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CHEMICAL PROPERTIES OF YLIDENE DERIVATIVES OF AZINES. 3*. SYNTHESIS AND REACTIONS OF YLIDENE DERIVATIVES OF HALOPYRIMIDINES

O. A. Zagulyaeva, I. V. Semushkina, and V. P. Mamaev

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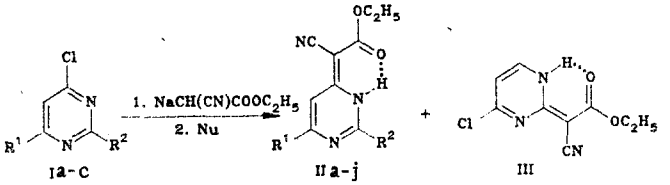
Some dihydropyrimidines have been obtained which contain a cyanoacetic ester or malononitrile residue and chlorine in positions, 2, 4, and 6. The reactions of these compounds with nucleophiles has been examined.

Continuing an investigation of methods of synthesis and chemical reactions of substituted ylidene derivatives of pyrimidine, we have examined the reaction of the dichloropyrimidines (Ia-c) with the sodium salts of cyanoacetic ester and malononitrile in aprotic bipolar solvents, and some further reactions of the products. Replacement of one chlorine atom by a cyanoacetic ester residue occurs with (Ia-c) to give the sodium salts of esters (IIa-c). The solvents used for the stable 4-chloro-derivative (IIa) may be DMSO or other aprotic bipolar solvents, but in the case of unstable pyrimidines such as (IIb), dimethoxy ethanes with the addition of DMF was employed. We have described the synthesis of the 4-chloro-compound (IIa) previously [2], but the spectral properties and some further reactions of this compound are described here.



I, II a R^1=Cl, b R^1=H, c R^1=C_6H_5; a R^2=H, b, c R^2=Cl; II d R^1=$N(CH_3)_2$, e R^1=NC_5H_{10}, f R^1=$NHNH_2$, g R^1=H, h–j R^1=C_6H_6; d–f R^2=H, g, i R^2=NC_5H_{10}, h R^2=$N(CH_3)_2$, j R^2=$NHNH_2$; Nu=$NH(CH_3)_2$, NHC_5H_{10}, NH_2NH_2

We have failed to observe replacement of a second chlorine atom by the cyanoacetic ester residue in pyrimidines (Ia-c), even in the presence of a large excess of sodiocyanoacetic ester, apparently as a result of the considerable deactivation of the chlorine in the sodium salts of (IIa-c). For example, we were unable to replace the chlorine atom in (IIa-c) by an alkoxy group on treatment with sodium alkoxides, this treatment also resulting, according to the UV spectra, in ionization. If, however, no ionization takes place, such as in the N-methylation product of (IIa) (IV), obtained as described in [1], the pyrimidine (IV) reacts with sodium ethoxide under mild conditions to give the ethoxy-compound (Va).

*For communication 2, see [1].

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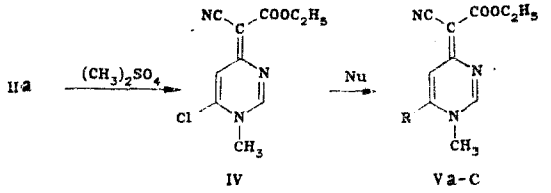
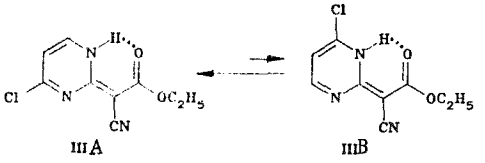


V a $R=OC_2H_5$; b $R=N(CH_3)_2$; c $R=NC_5H_{10}$; $Nu=C_2H_5ONa$, $NH(CH_3)_2$, NHC_5H_{10}

Replacement of one of the chlorines in 2,4-dichloropyrimidine (Ib) and its 6-phenyl derivative (Ic) can give two isomeric products by the replacement of the chlorine in the 2- or 4-position. We have determined the proportions of these isomers in the reaction of 2,4-dichloropyrimidine (Ib) with sodiocyanoacetic ester in dimethoxyethane. According to TLC, the reaction mixture following neutralization contains the two isomeric products (IIb) and (III), which were separated on a silica gel column in the pure state.

