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Successive Reaction of 2-Aryl-4-dichloromethylideneoxazol-54*H*-ones with 2-Amino-1,3-thiazoles and Strongly Basic Nitrogen-containing Reagents

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Abstract—Treatment of 2-aryl-4-dichloromethylideneoxazole-5(4*H*)-ones first with 2-aminothiazoles and then with benzylamine, piperidine, or morpholine gave the corresponding 5-amino-4-(1,3-thiazol-2-ylcarbamoyl)-1,3-oxazole derivatives whose structure was confirmed by a combination of spectral data and X-ray analysis.

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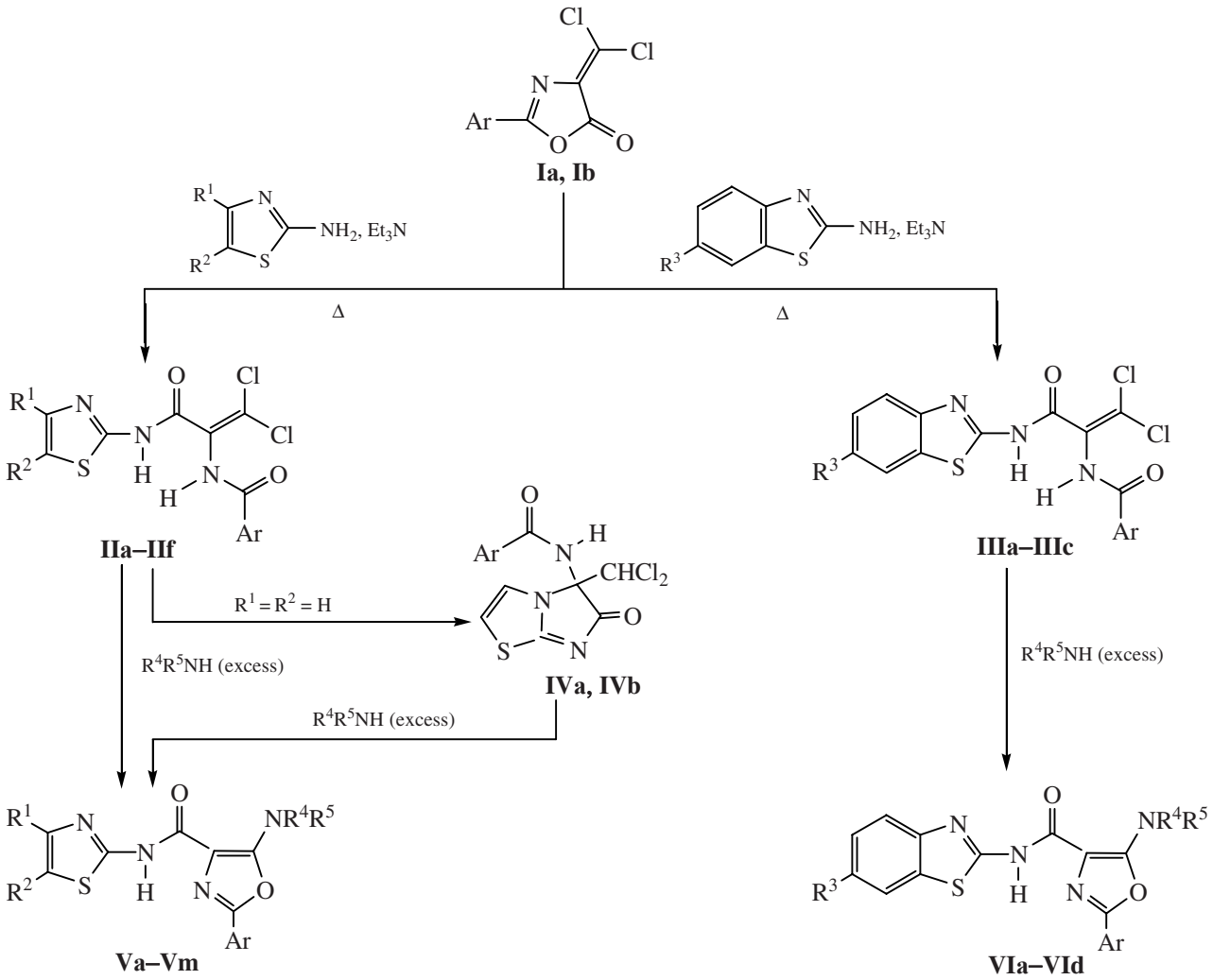
Accessible chlorine-containing unsaturated azlactones **I** are multicenter electrophilic compounds capably of reacting with soft and hard bases in different ways to produce a number of functionally substituted heterocycles [1–12]. We now report for the first time on the reaction of compounds **I** with 2-amino-1,3-thiazole and its analogs, 2-amino-4-phenyl-1,3-thiazole, 2-amino-4,5-dimethyl-1,3-thiazole, 2-amino-benzothiazole, and 2-amino-6-methylbenzo-thiazole. In all cases, the primary amino group in the thiazole derivatives attacks the C^5 atom of the oxazole ring in compounds **I**, leading to the formation of enamides **II** and **III** via opening of the oxazole ring. As a rule, such enamides can be isolated individual (Scheme 1, Table 1). However, we failed to isolate primary products of the reactions of **Ia** and **Ib** with 2-amino-1,3-thiazole, for they readily underwent intramolecular cyclization into fused imidazothiazoles **IVa** and **IVb**, respectively.

