INVESTIGATION OF HETEROCYCLES CONTAINING NITROGEN OR SULFUR.

47.* REACTIONS OF 1,2-DIOXO-3a-ALKYL-4,7-DICHLOR-oXAZOLIDINO[3,2-f]PYRIDO[2,3-b]-1,4-THIAZINES WITH SATURATED CYCLIC AMINES. SYNTHESIS AND STRUCTURE OF DERIVATIVES OF 1,2-DIHYDROTHIAZOLO[5,4-b]PYRIDINE

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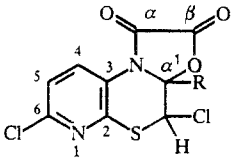
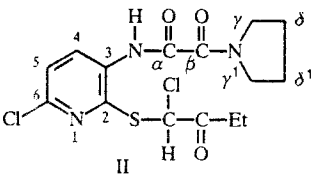
It has been established spectroscopically that 1-N-oxalamides of 2-acyl-5-chloro-1,2-dihydrothiazolo[5,4-b]pyridine are formed in the reaction of 4,7-dichloroxazolidino[3,2-f]pyrido[2,3-b]-1,4-thiazines with morpholine, piperidine and pyrrolidine. The products are a mixture of amide conformers in solution. The reaction intermediate 2-(1-chloro-2-oxobutylthio)-3-pyrrolidinooxamoyl-6-chloropyridine has been isolated and characterized. A proposed reaction scheme is presented.

We established previously that the reaction of oxazolidino[3,2-f]pyrido[2,3-b]-1,4-thiazines with a mixture of PCl_5 and $POCl_3$ gave the 4,7-dichloro derivatives (Ia,b) which existed in solution as mixtures of the *SS/RR(cis)* and *SR/RS(trans)* diastereoisomers [2].

In this paper the reactions of compounds Ia,b with saturated cyclic amines have been studied. These compounds react extremely readily with these amines with opening of the oxazolidine and 1,4-thiazine rings to give pyridine derivatives of type (II) which are easily converted to the N,N-disubstituted aminooxalyl-2-acyl-5-chloro-1,2-dihydrothiazolo[5,4-b]pyridines (IIIa-



Ia, b



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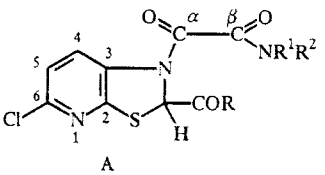
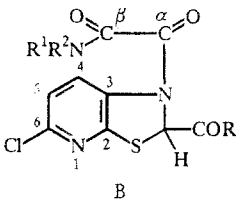


A



B

IIIa- d

I, III a R = Et, NR^1R^2 = morpholino; bR = Pr, NR^1R^2 = morpholino; c R = Et, NR^1R^2 = piperdino, d R = Et, NR^1R^2 = pyrrolidino

* For Communication 46 see [1].

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TABLE 1. Characteristics of Compounds II and IIIa-d

Compound	Molecular formula	mp, °C	R_f	IR spectrum, ν, cm^{-1}, C=O		UV spectrum, λ_{max}, nm (lg ε)	Yield, %
				amide	ketone		
II*	$C_{15}H_{17}Cl_2N_3O_3S$	125...127	0,7	1630,1700	1745	235(3,97), 292(3,95)	74
IIIa	$C_{15}H_{16}ClN_3O_4S$	185...186	0,6	1630...1660	1720	225(4,22), 284(3,89), 324(4,01)	74
IIIb	$C_{16}H_{18}ClN_3O_4S$	150...152	—	1650...1660	1720	228(4,21), 284(3,92) 324(3,98)	70
IIIc	$C_{16}H_{18}ClN_3O_3S$	152...154	0,76	1640...1670	1725	226(3,63), 280(4,14) 324(3,87)	86
IIId	$C_{15}H_{16}ClN_3O_3S$	154...156	0,64	1650	1720	228(4,14), 284(3,57) 318(3,69)	90

*For compound II ν_{NH} = 3280 cm^{-1}.

d). Thus compounds IIIa-d were obtained in 79-90% yields when the dichloro compounds Ia,b were boiled with morpholine, piperidine, and pyrrolidine. The structures of the products were established by analysis of their IR, UV, mass, ^1H and ^{13}C NMR spectra* (Tables 1-4).

