SYNTHESIS OF SUBSTITUTED 2-AMINO-3-CYANOPYRROLES

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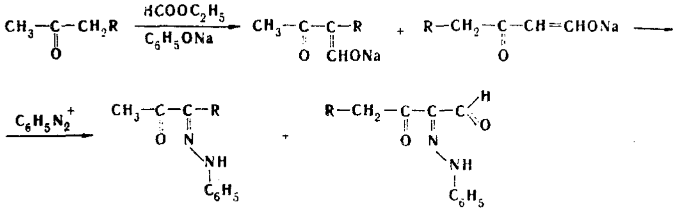
A method was worked out for the preparation of substituted 2-amino-3-cyanopyrroles from N-acyl-α-amino ketones and malononitrile. Acyl derivatives of 2-amino-3-cyanopyrroles were obtained. A number of Mannich bases were synthesized by aminomethylation of 2-acetamido-3-cyano-4-phenylpyrrole.

In a previous communication [1] we described the synthesis of 2-amino-3-cyano-4,5,6,7-tetrahydroindole from malononitrile and 2-acetamidocyclohexanone.

In the present research we have shown that N-acetyl-α-amino ketones with an open aliphatic chain, which we obtained by reductive acetylation of monoarylhydrazones of acyclic α-diketones, can be sucessfully used for the synthesis of substituted aminopyrroles. The ketones were initially formylated with ethyl formate for the synthesis of these monophenylhydrazones, and the resulting formyl derivatives were coupled, without isolation, with benzenediazonium chloride. As a result, we obtained monophenylhydrazones of dicarbonyl (Ia-c) and tricarbonyl (IIa-c) compounds. Consequently, formylation of methyl alkyl ketones proceeds both at the methylene and methyl groups. The percentage compositions of the components were determined by gas –liquid chromatography (GLC), and it was shown that the yields of monophenylhydrazones of dicarbonyl compounds (Ia-c) decrease as the length of the side chain of the ketone increases and that the yields of monophenylhydrazones of tricarbonyl compounds (IIa-c) increase correspondingly (see Table 1).



Ia-c IIa-c

I, II a $R = CH_3$; b $R = C_2H_5$; c $R = C_3H_7$

The IR spectra of Ia-c (in mineral oil) contain the absorption band of an NH group at 3260-3280 cm^{-1} and the band of a conjugated carbonyl group at 1650 cm^{-1}. However, the spectra of IIa-c in mineral oil, and in dilute CCl_4 solutions do not contain the absorption band of a secondary amino group, and this is apparently associated with the formation of an intramolecular hydrogen bond with the aldehyde carbonyl group [2]. Two bands corresponding to aldehyde (1635-1660 cm^{-1}) and ketone (1660-1670 cm^{-1}) groups are observed in the region of absorption of carbonyl groups. Singlets of the protons of the NH group of Ia-c are found at 8 ppm in the PMR spectra, whereas the corresponding singlets in the spectra of IIa-c are shifted to weaker field (14.5 ppm), and this also confirms the presence of a hydrogen bond.

The UV spectra of IIa-c contain three absorption maxima at 236, 295, and 340 nm, but the absorption maximum at 295 nm vanishes in the spectra of IIa-c, and the other two maxima are shifted by 12-30 nm, respectively, to the long-wave region.

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TABLE 1. Monophenylhydrazones of Di- and Tricarbonyl Compounds