Presumably, the cyclization involves prototropic tautomers of **IIa** and **IIb** possessing clearly electron-deficient *N*-acylimino group which is capable of interacting with the lone electron pair on the nitrogen atom in the thiazole ring (transformation sequence **A**→**B**→**C** in Scheme 2. The presence of a dichloromethyl substituent in structure **C** follows from the ^{1}H NMR spectra which contained a characteristic signal at δ 6.51–6.61 ppm.

Introduction of a substituent into the 4-position of the thiazole ring in structure **A** is likely to create steric hindrances to intramolecular cyclization; therefore, primary products of the reactions of **I** with 2-amino-4-phenyl-1,3-thiazole, 2-amino-4,5-dimethyl-1,3-thiazole, 2-amino-1,3-benzothiazole, and 2-amino-6-methyl-1,3-benzothiazole can be isolated with no difficulties. The structure of enamides **II** and **III** was confirmed by spectral data and chemical methods. Comparison of the IR spectra of compounds **I**–**III** showed that the oxazole ring is opened in the early reaction; this follows from disappearance of carbonyl absorption typical of oxazolone carbonyl group (1790–1800 cm^{-1}; Table 2) from the IR spectra. On the other hand, the structure of compounds **II** and **III** as representatives of a broad series of chlorine-containing enamides with the general formula $Cl_2C{=}C(X)NHCOR$, where X is an electron-withdrawing group, is confirmed by their cyclocondensation with benzylamine, piperidine, and morpholine (transformations **II**→**V**→**III**→**VI** in Scheme 1). These cyclizations may be regarded as particular cases of the general synthesis of 5-amino-1,3-oxazole derivatives from chlorine-containing enamides [13].

Scheme 1.



Ia, **IIa**, **IIc**, **IIe**, **IIIa**, **IIIb**, **IVa**, **Va**, **Vc**, **Ve**, **Vg**, **Vi**, **Vk**, **Vm**, **VIa**, **VIb**, Ar = Ph; **Ib**, **IIb**, **IId**, **IIf**, **IIIc**, **IVb**, **Vb**, **Vd**, **Vf**, **Vh**, **Vj**, **Vl**; **VIc**, **VId**, Ar = 4-MeC_6H_4; **IIa**, **IIb**, **Va–Vd**, $R^1 = R^2 = H$; **IIc**, **IId**, **Ve–Vh**, R^1 = Ph, R^2 =H; **IIe**, **IIf**, **Vi–Vm**, $R^1 = R^2$ = Me; **IIIa**, **VIa**, R^3 = H; **IIIb**, **IIIc**; **VIb–VId**, R^3 = Me; **Va**, **Vb**, **Ve**, **Vf**, **Vi**, **Vj**, R^4R^5N = piperidino; **Vc**, **Vd**, **Vg**, **Vh**, **Vk**, **Vl**, **VIa–VIc**, R^4R^5N = morpholino; **Vm**; **VId**, R^4 = H, $R^5 = PhCH_2$.

