 690, 660, 580, 560, 470
IIIb	2.08 q (2H, CH_2), 1.62 s (3H, CH_3), 0.76 m (3H, CH_3)	7.03	2850–3200, 1670, 1570, 1420, 1390, 1350, 1320, 1270, 1230, 1160, 1120, 1080, 1050, 1010, 950, 930, 870, 700, 650, 590, 570
IIIc	1.77 m (10H, $5CH_2$)	7.02	2900–3230, 1670, 1570, 1550, 1460, 1450, 1410, 1380, 1200, 1150, 1130, 1105, 1060, 970, 960, 940, 800, 860, 810, 740, 705, 670, 580
VIIa	1.46 s (6H, $2CH_3$)	6.54	3360, 3340, 2970, 2910, 1655, 1570, 1540, 1450, 1400, 1370, 1360, 1350, 1330, 1270, 1195, 1130, 1080, 970, 940, 910, 860, 850, 840, 760, 700, 670, 570
VIIb	1.81 q (2H, CH_2), 1.42 s (3H, CH_3), 0.76 m (3H, CH_3)	6.55	3310, 3200, 2970, 2920, 1660, 1590, 1460, 1450, 1370, 1350, 1340, 1290, 1200, 1160, 1130, 1100, 1080, 1070, 940, 900, 870, 860, 700, 600
VIIc	1.77 br.d (10H, $5CH_2$)	6.56	3470–3360, 3280–3180, 2920, 2850, 1650, 1570, 1540, 1450, 1360, 1330, 1280, 1200, 1140, 1120, 1085, 970, 950, 930, 800, 860, 840, 810, 730, 600, 550

Table 3. Reaction of alkyne **Ia** with iodide ion: conditions and yields of 2-imino-4-iodo-5,5-dimethyl-2,5-dihydrofuran (**IIIa**) and 4-hydroxy-3-iodo-4-methyl-2-pentenenitrile (**Va**)[a]

Run no.	H_2O, ml	Temperature, °C	Time, h	Yield,[b] %		Conversion, %
				IIIa	**Va**	
1[c]	0.4	20	8			
		60	2	–	90	52
2[c]	–	55–60	15	–	80	63
3[d]	0.4	20	5			
		60	3	–	39	59
4[c,e]	0.4	55–60	10	5	86	71
5[c,e]	–	55–60	15	1	40	81

[a] In all experiments 98% CH_3COOH was used.
[b] According to the ^1H NMR data.
[c] Ratio **Ia**:KI = 1:2.
[d] Ratio **Ia**:KI = 1:1.
[e] In the presence of dibenzo-18-crown-6.

We showed in [1, 14] that 5,5-dialkyl-4-dialkylamino(or alkylthio)-2-imino-2,5-dihydrofuran hydrochlorides can readily be converted into the corresponding free bases by treatment with potassium hydroxide (ethanol, 20–25°C, 3–5 h). However, neutralization of iminodihydrofuran hydrochlorides **II** was not selective. By the action of KOH or K_2CO_3 in ethanol at room temperature hydrochlorides **IIa** and **IIb** underwent partial ring opening. As a result, we obtained mixtures consisting of 3-chloro-4-hydroxy-4-methyl-2-alkenenitriles **IVa** and **IVb** and 4-chloro-2-iminodihydrofurans **VIa** and **VIb** (Scheme 3).

In the IR spectra of the products (film) we observed absorption bands at 3320–3300 and 1680 cm^{-1}, which belong to vibrations of the =N–H group. A medium-intensity band in the region 2200–2205 cm^{-1} belongs to the cyano group of 3-chloro-2-alkenenitriles **IVa** and **IVb**. The double bond gives rise to absorption at 3100 and 1630 cm^{-1} (compounds **VIa** and **VIb**) and 3100 and 1610 cm^{-1} (**IVa** and **IVb**). The ^1H NMR spectrum ($CDCl_3$) contained two signals from olefinic protons at δ 6.22–6.29 and 6.06–6.13 ppm with an intensity ratio of about 1:3. These signals correspond to 3-chloro-4-hydroxy-4-methyl-2-alkenenitriles **IVa** and **IVb** and 4-chloro-2-iminodihydrofurans **VIa** and **VIb**, respectively. Protons of the methyl group appear as a singlet at δ 1.45–1.47 ppm.