The $\nu_{C=O}$ band for the acyl fragment in the IR spectra of compounds IIIa-d was observed at 1716-1720 (crystal) and 1720-1725 cm^{-1} ($CHCl_3$ solution) while the intense carbonyl band of the amide group was observed at 1650-1654 (crystal) and 1630-1670 cm^{-1} (in $CHCl_3$) which differs considerably from the starting compounds Ia,b for which carbonyl stretching vibrations of the oxazolidine ring were recorded (1730-1755 and 1810-1830 cm^{-1}) [2]. In contrast the bands for the $\nu_{C=C}$ and $\nu_{C=N}$ double bonds (1555-1570 cm^{-1}) arising from the presence of the pyridine ring changed little in the spectra of compounds IIIa-d from those in the starting compounds Ia,b ($\nu_{C=C}$ and $\nu_{C=N}$ 1550-1570 cm^{-1}).

The UV spectra of compounds IIIa-d differ markedly from those of compounds Ia,b. Three wavelength maxima were observed for IIIa-d: 225-228 (lg ε 3.6-4.2), 280-284 (3.6-4.1) and 318-324 nm (3.7-4.0) whereas two wavelength maxima are present in the spectra of compounds Ia,b: 240-242 (lg ε 4.0-4.1) and 296 nm (3.8-3.9) [2].

The mass spectra of the products have molecular ions with odd numbered masses (Table 2) which indicates an odd number of nitrogen atoms in the molecules in distinction from compounds Ia,b. The ratios of the intensities of the isotopic peaks indicate the presence of a single chlorine atom. The character of the mass spectroscopic decomposition of these compounds is sharply different from those of the dichloro compounds Ia,b. For example, peaks of ions corresponding to the elimination of CO and CO_2 units from the oxazolidine ring and elimination of the chlorine atom α to the sulfur atom are absent (this fragmentation route is basic for compounds Ia,b and the corresponding ions have maximum intensities [2]). Peaks for the ions $[M - CO - CO_2]^+$ corresponding to bond rupture in the oxazolidine ring, characteristic of the fragmentation of unsubstituted oxazolidinothiazines [3] were absent. The mass spectra of these compounds are distinguished by the extremely high selectivity of the fragmentation ($s_{1/2}$ = 2). The base peaks correspond to the ions $CONR^1R^{2+}$, $[CONR^1R^2 - C_2H_4O]^+$ (IIIa,b) and $[CONR^1R^2 - CONH]^+$ (IIIc,d) (see Table 2). The compositions of these peaks were determined from high resolution spectra of compounds IIIa, c and d. According to data from the DADI spectra the $CONR^1R^{2+}$ ion is formed directly from the molecular ion with later elimination of C_2H_4O (IIIa,b) and CONH fragments (IIIc,d). These results show the presence of $CONR^1R^2$ terminal groups in compounds IIIa-d, while the appearance of the $[M - COR]^+$ ion shows the presence of the COR group.

The ^{13}C NMR spectra of products IIIa, c and d also differ considerably from those of the starting materials Ia,b (see Table 3). For example the former contain signals for carbon atoms of the NR^1R^2 fragment and acyl groups (at 200 ppm) while the signal of the quaternary sp^3 hybridized carbon Cα', observed in the spectra of the starting compounds I (δ ~ 90 ppm), is absent. In addition, the signals of the two amide carbonyl groups are observed at weaker field than in the spectra of the starting materials I. In contrast, the chemical shifts of the carbon atoms of the pyridine units in compounds I and III are similar.

Thus the spectroscopic data cited are in excellent agreement with the structures of compounds IIIa-d as derivatives of dihydrothiazolopyridine.

* To ease examination of the spectroscopic results for compounds I-III we have used numbering of the atoms of the pyridine ring and the dicarbonyl fragment corresponding to those of compound II, but the compounds have been named by the normal nomenclature rules (see text).

TABLE 2. Mass Spectral Data for Compounds IIIa-d, m/z (I_{rel}, %)

Compound	Characteristic ions							Other ions*
	M^+	$[M-COR]^+$	$[M-CONR^1R^2]^+$	$[CONR^1R^2]^+$	$[CONR^1R^2]^{+3*}-C_2H_4O]^+$	$[CONR^1R^2]^{+4*}-CONH]$	$[M-(C_2O_2NR^1R^2-H)]^+$	
IIIa	369(2)	312(6)	254(1)	114(100)	70(53)	—	227(12)	171(3), 115(7), 57(5), 56(3), 42(15)
IIIb	383(1)	312(3)	268(1)	114(100)	70(46)	—	241(6)	115(6), 57(3), 43(9), 42(7)
IIIc	367(1)	310(2)	254(1)	112(100)	—	69(40)	227(8)	113(7), 56(5), 41(27)
IIId	353(2)	296(3)	254(1)	98(100)	—	55(43)	227(10)	99(6), 70(8), 57(5), 56(16)

*Ions with $I_{rel} \geq 3\%$ (^{35}Cl).