Table 1. Yields, melting points, and elemental analyses of compounds **II–VI**

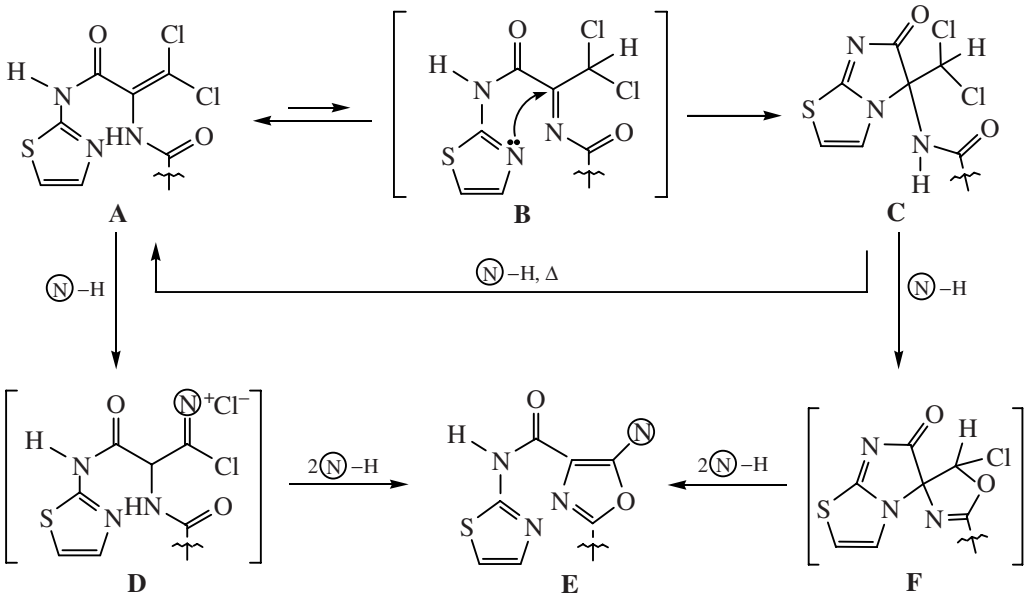
Comp. no.	Yield, %	mp, °C (solvent)	Found, %		Formula	Calculated, %	
			Cl (S)	N		Cl (S)	N
IIc	50	184–185 (EtOH)	16.15	10.28	$C_{19}H_{13}Cl_2N_3O_2S$	16.95	10.05
IId	50	190–192 (EtOH)	16.38	9.70	$C_{20}H_{15}Cl_2N_3O_2S$	16.40	9.72
IIe	46	195–196 (AcOH)	19.49	10.32	$C_{15}H_{13}Cl_2N_3O_2S$	19.15	11.35
IIf	61	181–182 (AcOH)	18.38	10.49	$C_{16}H_{15}Cl_2N_3O_2S$	18.45	10.93
IIIa	50	212–213 (AcOH)	17.95	11.14	$C_{17}H_{11}Cl_2N_3O_2S$	18.08	10.71
IIIb	57	177–178 (EtOH)	16.95	10.10	$C_{18}H_{13}Cl_2N_3O_2S$	17.45	10.34
IIIc	47	192–193 (CH_3CN)	16.84	9.97	$C_{19}H_{15}Cl_2N_3O_2S$	16.87	10.00
IVa	58	241–243 (DMF)	20.56	12.41	$C_{13}H_9Cl_2N_3O_2S$	20.72	12.28
IVb	71	196–197 (CH_3CN)	19.84	11.36	$C_{14}H_{11}Cl_2N_3O_2S$	19.90	11.80

Table 1. (Contd.)