The IR, UV, and PMR spectra of (IIa-c) and (III) (Tables 2 and 3) show that in the solid state and in solution in solvents of low polarity these compounds exist in the ylidene tautomeric form with an intramolecular hydrogen bond, as shown by the presence in the IR spectrum of absorption for the carbonyl group chelated by an intramolecular hydrogen bond at < 1700 cm^{-1} and very strong $\nu_{C\equiv N}$ absorption at 2200-2210 cm^{-1}, in the UV spectra of a long-wavelength absorption maximum with $\lambda > 320$ nm, and in the PMR spectrum of signals for the NH...O protons at $\sim$ 13 ppm.

The greatest difference between the isomeric compounds (IIb) and (III) is seen in the PMR spectra in the region of signals for the heteromatic protons. In the case of (IIb), the signals for 5- and 6-H are seen as two doublets at 6.98 and 7.82 ppm. The structure of pure (III) confirms that in $CDCl_3$ it has the tautomeric structure (IIIA), the possible ylidene tautomer (IIIB) being virtually absent, since the signal for 5-H is seen as a doublet at 6.53 ppm, and that for 6-H is further split by the adjacent NH group with a J_{6-NH} value similar to that of J_{5-6}, appearing as a triplet at 7.72 ppm (cf. [3]). The ratio of isomers (IIb)/(III), as found by PMR, is $\sim$ 7:1.



Hence, the preferred product in the reaction of 2,4-dichloropyrimidine with sodiocyanoacetic ester, as in the reaction with amines in polar solvents [4], is the product of replacement of the 4-chlorine atom. It is known [5] that the introduction of weak donor substituents into the 6-position does not affect the selectivity of substitution of the chlorine atoms in 2,4-dichloro-pyrimidines in polar solvents. Therefore, by analogy with the results obtained in the reaction of (Ib) with sodiocyanoacetic ester, we assign the structure (IIc) to the monosubstitution product of 6-phenyl-2,4-dichloropyrimidine (Ic). The instability of ylidene derivatives of 2-chloropyrimidines is noteworthy, especially that of (IIb). Its preparation required milder reaction conditions (see Experimental), and on standing in air for a few days it was almost completely converted into a chlorine-free compound.

In the reaction of the chloro-compounds (IIa-c) with amines and hydrazine, it would be expected, firstly, that nucleophilic replacement of halogens as in vinyl substitution in halo-derivatives of ylidenemalononitriles [6] would occur, and secondly, reaction of the nucleophiles with the ester group side chain and the exocyclic C=C bond. In fact, in the chloro-derivatives (IIa-c) chlorine was replaced quantitatively by dimethylamino-, piperidino-, or hydrazino-groups on heating in ethanol or DMF with the appropriate nucleophile. Comparing the reaction conditions and the time required for 50% reaction of these compounds with other substituted chloropyrimidines [7], it will be seen that they are similar to the nucleophilic substitution conditions for chloropyrimidines containing weakly-donating alkyl substituents. The ester groups in (IIa-c) do not react with amines or hydrazine as a result of deactivation due to conjugation of the carbonyl group in $COOC_2H_5$ with the NH group in the β-aminocarbonyl system (cf. [8]). The influence of the intramolecular CO...NH hydrogen bonding appears to

TABLE. Properties of Pyrimidines (II-X)