2*Composition, found/calculated (here and below on the basis of high resolution mass spectroscopic results) for the ions $C_5H_8NO_2$ (IIIa) 114.0547/114.0555, $C_6H_{10}NO$ (IIIc) 112.0748/112.0762, and C_5H_8NO (IIId) 98.0608/98.0606.

3*Composition of the ion $C_3H_4NO_2$ (IIIa): 70.0291/70.0293.

4*Composition of the ion C_5H_9 (IIIc) 69.0702/69.0704; C_4H_7 (IIId) 55.0566/55.0556.

The room temperature ^1H NMR spectra of compounds IIIa, c and d show a doubling of all signals (see Table 4). These signals broaden and then coalesce as the temperature is raised which clearly shows that compounds IIIa, c and d exist as two amide conformers relative to the $-N_{(1)}-C_{(\alpha)}=O$ bond. The $C_{(4)}H$ signal for the major conformer (A) appears at considerably weaker field than that for the minor isomer (B) ($\Delta\delta$ $C_{(4)}H$ ~ 1.19-1.27 ppm) whereas $\Delta\delta$ $C_{(5)}H$ is small (~0.05-0.07 ppm). The different values of δ $C_{(4)}H$ in the amide conformers may be explained by the influence of the $-C_{(\alpha)}=O$ group which is close to position 4 of the pyridine ring in conformer A, but distant from it in conformer B. Starting material I, in which the analogous orientation of $-C_{(\alpha)}=O$ relative to $C_{(4)}H$ of the pyridine ring is firmly fixed by the oxazolidine ring, may serve as a model for conformer A. The values of the chemical shifts δ $C_{(4)}H$ for Ia,b [2] are close to those for conformer A of compounds III (see Table 4).

The ASIS effect [4] confirmed the assignment of the amide conformers. The ^1H NMR signals of the $CONR^1R^2$ fragment oriented *trans* to the C=O group of the N-alkyl substituent underwent a larger strong field shift on addition of benzene to the solution than the signal of the *cis* oriented group. When 10% C_6D_6 was added to a $CDCl_3$ solution of IIIa the ^1H signal of the methine proton of the $-S-CH-(NC_\alpha O)CO$ fragment of the predominant conformer underwent a larger shift to strong field (0.11 ppm) and the signal for the minor conformer underwent a considerably smaller shift (0.04 ppm). Thus the predominant conformer has structure A.

The existence of compounds IIIa-d as two conformers with respect to the $-N_1-C_{(\alpha)}=O$ bond was confirmed by the ^{13}C NMR spectra which also showed two sets of signals in the same intensity ratio as in the ^1H NMR spectra. The largest ^{13}C chemical shift difference between the conformers is for $C_{(4)}$ ($\Delta\delta$ $C_{(4)}$ ~ 4.4-4.7 ppm) while the differences are somewhat smaller for the carbons of the ketone groups ($\Delta\delta$ C=O ~ 1.3-2.2 ppm), the methine carbon of the $-S-CH(N-C_\alpha=O)-C=O$ ($\Delta\delta$ CH ~ 0.9-1.1 ppm) and the $\gamma\gamma'$-carbons of the aminocarbonyl fragment ($\Delta\delta$ γ-CH_2 ~ 0.6 ppm). The position of the methine carbon signal of the $-S-CH(N-C_\alpha=O)-C=O$ fragment of the minor conformer ($\Delta\delta$ CH ~ 1.1-0.9 ppm) at higher field agrees with literature data [5] and shows that this conformer has structure B in agreement with the assignment made above on the basis of the ^1H NMR spectrum.