Comp. no.	Yield, %	mp, °C (solvent)	Found, %		Formula	Calculated, %	
			Cl (S)	N		Cl (S)	N
Va	70	188–190 (DMF)	(9.14)	15.68	$C_{18}H_{18}N_4O_2S$	(9.05)	15.81
Vb	66	181–182 (DMF)	(8.59)	15.06	$C_{19}H_{20}N_4O_2S$	(8.70)	15.21
Vc	66	228–229 (DMF)	(8.87)	15.55	$C_{17}H_{16}N_4O_3S$	(8.99)	15.72
Vd	41	247–248 (DMF)	(8.48)	14.92	$C_{18}H_{18}N_4O_3S$	(8.66)	15.13
Ve	60	197–198 (CH_3CN)	(7.30)	12.55	$C_{24}H_{22}N_4O_2S$	(7.45)	13.01
Vf	46	204–205 (CH_3CN)	(7.59)	11.96	$C_{25}H_{24}N_4O_2S$	(7.21)	12.60
Vg	52	227–228 (CH_3CN)	(7.22)	11.97	$C_{23}H_{20}N_4O_3S$	(7.41)	12.95
Vh	61	225–226 (CH_3CN)	(7.34)	12.39	$C_{24}H_{22}N_4O_3S$	(7.18)	12.55
Vi	66	205–206 (EtOH)	(8.53)	14.48	$C_{20}H_{22}N_4O_2S$	(8.38)	14.65
Vj	55	231–232 (EtOH)	(8.21)	13.43	$C_{21}H_{24}N_4O_2S$	(8.09)	14.13
Vk	68	198–199 (EtOH)	(8.38)	14.68	$C_{19}H_{20}N_4O_3S$	(8.34)	14.57
Vl	48	234–235 (EtOH)	(7.40)	12.52	$C_{20}H_{22}N_4O_3S$	(8.05)	14.06
Vm	54	188–189 (*i*-PrOH)	(7.72)	13.25	$C_{22}H_{20}N_4O_2S$	(7.93)	13.85
VIa	61	251–252 (AcOH)	(8.12)	13.56	$C_{21}H_{18}N_4O_3S$	(7.89)	13.78
VIb	64	252–253 (AcOH)	(7.61)	12.81	$C_{22}H_{20}N_4O_3S$	(7.63)	13.32
VIc	64	240–241 (AcOH)	(7.16)	12.10	$C_{23}H_{22}N_4O_3S$	(7.38)	12.89
VId	50	212–213 (EtOH)	(7.42)	12.19	$C_{26}H_{22}N_4O_2S$	(7.05)	12.33

It is interesting that treatment of fused heterocyclic compounds **IV** with nitrogen bases also leads to the formation of analogous 1,3-oxazole derivatives. For example, by heating compound **IVa** (Ar = Ph) with morpholine we obtained 5-morpholino-2-phenyl-*N*-(1,3-thiazol-2-yl)-1,3-oxazole-4-carboxamide (**Vc**) whose structure was unambiguously determined by X-ray analysis. The general view of molecule **Vc** is shown in figure, and its principal geometric parameters are collected in Table 3.

Scheme 2.



Ⓝ = piperidino, morpholino, $PhCH_2NH$.

Table 2. Spectral parameters of some compounds **II–VI**

Comp. no.	^{1}H NMR spectrum (DMSO-d_6), δ, ppm
IId	2.40 s (3H, CH_3), 7.28–7.42 m (5H, C_6H_5), 7.55 s (1H, 5-H), 7.88–7.90 m (4H, H_{arom}), 10.17 s (1H, NH), 12.93 s (1H, NH)
IIf	2.18–2.40 m (9N, 3CH_3), 7.30–7.84 m (4H, H_{arom}), 10.10 s (1H, NH), 12.46 s (1H, NH)
IVa	6.61 s (1H, $CHCl_2$), 7.02–7.85 m (5H, H_{arom}, 2-H, 3-H), 9.67 s (1H, NH)
IVb[a]	2.39 s (3H, CH_3), 6.51 s (1H, $CHCl_2$), 6.97 d, 7.55 d (2H, 2-H, 3-H), 7.29 d, 7.72 d (4H, H_{arom}), 9.41 s (1H, NH)
Va[b]	1.65 s, 3.77 s [10H, $(CH_2)_5N$., 7.19–7.92 m (5H, H_{arom}, 4-H, 5-H), 10.81 s (1H, NH)
Vb[b]	1.65 s, 3.77 s [10H, $(CH_2)_5N$., 2.36 s (3H, CH_3), 7.18 d, 7.49 d (2H, 4-H, 5-H), 7.32 d (2H, H_{arom}), 7.85 d (2H, H_{arom}), 10.78 s (1H, NH)
Vc[b]	3.80 m [8H, $O(CH_2)_4N$., 7.21–7.98 m (5H, H_{arom}, 4-H, 5-H), 11.07 s (1H, NH)
Vd[b]	2.37 c (3H, CH_3), 3.79 m [8H, $O(CH_2)_4N$., 7.20 d, 7.49 d (2H, 4-H, 5-H), 7.33 d (2H, H_{arom}), 7.87 d (2H, H_{arom}), 10.83 s (1H, NH)
Vf	1.64 s, 3.76 s [10H, $(CH_2)_5N$., 2.36 s (3H, CH_3), 7.33–7.43 m (5H, C_6H_5), 7.58 s (1H, 5-H), 7.85–7.93 m (4H, H_{arom}), 10.82 s (1H, NH)
Vh[c]	2.38 s (3H, CH_3), 3.81 m [8H, $O(CH_2)_4N$], 7.34–7.44 m (5H, H_{arom}), 7.64 s (1H, 5-H), 7.97 m (4H, H_{arom}), 11.12 s (1H, NH)
VIg	2.37 s, 2.43 s (6H, 2CH_3), 2.98 m (2H, CH_2), 7.26–7.73 m (12H, H_{arom}), 8.48 m (1H, NH), 10.90 s (1H, NH)