Compound	mp, °C (from ethanol)	Found, %				Empirical formula	Calculated, %			
		C	H	N	Cl (Br)		C	H	N	Cl (Br)
IIb	146—150	47,6	3,85	18,4	16,2	$C_9H_8ClN_3O_2$	47,9	3,67	18,6	15,7
IIc	160—163	59,9	4,12	13,9	11,8	$C_{16}H_{12}ClN_3O_2$	59,7	4,01	13,9	11,7
IId	149—153	54,5	6,27	23,2		$C_{11}H_{14}N_4O_2 \cdot 0,5\ H_2O$	54,3	6,21	23,0	
IIe	149—151	61,4	6,59	20,5		$C_{14}H_{18}N_4O_2$	61,3	6,61	20,4	
IIf	230—233	48,8	5,04	32,0		$C_9H_{11}N_5O_2$	48,9	5,01	31,7	
IIg	149—151	61,2	6,69	20,6		$C_{14}H_{18}N_4O_2$	61,3	6,61	20,4	
IIh	216—221	65,4	5,98	18,3		$C_{17}H_{18}N_4O_2$	65,8	5,85	18,0	
IIi	225—230 (decomp.)	69,0	6,38	16,4		$C_{20}H_{22}N_4O_2$	68,6	6,33	16,0	
IIj	255 (decomp.)	61,0	4,95	23,4		$C_{15}H_{15}N_5O_2$	60,6	5,09	23,6	
III	185—192	47,8	3,49	18,8	16,1	$C_9H_8ClN_3O_2$	47,9	3,67	18,6	15,7
IV	260—262	49,7	4,20	17,5	14,7	$C_{10}H_{10}ClN_3O_2$	50,1	4,21	17,5	14,8
Va	193—194	57,9	6,20	17,0		$C_{12}H_{15}N_3O_3$	57,8	6,07	16,8	
Vb	202—203	58,0	6,85	23,0		$C_{12}H_{16}N_4O_2$	58,0	6,49	22,6	
Vc	205—207	63,0	7,13	19,3		$C_{15}H_{20}N_4O_2$	62,5	6,99	19,4	
VI	230—235	61,5	2,74	21,6	14,1	$C_{13}H_7ClN_4$	61,3	2,77	22,0	13,9
VII	238—244	55,4	5,04	39,5		$C_{13}H_{14}N_8$	55,3	5,00	39,7	
VIII	240—249	71,1	5,67	23,0		$C_{18}H_{17}N_5$	71,3	5,64	23,1	
IXa	218—220	37,6	2,45	16,3	(31,1)	$C_8H_6BrN_3O_2$	37,5	2,36	16,4	(31,2)
Xa	205—208	46,0	5,04	16,2	(22,6)	$C_{13}H_{17}BrN_4O_2$	45,8	5,02	16,4	(22,5)
Xb	185—189	47,3	5,41	15,6	(22,6)	$C_{14}H_{19}BrN_4O_2$	47,3	5,39	15,8	(22,4)

TABLE 2. UV Spectra of (II-X)

Compound	λ_{max}, nm (lg ε) in ethanol	Compound	λ_{max}, nm (lg ε) in ethanol
IIa	310 (4,30), 350 pl. (3,92)	III	306 (4,46), 380 (3,38)
IIaNa salt	325 (4,15)	IV	354 (4,49)
IIb	340 (4,25)	Va	348 (4,52)
IIc	234 (4,08), 300 (4,27), 3,76 (4,18)	Vb	275 (3,83), 366 (4,55)
		Vc	276 (3,86), 368 (4,66)
IIc Na salt	236 (4,27), 263 (4,11), 310 (4,17), 364 (4,08)	VI	260 (4,24), 315 (4,37), 367 (4,09)
IId	253 (4,24), 275 (4,20), 333 (4,68)	VII	252 (4,36), 350 (4,22)
		VIII	247 (4,43), 288 (4,23), 357 (4,35)
IIe	266 (4,23), 278 (4,19), 336 (4,61)	IXa	307 (4,61) 398 (3,46)
IIf	253 (4,11), 328 (4,59)	IXb	303 (4,78), 390 (3,60)
IIg	276 (4,04), 367 (4,39)	Xa	233 (4,19), 314 (4,60)
IIh	280 (4,48), 395 (4,35)	Xb	233 (4,12), 315 (4,54)
IIi	281 (4,20), 397 (4,30)	Xc	235 (4,08), 314 (4,46)
IIj	282 (4,54), 394 (4,45)		

be small, since the N-methyl derivative (IV) displays similar properties, affording products of the replacement of chlorine (Vb, c) without reaction of the ester group in the side chain.

Under our reaction conditions, in the case of (IIa-c) TLC failed to reveal the presence of other products such as those of reaction of the nucleophiles at the external C=C bond, the reactivity of which is less than that of other ylidenemalonotriles [9].

The elemental composition and structures of the pyrimidines (IId-j) and (Va-c) were confirmed by analysis and their spectral properties (Tables 1-3). According to the UV, IR, and PMR spectra, (IId-j) exist in the ylidene tautomeric form with intramolecular hydrogen bonding, as in the original chloro-compounds (IIa-c).

Reaction of the dichloropyrimidine (Ic) with sodiomalonitrile also affords the product of substitution of a single chlorine atom, this probably possessing, by analogy with (IIb, c) the ylidene structure (VIA $\rightleftarrows$ VIB). The spectral data obtained for this compound do not enable a further chloice to be made between these two tautomeric forms.