Compound	mp, °C	Empirical formula	Found, %			Calc., %			PMR spectra							IR spectra, cm⁻¹		UV spectra		Yield, %
			C	H	N	C	H	N	CH_3	α-CH_2	β-CH_2	CH_3CO	C_6H_5	HCO	NH	ν_{NH}	ν_{CO}	λ_{max}, nm	lg ε	
Ia	135—136*	$C_{10}H_{12}N_2O$	—	—	—	—	—	—	1,96s,†	—	—	2,45	6,81—7,32m	—	8,0s	3260	1650	236 296 340	4,08 3,87 4,38	94
Ib	118—119*	$C_{11}H_{14}N_2O$	—	—	—	—	—	—	1,03t	2,97q	—	2,47	6,55—8,85m	—	8,31s	3260	1650	236 296 342	4,11 3,86 4,42	73
Ic	108—109	$C_{12}H_{16}N_2O$	70,3	7,8	13,8	70,6	7,8	13,7	0,95t	2,67t	1,11—1,5m	2,47	6,45—7,2m	—	8,22s	3280	1650	236 295 342	4,08 3,84 4,37	85
IIa	82—83	$C_{11}H_{12}N_2O_2$	64,6	6,0	13,8	64,7	5,9	13,7	1,17t	2,94q	—	—	7,04—7,58m	10,06	14,59	—	1648 1670	248 372	4,10 4,26	5
IIc	54—55	$C_{13}H_{16}N_2O_2$	67,1	6,8	12,3	67,2	6,9	12,1	0,92	2,88t	1,15—1,87m	—	7,01—7,4m	10,06	14,58	—	1670 1600	248 374	4,10 4,32	14,5
IIb	64—65	$C_{12}H_{14}N_2O_2$	65,8	6,2	12,8	66,0	6,4	12,8	1,02t	2,86t	1,55—1,97m	—	7,04—7,58m	10,05	14,53	—	1635 1660	248 372	3,96 4,24	26
III	92—93	$C_{13}H_{18}N_2O_2$	71,8	8,4	13,1	71,5	8,3	12,8	—	—	—	—	—	—	—	3280	1650	265 296 342	4,04 3,79 4,32	81

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* According to the literature data, Ia has mp 133° and Ib has mp 116-117°.

†Protons of the α and γ methylene groups; here and subsequently, s is singlet, t is triplet, q is quartet, and m is multiplet.

TABLE 2. N-Acetyl-α-amino Ketones

Compound	R	R′	bp, °C (mm)	Empirical formula	Found, %			Calc., %			IR spectra, cm⁻¹				Yield, %
					C	H	N	C	H	N	ν_{NH} liq.	ν_{NH} CCl_4	ν_{CO} liq.	ν_{CO} CCl_4	
IVa	CH_3	CH_3	102— —106 (2)*	$C_6H_{11}NO_2$	—	—	—	—	—	—	—	—	—	—	77
IVb	CH_3	C_2H_5	124 (4)	$C_7H_{13}NO_2$	58,4	9,0	10,1	58,7	9,1	9,8	3290 3080	3420	1720 1650 1535 1290	1720 1680 1370	72
IVc	CH_3	C_3H_7	125 (3)	$C_8H_{15}NO_2$	61,2	9,6	9,2	61,1	9,6	8,9	—	—	—	—	59
IVd	C_2H_5	CH_3	115 (4)	$C_7H_{13}NO_2$	59,0	9,1	9,7	58,7	9,1	9,7	3280 3080	3420	1720 1650 1535 1290	1720 1680 1370	71
IVe	C_3H_7	C_2H_5	126 (2)	$C_9H_{17}NO_2$	63,3	10,1	7,8	63,1	10,0	8,1	3280 3060	3420	1720 1650 1535 1370	1720 1670 1370	60

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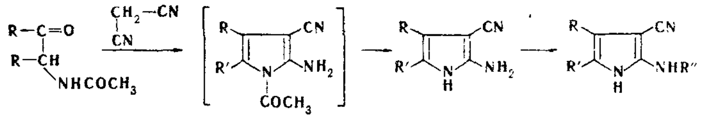
*According to [10], this compound has bp 102-106° (2 mm).