[a] IR spectrum, ν, cm^{-1}: 1675 (NC=O), 1725 (C=O), 3050–3450 (N–H_{as}). [b] IR spectrumy, ν, cm^{-1}: 1660 (NC=O), 3170–3450 (N–H_{as}). [c] IR spectrum, ν, cm^{-1}: 1675 (NC=O), 3380 (N–H).

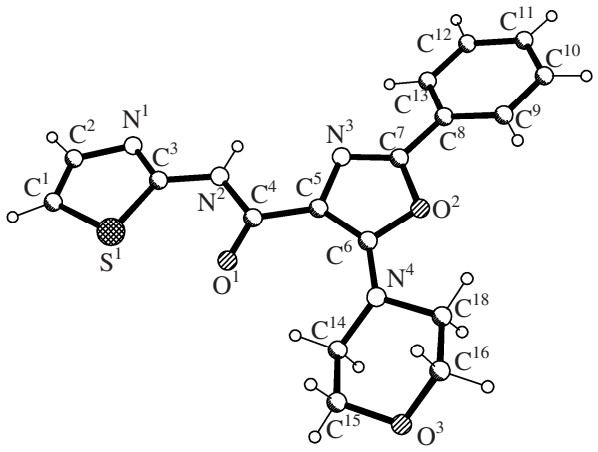
The oxazole ring in molecule **Vc** is almost planar: the mean-square deviation of atoms from the corresponding plane is 0.008 Å. The benzene ring C^8–C^{13} is turned by 18.1°, and the thiazole ring, by 10.6° with respect to the oxazole ring. The sum of the bond angles at N^2 is 360°, and the C^3–N^2 and C^4–N^2 bonds [1.378(2) and 1.370(2) Å, respectively] are shorter than the standard single C–N bond (1.45 Å), indicating conjugation between the lone electron pair on N^2 and π systems of the tiazole ring and carbonyl group. The C^4–C^5 bond is also slightly shortened as a result of effective conjugation between the oxazole and thiazole rings.

Table 3. Principal bond lengths (*d*) and bond angles (ω) in the molecule of 5-morpholino-2-phenyl-*N*-(1,3-thiazol-2-yl)-1,3-oxazole-4-carboxamide (**Vc**)

Bond	*d*	Bond angle	ω
C^1–C^2	1.328(3)	$C^2C^1S^1$	110.98(16)
C^1–S^1	1.711(2)	$C^1C^2N^1$	116.09(17)
C^2–N^1	1.375(2)	$N^1C^3N^2$	121.10(15)
C^3–N^1	1.3058(19)	$N^1C^3S^1$	115.67(13)
C^3–S^1	1.7205(17)	$N^2C^3S^1$	123.23(12)
C^3–N^2	1.378(2)	$N^2C^4C^5$	114.99(14)
C^4–N^2	1.370(2)	$C^6C^5N^3$	108.15(14)
C^4–C^5	1.447(2)	$C^6C^5C^4$	130.75(14)
C^5–C^6	1.381(2)	$N^3C^5C^4$	120.90(13)
C^5–N^3	1.398(2)	$O^2C^6C^5$	107.39(13)
C^6–O^2	1.367(2)	$N^3C^7O^2$	113.01(14)
C^7–N^3	1.289(2)		
C^7–O^2	1.3798(18)		