TABLE 3. Chemical Shifts (ppm) and Coupling Constants (J, Hz) in the ^{13}C NMR Spectra of Compounds I-III*

Compound	Isomer type	Pyridine ring					Amidocarbonyl group				$COCH_2CH_3$ fragment			Fragment*5 ($^1J_{CH}$)
		$C_{(2)}$ ($\Sigma\ ^3J$)	$C_{(3)}$ ($^3J_{C(3)H(5)}$)	$C_{(4)}$ ($^1J_{CH}$)	$C_{(5)}$ ($^1J_{CH}$)	$C_{(6)}$ ($^3J_{C(6)H(4)}$)	C_α=O	C_β-O	γ-CH_2; γ'-CH_2 ($^1J_{CH}$)	δ-CH_2; δ'-CH_2 ($^1J_{CH}$)	C=O ($\Sigma\ ^3J_{COH}$ + $^2J_{COH}$)	CH_2 ($^1J_{CH}$)	CH_3 ($^1J_{CH}$)	
Ia	*SS/RS*	142,2 q (~8,0)	125,4 d (7,6)	131,6 dd (171,4)*2 (~1,3)*2	121,9 d (175,2)	148,6 d (11,7)	155,8 s	149,8 s	—	—	—	28,4 t (130,4)	5,9 q (128,3)	62,4 d (169,3)
	SS/RR	146,5 q (~8,0)	124,5 d (7,0)	131,2 dd (171,5)*2 (~1,3)*2	121,1 d (175,1)	148,3 d (11,0)	156,1 s	149,7 s	—	—	—	21,9 t (131,2)	4,5 q (128,0)	57,5 d (177,2)
II	—	145,4 m (~10,0)	130,9 d (6,0)	131,2 d (170,1)	122,1d (172,9)	146,1d (11,0)	158,4 s	157,6 br.s (~3)*3	48,3; 48,8 t (142,5) (146,7)	23,4, 26,8 t (131,0) (131,3)	199,6 m (~15,0)*3	32,4 t (126,7)	8,0 q (128,6)	64d (166,6)
IIIa	A	150,8 m (~10)	132,5 m (14)	126,3 dd (174,1)*2 (1,5)*2	120,2 d (172,5)	146,6d (10,8)	161,0br.s	160,3 br.s (~5)*3	42,0; 47,0 t (141,1)	66,2; 66,4 t (144,7)	201,4 m (~15,0)	30,6t (126,1)	7,8 q (128,7)	66,0 d (160,9)
	B	150,8 m (~10)	132,5 m (14)	121,6 d (168,3)	120,2 d (172,5)	146,6d (10,8)	161,0br.s	160,3 br.s	41,8; 46,3 t (140,0)	66,2; 66,4 t (144,7)	199,2 m (~15,0)	30,6t (126,1)	7,5 q (128,0)	65,1 d (160,0)
IIIb	A	151,5 m (~10)	133,0 m (10)	126,4 dd (174,4)*2 (1,3)*2	120,1 d (172,7)	146,3 d (10,5)	161,7 d	159,3 br.s (~6)*3	46,1; 47,8 t (143,0) (144,0)	23,6; 25,9 t (133,0) (133,0)	201,5 m (15,7)	30,3t (126,5)	7,6 q (128,0)	66,0 d (161,1)
	B	151,5 m (~10)	133,0 m (11)	121,9 d (168,8)	120,2 d (172,9)	146,3 d (10,5)	161,8s	159,2 s	45,4; 46,8 t (143,0) (143,0)	23,9; 25,6 t (133,0) (133,0)	199,7 m (~15,0)	30,1t (126,0)	7,5 q (128,0)	64,9 d (160,5)
IIIc*4	A	151,2 m (~11)	132,6 m (1,1)	126,3 dd (174,4)*2 (1,5)*2	120,3 d (172,6)	146,4 d (11,2)	161,8s	160,2 br.s (~5)*3	242,6; 47,6 t (139,0)	25,2; 25,7 t (128,6)	201,2 m (~15,0)*3	30,3t (126,2)	7,6 q (128,8)	65,9d (160,3)
		151,2 m (~11)	132,7 m (~11)	121,9 d (168,3)	120,2 d (172,6)	146,4 d (11,2)	162,0s	160,7 br.s (~5)*3	42,2; 47,0 t (139,0)	25,1; 25,9 t (129,9)	199,9 m (~15,0)*3	30,5 t (126,0)	7,4 q (128,8)	65,0d (160,5)

*Spectra recorded at room temperature in $CDCl_3$ or 10:1 $CDCl_3$–DMSO (compound Ia).

*2For $^2J_{C(4)H(5)}$.

*3$l_{1/2h}$

*4The ^{13}C signal for compound IIIc (A and B) in ε-CH_2: 24 t (129.5).