The reductive acetylation of Ia-c, 3,4-heptadione 3-phenylhydrazone (III), and the previously described 2,3-pentadione 2-phenylhydrazone [3] with zinc dust in glacial acetic acid in the presence of acetic anhydride and sodium acetate gives N-acetyl-α-amino ketones (IVa-e). The IR spectra of IVb-e (liquid films) contain a strong broad band at 2380 cm^{-1}, which can be assigned to the trans form of amides that form an intermolecular hydrogen bond [4]. In addition, there is a weaker band at 3080 cm^{-1}. It is also associated with NH vibrations. In dilute CCl_4 solution both of these bands vanish, and an absorption band appears at 3420 cm^{-1}. Four bands – ketone (1720 cm^{-1}), amide I band (1650 cm^{-1}), amide II band (1535 cm^{-1}), and amide III band (1290 cm^{-1}) – are observed in the region of absorption of carbonyl groups. In dilute CCl_4 solution the amide I and III bands are shifted to the long-wave region by 30-80 cm^{-1}, respectively, whereas the amide II band vanishes.

Substituted 2-amino-3-cyanopyrroles were obtained by reaction of N-acetyl-α-aminoketones with malononitrile in the presence of both catalytic and equimolar amounts of piperidine.

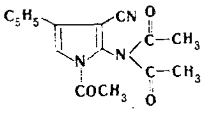


IVa-e V VIa-f VIIa-h

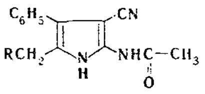
The condensation proceeds through a step involving the formation of N-acetyl-2-amino-3-cyanopyrroles. However, we were able to isolate only one of them – N-acetyl-2-amino-3-cyano-4,5-dimethylpyrrole (V) – which was obtained by reaction of malononitrile with 3-acetamido-2-butanone. Only 2-amino-3-cyanopyrroles (VIa-f) were obtained in all other cases.

The method described above enabled us to obtain 2-amino-3-cyano-4-phenylpyrrole (VIf) from 1-acetamidoacetophenone [5] and malononitrile. In this case, the formation of a substituted piperazine [6] that is observed when ω-aminoacetophenone hydrochloride and malononitrile are used for the reaction [7] is excluded.

The acetylation of aminopyrroles VIa-f with acetic anhydride gives 2-acetamido derivatives of pyrrole (VIIa-f), whereas a triacyl derivative (VIII) is also formed in the reaction of acetic anhydride with pyrrole VIf in pyridine.



VIII



IX a-e

IX a R = $-N(CH_3)_2$; b R = $-N(C_2H_5)_2$; c R = $-N(C_4H_9)_2$; d R = morpholino; e R = piperidino.

Benzamido (VIIg) and tosylamino (VIIh) derivatives, respectively, were obtained by acylation of VIf with benzoyl chloride in dioxane and with p-toluenesulfonyl chloride in pyridine.

The signals of the protons of the acetyl groups of VIII appear in the PMR spectrum as two singlets at 2.32 (6H) and 2.57 (3H) ppm, as compared with 2.27 ppm (3H) in the spectra of VIIf, which we assigned to the protons of the $COCH_3$ group attached to the amino group in the 2 position. The singlets at 10.66 and 11.58 ppm belong to the protons of the NH groups in the 2 and 1 positions, respectively. The frequencies of the vibrations of the CN group in the IR spectra of VIf, VIIf, and VIII are found at 2200, 2210, and 2220 cm^{-1}, respectively. Their intensities decrease sharply from VIf to VIII, and this indicates weakening of the conjugation of the nitrile group with the other multiple bonds.