Taking into account similarity of the spectral data of compounds **V** and **VI** (Table 2), we can contend with certainty that they have the structure of 5-amino-1,3-oxazole derivatives having an N-substituted carboxamide group on C^4 due to regioselective cleavage of the oxazole ring in **I** by the primary amino group in 1,3-thiazole bases.

The subsequent transformations of enamides **A** are illustrated by Scheme 2. It should be noted that the first stages of the transformation **A**→**B**→**C** resemble cycloaddition of benzamidine to compounds **I** [12]. However, cycloaddition products derived from benzamidine are converted into pyrimidine derivatives



Structure of the molecule of 5-morpholino-2-phenyl-*N*-(1,3-thiazol-2-yl)-1,3-oxazole-4-carboxamide (**Vc**) according to the X-ray diffraction data.

on heating with morpholine or piperidine [14], while compounds **C** react with nitrogen bases according to the scheme **C** → **A** → **D** → **E** or **C** → **F** → **E**. Although it is difficult to choose between these alternative pathways, the final products are the same 5-amino-4-carbamoyl-1,3-oxazole derivatives. Additional studies are necessary to elucidate the reasons for the different reactivities of the cycloaddition products of benzamidine and 2-amino-1,3-thiazoles to compounds **I**; nevertheless, the basicity of reagents having a –C(NH_2)=N– fragment is likely to be important in both cases.

EXPERIMENTAL

The IR spectra were recorded on a Specord M-80 spectrometer from samples prepared as KBr pellets. The ^{1}H NMR spectra were measured on a Varian VXR-300 instrument from solutions in DMCO-d_6 using tetramethylsilane as internal reference.

X-Ray analysis of compound Vc. The X-ray diffraction data for a 0.15×0.25×0.50-mm single crystal of compound **Vc** were obtained on a Bruker Smart Apex II diffractometer (λMoK_α irradiation, graphite monochromator, θ_{max} 26.54°, spherical segment $-10 \le h \le 10$, $-11 \le k \le 12$, $-24 \le l \le 26$). Monoclinic crystals, space group $P2_1/c$ (no. 14); $C_{17}H_{16}N_4O_3S$; M 356.40; a = 8.1763(3), b = 9.5996(3), c = 20.8397(6) Å; β = 90.729(2)°; V = 1635.56(9) Å^3; Z = 4; d_{clac} = 1.447 g/cm^3; μ = 0.223 mm^{-1}; $F(000)$ = 744. Total of 12046 reflections were collected, 3682 of which were independent (averaging factor R = 0.0275). The structure was solved by the direct method and was refined by the least-squares procedure in full-matrix anisotropic approximation using SHELXS-97 and SHELXL-97 software packages [15, 16]. The refinement procedure was performed using 2821 reflections with $I > 2\sigma(I)$ (290 refined parameters, 9.73, reflections per parameter) and the weight scheme $\omega = 1/[\sigma^2(Fo^2) + (0.0580P)^2 + 0.2891R]$, where $R = (Fo^2 + 2Fc^2)/3$; the ratio of the maximal (average) displacement to the error in the last cycle was 0.016 (0.002). A correction for absorption was intriduced using SADABS program (the minimal-to-maximal correction ratio was T_{min}/T_{maks} = 0.844037). All hydrogen atoms were visualized objectively from the Fourier difference series, and their positions were refined in isotropic approximation. The final divergence factors were $R_1(F^2)$ = 0.0557, = $R_W(F^2)$ 0.1114 (for all independent reflections; GOF 1.017) and $R_1(F)$ = 0.0394, $R_W(F^2)$ = 0.1016 [for reflections with $I > 2\sigma(I)$; GOF 1.017]. The residual electron density from the Fourier difference series after the last iteration cycle was 0.23 and –0.19 e Å^{-3}.