Treatment of hydroiodides **IIIa**–**IIIc** with K_2CO_3 in ethanol at room temperature (10–12 h) leads to formation of 78–100% of 5,5-dialkyl-2-imino-4-iodo-2,5-dihydrofurans **VIIa**–**VIIc** (Scheme 3). Their yields, melting points, elemental analyses, and spectral parameters are given in Tables 1 and 2.

EXPERIMENTAL

The ^1H NMR spectra were obtained on a Jeol spectrometer (90 MHz) in $CDCl_3$ and CD_3OD using HMDS as internal reference. The IR spectra were recorded on a Specord 75IR instrument from samples prepared as thin films or KBr pellets. The progress of reactions was monitored by TLC on Al_2O_3 using chloroform–benzene–ethanol (20:4:1) as eluent.

Initial alkynes **Ia**–**Ic** were synthesized by the procedure reported in [15]. Hydrogen halides (HF, HCl, and HI) were prepared as described in [16].

5,5-Dialkyl-4-chloro-4-imino-2,5-dihydrofuran hydrochlorides IIa–**IIc.** *a.* Gaseous HCl was passed over a period of 12 h through a solution of 2.5 mmol of alkyne **Ia**–**Ic** in 5 ml of anhydrous dioxane, stirred at 20–25°C. The solvent was removed under reduced pressure, and the crystals were washed with dry diethyl ether.

b. A mixture of 0.27 g (2.5 mmol) of alkyne **Ia** and 10 ml of 33% hydrochloric acid was heated for 3 h on a boiling water bath. The solvent was removed under reduced pressure, and the crystals were washed with diethyl ether to obtain 0.2 g of hydrochloride **IIa**.

Neutralization of 4-chloro-2-imino-5,5-dimethyl-2,5-dihydrofuran hydrochloride (IIa). A solution of 0.45 g (2.5 mmol) of hydrochloride **IIa** in 3 ml of ethanol was slowly added to a suspension of 0.52 g (3.8 mmol) of K_2CO_3 or 0.21 g (3.8 mmol) of KOH in 2 ml of ethanol. The mixture was kept for 3 h at 20–22°C and was passed through a column charged with Al_2O_3 (eluent chloroform–benzene–alcohol,

20:4:1). The solvent was removed under reduced pressure to obtain 0.2 g (55%) of a yellow oily liquid containing (according to the ^{1}H NMR data), 0.15 g (41%) of iminodihydrofuran **VIa** and 0.05 g (14%) of 3-chloro-4-hydroxy-4-methyl-2-pentenenitrile (**IVa**). IR spectrum, ν, cm^{-1}: 3300–3200 (OH, NH), 3110 (=C–H); 2990, 2940 (CH); 2205 (C≡N); 1680 (δC=N); 1630, 1610 (C=C); 1580, 1470, 1390, 1370, 1330, 1300 (δC–N); 1200, 1150 (C–O–C); 1120 w, 1010 (δC–H); 980, 960 w, 930 (δ=C–H); 870, 830, 730, 690 (δC–H); 600, 530 (C–Cl). ^{1}H NMR spectrum, δ, ppm: 6.06 s (1H, =CH) (**VIa**), 6.22 s (1H, =CH) (**IVa**), 1.47 s (12H, $4CH_3$).

Following a similar procedure, from 0.49 g (2.5 mmol) of hydrochloride **IIb** and 0.52 g (3.8 mmol) of K_2CO_3 in 5 ml of ethanol we obtained 0.23 g (58%) of a yellow oily liquid containing (according to the ^{1}H NMR data), 0.16 g (40%) of iminodihydrofuran **VIb** and 0.07 g (18%) of 3-chloro-4-hydroxy-4-methyl-2-hexenenitrile (**IVb**). IR spectrum, ν, cm^{-1}: 3300–3200 (OH, NH); 3110 (=CH); 2970, 2940, 2870 (CH); 2200 (C≡N); 1680 (δC=N); 1630, 1610 (C=C); 1560, 1460, 1430 w, 1370, 1330, 1300 (δC–H); 1250, 1180 w, 1140 (C–O–C); 1070, 1060 (δC–H); 1000, 960, 930, 900 w (δ=C–H); 880, 820, 705, 670 (δC–H); 610, 530 (C–Cl). ^{1}H NMR spectrum, δ, ppm: 6.13 s (1H, =CH) (**VIb**), 6.29 s (1H, =CH) (**IVb**), 1.80 q (4H, $2CH_2$), 1.45 s (6H, $2CH_3$), 0.82 t (6H, $2CH_3$).