TABLE 4. ^{1}H NMR Spectra of Compounds II and IIIa-d ($CDCl_3$, 23°C), ppm

Compound	Isomer type	S-CH-CO s	$C_{(4)}H$ d	$C_{(5)}H^{**}$ d	NH br.s	CH_3CH_2 t	CH_3CH_2 q(u), m III	NR^1R^2 m	Isomer content, %
II	—	6.34	8.35	7.20	9.66	1.19	2.90	1.96 (δ-CH_2) 3.60...4.00 (γ-CH_2)	—
IIIa	A	6.46	8.38	7.07	—	1.13	2.30...2.90	3.44...3.90 (γ-CH_2,δ-CH_2)	86
	B	6.26	7.15	7.02	—	1.14	2.30...2.90		14
IIIc	A	6.32	8.37	7.07	—	1.11	2.24...2.92	157...1.68 (δ,ε-CH_2)	80
	B	6.27	7.17	7.00	—	1.14	2.24...2.92	3.50 (γ-CH_2)	20
IIId	A	6.57	8.39	7.07	—	1.10	2.34...2.86	1.92 (δ-CH_2)	95
	B	6.23	7.12	7.01	—	*	2.34...2.86	3.30...3.90 (γ-CH_2)	5

*Signal of the CH_3 group masked by the analogous signal of the major isomer.
**$^3J_{C(4)H(5)}$ = 8.5 Hz.

It follows from analysis of the direct heteroatom coupling constant ^1J_H for atom $C_{(4)}$ of the pyridine ring that the coupling constant is 5.6-6.1 Hz smaller in conformer B than in conformer A. The different values of $^1J_{C(4)H}$ for the conformers of compounds IIIa, c and d may be explained by partial deformation of the pyridine ring in conformer B as a result of steric hindrance. It is known that small changes in the angles in an aromatic ring ($\Delta\varphi \sim 6°$) lead to notable changes ($\sim$7 Hz) in the direct ^{13}CH coupling constant [6]. An examination of molecular models of these compounds also leads to the conclusion that conformers show relatively large steric hindrance. Evidently this factor provides a large energetic advantage to conformer A of compounds IIIa, c and d relatively to conformer B which explains the predominance of the former in solutions (see Table 4).

Freshly prepared compound Ia, containing 55% of the *RS/SR* (*trans*) and 45% of the *SS/RR* (*cis*) diastereomers according to ^{1}H NMR spectroscopy, was used in the amination reaction to determine the reactivity of the diastereomers of the starting materials qualitatively.

When the reaction was carried out at −10°C a considerable amount of unreacted *RS/SR* isomer of Ia* was isolated along with the product IIIa. This provides a basis for the suggestion that the sterically less stable *RR/SS* (*cis*) isomer reacts more rapidly with morpholine to give product IIIa. Increasing the temperature leads to partial (at 20°C) or complete (at 78°C) participation of the *RS/SR* isomer of Ia in this reaction.

When the reaction of oxazolidinopyridothiazoline Ia with pyrrolidine was carried out at −10°C, the intermediate II was isolated from the reaction mixture in high yield. From spectroscopic data II is 2-(1-chloro-2-oxobutylthio)-3-pyrrolidinooxamoyl-6-chloropyridine.

The solid state and $CHCl_3$ solution IR spectra of compound II contain stretching bands assigned to the amide C=O group (1700 and 1630), the ketonic C=O group (1745) and the NH group (3280 cm^{-1}).

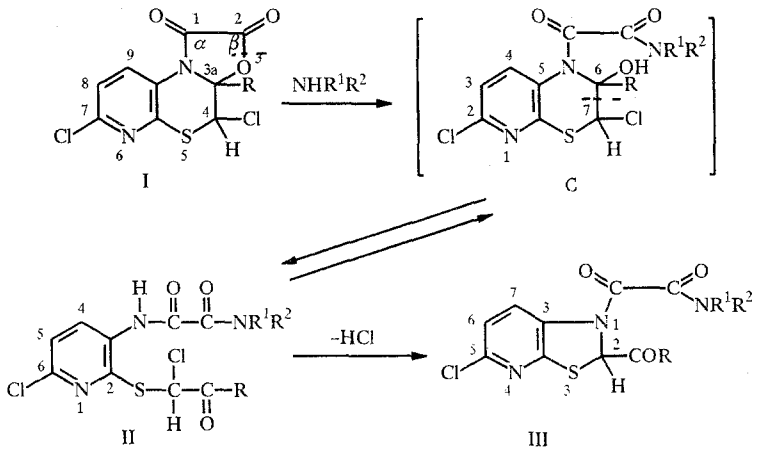
The mass spectrum of product II contains a molecular ion peak with mass 389 which corresponds to the addition of a molecule of pyrrolidine to compound Ia. The relative intensities of the isotope peaks in the molecular ion group show that both chlorine atoms are retained in compound II. The most intense peaks in the mass spectrum of II, as in that of the end product IIId, are those of the ions $CONR^1R^{2+}$ (98) and $[CONR^1R^2 - CONH]^+$ (55). An intense peak for the ion $COC_2H_5^+$ (57) was also observed in the spectrum of II. All of these confirm the presence of terminal amide and acyl groups in compound II.