***N*-[2,2-Dichloro-1-(4-phenyl-1,3-thiazol-2-ylcarbamoyl)vinyl]benzamides IIc and IId** (*general procedure*)**.** A mixture of 0.008 mol of compound **Ia** and **Ib**, 0.008 mol of 4-phenyl-1,3-thiazol-2-2-amine, and 0.008 mol of triethylamine in 20 ml of THF was heated for 7 h under reflux. After cooling, the precipitate of triethylamine hydrochloride was filtered off, the filtrate was evaporated under reduced pressure, the residue was treated with water, and the precipitate was filtered off and purified by recrystallization.

***N*-[2,2-Dichloro-1-(4,5-dimethyl-1,3-thiazol-2-ylcarbamoyl)vinyl]benzamides IIe and IIf** (*general procedure*)**.** A mixture of 0.008 mol of compound **Ia** or **Ib**, 0.008 mol of 4,5-dimethyl-1,3-thiazol-2-amine hydrochloride, and 0.016 mol of triethylamine in 20 ml of THF was heated for 7 h. The mixture was cooled, and the precipitate was filtered off and purified by recrystallization.

***N*-[1-(1,3-Benzothiazol-2-ylcarbamoyl)-2,2-dichlorovinyl]benzamide (IIIa).** A mixture of 0.008 mol of compound **Ia**, 0.008 mol of 1,3-benzothiazol-2-amine, and 0.008 mol of triethylamine in 20 ml of THF was heated for 7 h under reflux. After cooling, the precipitate of triethylamine hydrochloride was filtered off, the filtrate was evaporated under reduced pressure, the residue was treated with water, and the precipitate was filtered off and purified by recrystallization.

***N*-[2,2-Dichloro-1-(6-methyl-1,3-benzothiazol-2-ylcarbamoyl)vinyl]benzamides IIIb and IIIc** were synthesized in a similar way from compounds **Ia** and **Ib**, respectively, and 6-methyl-1,3-benzothiazol-2-amine.

***N*-(5-Dichloromethyl-6-oxo-5,6-dihydroimidazo-[2,1-*b*]thiazol-5-yl)benzamides IVa and IVb** (*general procedure*)**.** A mixture of 0.04 mol of compound **Ia** and **Ib**, 0.04 mol of 1,3-thiazol-2-amine, and 0.04 mol of triethylamine in 90 ml of THF was heated for 7 h under reflux. The precipitate was filtered off and purified by recrystallization.

2-Aryl-5-piperidino(morpholino)-*N*-(1,3-thiazol-2-yl)-1,3-oxazole-4-carboxamides Va–Vd (*general procedure*)**.** A mixture of 0.003 mol of compound **IVa** and **IVb** and 0.012 mol of piperidine or morpholine in 5 ml of pyridine was heated for 4 h under reflux. The mixture was cooled, 10 ml of 5% hydrochloric acid was added, and the precipitate was filtered off and purified by recrystallization.

2-Aryl-5-piperidino(morpholino)-*N*-(4-phenyl-1,3-thiazol-2-yl)-1,3-oxazole-4-carboxamides Ve–Vh (*general procedure*)**.** A mixture of 0.005 mol of compound **IIc** or **IId** and 0.02 mol of piperidine or morpholine in 10 ml of pyridine was heated for 4 h under reflux. The mixture was cooled, 15 ml of 5% hydrochloric acid was added, and the precipitate was filtered off and purified by recrystallization.

2-Aryl-*N*-(4,5-dimethyl-1,3-thiazol-2-yl)-5-piperidino(morpholino)-1,3-oxazole-4-carboxamides Vi–Vl were synthesized in a similar way from enamides **IIe** and **IIf**.

5-Benzylamino-*N*-(4,5-dimethyl-1,3-thiazol-2-yl)-2-phenyl-1,3-oxazole-4-carboxamide (Vm) was synthesized in a similar way from enamide **IIe** and benzylamine.

2-Aryl-*N*-(1,3-benzothiazol-2-yl)-5-morpholino (benzylamino)-1,3-oxazole-4-carboxamides VIa–VId were synthesized as described above for **Ve–Vh** from enamides **IIIa–IIIc** and morpholine or benzylamine.

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