5,5-Dialkyl-2-imino-4-iodo-2,5-dihydrofuran hydroiodides IIIa–IIIc. Gaseous HI was passed over a period of 1 h through a stirred solution of 2.5 mmol of alkyne **Ia–Ic** in 10 ml of dioxane. The mixture was filtered, and the precipitate was washed with dry diethyl ether.

5,5-Dialkyl-2-imino-4-iodo-2,5-dihydrofurans VIIa–VIIc. A mixture of 0.5 mmol of compound **IIIa–IIIc** and 0.7 mmol of K_2CO_3 in 3 ml of ethanol was kept for 12 h at 20–25°C. The mixture was then passed through a 5-cm layer of Al_2O_3 using chloroform–benzene–alcohol (20:4:1) as eluent. The solvent was removed under reduced pressure, and the crystals were washed with diethyl ether.

Reaction of 4-hydroxy-4-methyl-2-pentynenitrile with the system KI–acetic acid. *a.* A solution of 0.27 g (2.5 mmol) of alkyne **Ia** in 5 ml of dioxane was slowly added to a mixture of 0.86 g (5.2 mmol) of KI, 1.50 g (25 mmol) of acetic acid, 0.4 ml of water, and 5 ml of dioxane. The mixture was stirred for 8 h at 20–25°C and for 2 h at 55–60°C. It was then passed through a 5-cm layer of Al_2O_3 using diethyl ether as eluent. The solvent was removed under reduced pressure to obtain 0.42 g of a mixture containing (according to the ^{1}H NMR data), 0.12 g of unreacted alkyne **Ia** and 0.30 g of 4-hydroxy-3-iodo-4-methyl-2-pentenenitrile (**Va**). IR spectrum, ν, cm^{-1}: 3420–3480 (OH); 3040 (=C–H); 2970, 2930 (C–H); 2290, 2260 (C≡C, C≡N); 2220 (C≡N); 1590 (C=C); 1450, 1360, 1260 w, 1230 w (δC–H); 1170, 1120, 1030 (δC–H); 960 (δ=C–H); 805, 580, 560 (δC–H); 495 (C–I). ^{1}H NMR spectrum, δ, ppm: 6.73 s (1H, =CH), 1.50 s (6H, $2CH_3$) (**Va**), 1.56 s (6H, $2CH_3$) (**Ia**).

b. A solution of 0.27 g (2.5 mmol) of alkyne **Ia** in 5 ml of dioxane was slowly added to a mixture of 0.86 g (5.2 mmol) of KI, 1.50 g (25 mmol) of acetic acid, and 5 ml of dioxane. The mixture was stirred for 15 h at 55–60°C and was treated as described above in *a* to isolate 0.42 g of a mixture containing 0.10 g of unreacted alkyne **Ia** and 0.32 g of compound **Va**.

c. A solution of 0.27 g (2.5 mmol) of alkyne **Ia** in 5 ml of dioxane was slowly added to a mixture of 0.43 g (2.6 mmol) of KI, 1.50 g (25 mmol) of acetic acid, 0.4 ml of water, and 5 ml of dioxane. The mixture was stirred for 5 h at room temperature and for 3 h at 55–60°C. It was then treated as described above to isolate 0.25 g of a mixture containing 0.11 g of unreacted alkyne **Ia** and 0.14 g of compound **Va**.

d. A solution of 0.27 g (2.5 mmol) of alkyne **Ia** in 5 ml of dioxane was slowly added to a mixture of 0.86 g (5.2 mmol) of KI, 0.19 g (0.5 mmol) of dibenzo-18-crown-6, 1.50 g (25 mmol) of acetic acid, 0.4 ml of water, and 5 ml of dioxane. The mixture was stirred for 10 h at 55–60°C and was treated as described above to isolate 0.48 g of a mixture containing 0.08 g of alkyne **Ia**, 0.37 g of compound **Va**, and 0.03 g of compound **IIIa**.

e. A solution of 0.27 g (2.5 mmol) of alkyne **Ia** in 5 ml of dioxane was slowly added to a mixture of 0.86 g (5.2 mmol) of KI, 0.19 g (0.5 mmol) of dibenzo-18-crown-6, 1.50 g (25 mmol) of acetic acid, and 5 ml of dioxane. The mixture was stirred for 15 h at 55–60°C and was treated as described above to isolate 0.25 g of a mixture containing 0.05 g of alkyne **Ia**, 0.19 g of nitrile **Va**, and 0.01 g of iminodihydrofuran hydroiodide **IIIa**.

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