In contrast to the spectrum of compound IIId, the ^{1}H NMR spectrum contains a single set of signals, including some characteristic of IIId (protons of the pyridine and pyrrolidine rings and the $-S-CH-CO-CH_2CH_3$) together with a signal for a single mobile proton at 9.66 ppm, the position of which corresponds well to that of the proton of an amide NH group.

*The structure of the intermediate product in this reaction mixture will be discussed later.

It should be noted that the size of the direct spin–spin coupling constant $^1J_{CH}$ for the methine group in the intermediate product II ($^1J_{CH}$ = 166.6 Hz, δ CH = 64.7 ppm) differs considerably from those for IIIc (A, $^1J_{CH}$ = 161.1 Hz, δ CH = 66.0 ppm; B, $^1J_{CH}$ = 160.5 Hz, δ CH = 64.9 ppm) but coincides with that for the –S–CH(Cl)–CO–Alk (167 Hz) calculated by the additive method [7]. The data cited are in excellent agreement with the structure proposed for compound II.

If a reaction mixture containing intermediate II (from spectroscopic evidence) is kept at 80°C for 30 min, the end product IIId is formed. On this basis, the scheme below is proposed for the conversion of compound I into compound III.



In the first step the oxazolidine ring is opened at the Cβ–O bond to give a hydroxyamino compound of type C. It has been demonstrated [8] that opening of the thiazine ring at the $C_{(6)}–C_{(7)}$ bond is characteristic of such compounds. Compound II formed in this way is cyclized to product III by dehydrochlorination.

Intermediate compounds of types C and II can be regarded as tautomeric forms in ring–chain tautomerism. Tautomerism of this type has been described for derivatives of a series of pyrimidothiazines [9].

EXPERIMENTAL

Purity of compounds was confirmed by thin layer chromatography with 1:1 benzene–ethyl acetate on Silufol UV-254 strips. The chromatograms were developed with UV light. IR spectra of Nujol mulls or chloroform solutions were recorded with a Perkin Elmer 599 instrument and UV spectra of ethanolic solutions were recorded with a Perkin Elmer 575 spectrophotometer. ^1H and ^{13}C NMR spectra were recorded on a Varian XL-200 spectrometer with TMS as internal standard. Electron impact and DADI mass spectra were obtained with a Varian MAT 112 mass spectrometer with direct insertion of samples into the ion source, an ionizing electron energy of 70 eV, and an ionization chamber temperature of 180°C. High resolution mass spectra* were obtained with a Varian MAT 311a spectrometer (1 part in 15,000 resolution) by the peak superposition method. The ionizing electron voltage was 70 eV.

Characteristics of the compounds synthesised are given in the tables.

Elemental analyses for C, H, N, Cl, and S for compounds II and IIIa-d agreed with the calculated values.

2-(1-Chloro-2-oxobutylthio)-3-pyrrolidinooxamoyl-6-chloropyridine (II). A suspension of compound Ia (0.5 g, 1.5 mmole) in ethanol (10 cm^3) was stirred with pyrrolidine (0.11 g, 1.5 mmole) at –10°C for 1 h. The reaction mixture was warmed later to 18-20°C. The precipitate of II was filtered off, washed with water, dried and crystallized from ethanol. Mass spectrum, *m/z* (I_{rel}): 389 (1), 353 (3), 333 (4), 298 (15), 296 (11), 263 (3), 252 (26), 227 (11), 194 (4), 171 (17), 135 (4), 99 (13), 98 (100), 70 (45), 57 (33), 56 (45), 55 (90).

1-Aminooxalyl-2-acyl-5-chloro-1,2-dihydrothiazolo[5,4-b]pyridines (IIIa-d). A suspension of compound Ia,b (3 mmole) and the corresponding amine (4.5-5.0 mmole) in ethanol (25 cm^3) was stirred for 30 min at 78°C. The solution was cooled, the product IIIa-d was filtered off, washed with water, dried and crystallized from ethanol.

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