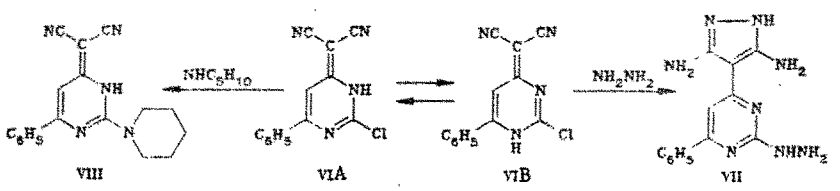


TABLE 3. PMR Spectra of (II-X)

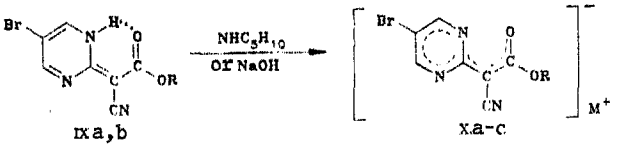
Compound	Solvent	Chemical shifts, δ, ppm (J, Hz)				
		2-H [4-H or 6-H]	5-H and Ar	CH_2CH_3*, q	CH_2CH_3*, t	Other protons
IIa	$CDCl_3$	8,12, br. s	7,13, s	4,28	1,32	13,8 (br. s, NH)
IIb	$CDCl_3$	[7,82, d] (6,0)	6,98, d (6,0)	4,30	1,35	13,9 (br. s, NH)
IIc	$CDCl_3$	—	7,28, s; 7,38—7,60; 7,88—8,06, m	4,25	1,28	13,7 (br. s, NH)
IId	$CDCl_3$	7,97, d (4,0)	5,84, s	4,15	1,22	3,10 [s, $N(CH_3)_2$]; 10,5 (NH)
IIe	$CDCl_3$	7,95, d (4,0)	5,95, s	4,15	1,25	1,65 and 3,60 (m, NC_5H_{10}); 13,30 (br. s, NH)
IIf	$CDCl_3$—DMSO-D_6	8,22, s	6,25, s	4,18	1,28	8,98 and 4,55 (s, $NHNH_2$); 12,90 (br. s, NH)
IIg	$CDCl_3$	[7,85, d] (6,0)	6,30, d (6,0)	4,25	1,33	1,70 and 3,68 (m, NC_5H_{10}); 13,63 (br. s, NH)
IIh	$CDCl_3$	—	6,75, d; 7,30—7,52; (1,3) 7,92—8,08, m	4,20	1,30	3,23 [s, $N(CH_3)_2$]; 10,25 (br. s, NH)
IIi	$CDCl_3$	—	6,75, s; 7,30—8,10, m	4,20	1,30	1,68 and 3,70 (m, NC_5H_{10}); 13,50 (br. s, NH)
IIj	DMSO-D_6	—	6,55, s; 7,30—8,15, m	4,15	1,20	3,35 and 9,65 (s, NH_2NH_2)
III	$CDCl_3$	[7,72, t] (6,5; 6,5)	6,53, d (6,5)	4,25	1,30	13,10 (br. s, NH)
IV	$CDCl_3$	8,15, s	8,05, s	4,25	1,32	3,70 (s, N—CH_3)
	DMSO-D_6	8,50, s	8,00, s	4,10	1,20	3,70 (s, N—CH_3)
Va	$CDCl_3$	8,52, s	7,45, s	4,15	1,30	1,48 t and 4,28 q (OC_2H_5*); 3,62 (s, N—CH_3)
Vb	$CDCl_3$	8,28, s	7,52, s	4,15	1,28	2,85 [s, $N(CH_3)_2$]; 3,65 (s, N—CH_3)
Vc	$CDCl_3$—DMSO-D_6	8,22, s	7,55, s	4,18	1,30	1,65 and 3,02 (m, NC_5H_{10}); 3,58 (s, N—CH_3)
VI	$CDCl_3$	—	6,78, s; 7,35—7,90, m	—	—	—
VII	DMSO-D_6	—	7,19, s; 7,37—7,41; 8,07—8,12, m	—	—	5,44 (br. s, NH_2 and NH); 7,51 (t, **NH**NH_2)
VIII	DMSO-D_6	—	6,29, s; 7,32—7,62, m	—	—	1,63 and 3,67 (m, NC_5H_{10}); 10,87 (br. s, NH)
IXa†	$CDCl_3$	[7,88, d; 8,64, d] (3,0) (3,0)	—	—	—	3,82 (s, OCH_3); 13,83 (br. s, NH)
Xa	DMSO-D_6	[8,27, s]	—	—	—	3,44 (s, OCH_3); 1,59 and 3,02 (br. m, $H_2\overset{+}{N}C_5H_{10}$); 3,96 (br. m, $\overset{+}{H}_2NC_5H_{10}$)
Xb	DMSO-D_6	[8,25, s]	—	3,92	1,15	6,50—7,50 (br. m, $H_2\overset{+}{N}C_5H_{10}$); 1,59 and 3,02 (br. m, $\overset{+}{H}_2NC_5H_{10}$)
Xc	DMSO-D_6	[8,34, s]	—	4,01	1,21	—

*J values ≈ 7.0-7.5 Hz.

†For PMR spectrum of (IXb), see [3].

Reaction of (VI) with hydrazine also takes place at the tautomeric side chain, as described for pyridines and pyridazines [10, 11]. The product obtained (VII) contains a hydrazino-group and a diaminopyrazole substituent. The spectral properties of this compound are in good agreement with those reported previously [11, 12]. In the reaction of (VI) with piperidine, only one chlorine atom is replaced to give the piperidino-compound (VIII).

In the reaction of 5-bromo derivatives of dihydro-2-pyrimidinylidenecyanoacetic esters (IXa, b) with amines such as dimethylamine and piperidine in nonpolar solvents at room temperature, salts are obtained. The piperidinium salts (Xa, b) are stable to recrystallization from anhydrous ethanol and DMF. The structure of (Xa, b) are confirmed by comparing their IR, UV, and PMR spectra with those of the salt obtained from (IXb) by treatment with alcoholic sodium hydroxide (Tables 2, 3). On heating (IXa, b) with piperidine in DMF, resinification occurs.



IXa,b Xa-c

IX, X a R=CH_3, b R=C_2H_5; $M^+=H_2\overset{+}{N}C_5H_{10}$; Xc R=$C_2H_5$; $M^+=Na^+$

EXPERIMENTAL

IR spectra were obtained on a UR-20 spectrophotometer in KBr disks; UV spectra on a Specord UV-VIS (sample concentration 10^{-4} mole/liter) in ethanol; and PMR spectra on a Varian A-56/60 instrument for 7-10% solutions, and a Bruker HX-90 (sample concentration 3-7%) in the impulse mode followed by Fourier transformation.

The course of the reactions and the purities of the products were followed by TLC on Silufol UV-254 plates in the solvent system chloroform–ethanol, 10:1, visualized in UV. The properties of the compounds obtained are given in Tables 1-3.

2,4-Dichloropyrimidine (Ib) was obtained as described in [3], 6-phenyl-2,4-dichloropyrimidine (Ic) as described in [14], 4-chloro-1,6-dihydro-6-pyrimidinylidenecyanoacetic ester (IIa) as described in [2], and 5-bromo-1,2-dihydro-2-pyrimidinylidenecyanoacetic ester (IXb) as described in [3].

Reaction of 2,4-Dichloropyrimidine (Ib) with Sodiocyanoacetic Ester. To a suspension of 3.3 g (70 mmole) of a 50% dispersion of NaH on oil in 30 ml of dimethoxyethane and 2 ml of DMF was added gradually, dropwise, 7.5 ml (70 mmole) of ethyl cyanoacetate followed after 0.5 h by 3.7 g (25 mmole) of (Ib). The mixture was stirred until (Ib) was no longer present (TLC, ∿ 16-18 h) at room temperature. After removal of the dimethoxyethane under reduced pressure on a rotary evaporator, the oily residue was treated with 5 ml of acetic acid and 3-5 drops of conc. hydrochloric acid, to give a thick yellow suspension. This was filtered, and the solid washed on the filter with 50 ml of water and 2 ml of ethanol to give 3.8 g (68%) of product, mp 140-145°C. TLC showed the presence of two spots, one with R_f 0.3 (bright yellow), and the other with R_f 0.7 (grayish-violet). After separation on a column (30 × 500 mm) of silica gel L 100/160 (Chemapol), elution with a mixture of chloroform and ethanol (10:1), and recrystallization from ethanol there were obtained 2.5 g (45%) of greenish-white IIb) and 0.63 g (11%) of bright yellow (III).

2-Chloro-4-phenyl-1,6-dihydro-6-pyrimidinylidenecyanoacetic ester (IIc) was obtained similarly to (IIb), from (Ic) and sodiocyanoacetic ester in DMF, yield 70%.

4-Chloro-1-methyl-1,6-dihydro-6-pyrimidinylidenecyanoacetic ester (IV) was obtained by methylating the sodium salt (IIa) with dimethyl sulfate in DMF, as described in [1] for N-methylation, yield 85%.

Reaction of (IIa-c) and (IV) with Amines and Hydrazine Hydrate. A mixture of 5 mmole of the chloropyrimidine (IIa-c) or (IV) in 3-5 ml of DMF with 10 mmole of the amine or hydrazine hydrate was warmed to 50-80°C until the starting material was no longer present (TLC) (∿ 2-5 h). The DMF was then removed under reduced pressure on a rotary evaporator, the residue treated with 10 ml of water, and the resulting solid filtered off, washed with 2 × 30 ml of water until neutral, dried, and recrystallized from ethanol (yield 60-80%).

Reaction of (IIa-c) and (IV) with Sodium Ethoxide. To a solution of sodium ethoxide in ethanol [from 0.2 g (9 mmole) of sodium in 8 ml of ethanol] was added 5 mmole of (IIa-c) or (IV) at room temperature, and the mixture stirred for 24 h. After removal of the ethanol, the solid residue was treated with 5 ml of water and neutralized with aqueous HCl (1:1) to pH 7, and the solid filtered off. Compounds (IIa-c) were recovered unchanged, but (IV) gave 80% of the pyrimidine (Va).

2-Chloro-4-phenyl-1,6-dihydro-6-pyrimidinylidenemalononitrile (VI) was obtained as for (IIb), from (Ic) and sodiomalononitrile in DMF, yield 87%.

6-(3',5'-Diamino-4'-pyrazolyl)-2-hydrazino-4-phenylpyrimidine (VII). A mixture of 6 g (24 mmole) of (VI), 8 g (200 mmole) of hydrazine hydrate, and 90 ml of ethanol was heated at 90°C for 18 h. After removal of the ethanol, the solid residue was treated with 10 ml of water, and the solid filtered off, washed with 3 × 20 ml of water, and dried to give 3 g (44%) of (VII).

2-Piperidino-4-phenyl-1,6-dihydro-6-pyrimidinylidenemalononitrile (VIII). A mixture of 2.9 g (10 mmole) of (VI) and 1.7 g (20 mmole) of piperidine in 50 ml of ethanol was heated at 90°C until the starting material (VI) was no longer present (∿ 8 h). The product (VIII) was isolated as described for (VII), yield 2.9 g (86%).

The methyl ester (IXa) was obtained as for (IXb), from 5-bromo-2-chloro-pyrimidine and methyl cyanoacetate, yield 80%.

Piperidinium Salt of 5-Bromo-1,2-dihydro-2-pyrimidinylidenecyanoacetic Ester (Xb). Piperidine (1 ml, 10 mmole) was added to 0.27 g (1 mmole) of (IXb), and the mixture stirred for 10 min at room temperature. It was then diluted with 20 ml of dry light petroleum, and the solid which separated was filtered off, washed with 20 ml of dry light petroleum, and dried to give 0.31 g (86%) of (Xb). When (IXb) was reacted with an equimolar amount of piperidine in chloroform or dimethoxyethane, the yield of (Xb) was 70%. When (IXb) was heated in dimethoxyethane or DMF, it resinified.

The Piperidinium salt of (Xa) was obtained as for (Xb), from the ester (IXa) and piperidine, yield 90%.

Sodium Salt of Ethyl 5-Bromo-1,2-dihydro-2-pyrimidinylidenecyanoacetate (Xc). To a solution of 0.27 g (1 mmole) of (IXb) in 10 ml of ethanol was added a solution of 0.04 g (1 mmole) of NaOH in 10 ml of ethanol. The mixture was cooled, and the solid which separated was filtered off an recrystallized from ethanol to give 0.25 g (80%) of (Xc).

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