The aminomethylation of VIIf proceeds under the conditions described for the aminomethylation of 2-aminothiophenes [8]. The dialkylaminomethyl group enters the substituent-free 5 position of pyrrole. A hydrogen atom of the amide group is not replaced. The disappearance of the signal at 6.70 ppm, which is affiliated with the proton in the 5 position of pyrrole, the appearance of a signal at 3.36 ppm of the CH_2 group, and the shift of the signals of the NH group at 9.66 and 10.77 ppm to stronger field as compared with the signals of the NH groups of starting VIIf confirm this. Absorption bands at 3360 and 3240-3280 cm^{-1}, which correspond to the stretching vibrations of the amino group, at 2200 cm^{-1}, which corresponds to a conjugated nitrile group, and at 1680 cm^{-1}, which corresponds to a carbonyl group, are observed in the IR spectra of IXa-e in all cases. The UV spectra of IXa-e contain only one maximum at 284-286 nm (log ε 4.15-4.30), and the second maximum of starting VIIf at 226 nm is converted to a shoulder (at 220 nm).

EXPERIMENTAL

The IR spectra of mineral oil suspensions, thin films, and CCl_4 solutions of the compounds were recorded with a Perkin–Elmer spectrometer. The UV spectra of ethanol solutions of the compounds were recorded with an EPS-3T spectrometer. The PMR spectra of deuterochloroform and deuterodimethyl sulfoxide solutions of the compounds were recorded with a JNM-4H-100, G 60 H spectrometer with tetramethylsilane as the internal standard. Chromatographic monitoring of the course of the reactions and the purity of the compounds obtained was carried out by thin-layer chromatography (TLC) on Silufol UV-254 plates in the following solvent systems: Acetone–heptane (1 : 1), benzene–methanol (9 : 1), benzene–ethyl acetate (1 : 1), and chloroform.

General Method for the Preparation of Monophenylhydrozones of Dicarbonyl and Tricarbonyl Compounds (a-c and IIa-c). Absolute ethanol [39.9 g (0.8 mole)] was added dropwise with stirring to a suspension of 5.75 g (0.25 g-atom) of sodium metal in 25 ml of toluene, and the mixture was heated until the sodium had dissolved completely. The ethanol was then removed by distillation, and a mixture of 37 g (0.5 mole) of ethyl formate and 0.25 mole of the ketone was added to the cooled residue at such a rate that the temperature of the mixture did not rise above 40°. It was then stirred at room temperature for another hour and allowed to stand overnight. The sodium derivative of the formyl ketone was extracted with water (two 150-ml portions), and the resulting solution was cooled to 0-5° and added to an aqueous solution of benzenediazonium chloride prepared in the usual way from 23.3 g (0.25 mole) of aniline, 75 ml of concentrated HCl, and 17.2 g (0.25 mole) of sodium nitrite at pH 5-6 (effected by the addition of sodium acetate). The precipitated crystals were removed by filtration, washed with cold water, and dried. The composition of the mixture was determined with a Pye-Unicam series 104 chromatograph with a flame-ionization detector and a 2.1 m by 4 mm column filled with a silicone elastomer (100-120 mesh); the carrier gas (nitrogen) flame rate was 30 ml/min, and the column temperature was 250°. The retention times (in minutes) were as follows: Ia 10.25, Ib 10.75, Ic 14.75, IIa 13.75, IIb 16.25, and IIc 20.5. The mixture of monophenylhydrazones of the di- and tricarbonyl compounds was washed with petroleum ether (bp 40-60°), and the residue was recrystallized from methanol to give hydrazones Ia-c. The mother liquors containing petroleum ether and methanol were evaporated to dryness, and the residue was triturated with benzene. The solids were then removed by filtration to give IIa-c.

3,4-Heptadione 3-phenylhydrazone (III) was similarly obtained. Data on the monophenylhydrazones are presented in Table 1.

General Method for the Preparation of N-Acetyl-α-amino Ketones (IVa-e). Zinc dust [40 g (0.6 g-atom)] was added in small protions with vigorous stirring to a heated (to 40°) mixture consisting of 0.2 mole of the monophenylhydrazone of a dicarbonyl compound, 20 g (0.24 mole) of fused sodium acetate, 55 g (0.54 mole) of acetic anhydride, and 200 ml of acetic acid, after which the mixture was stirred and refluxed for 1 h. The sludge was removed by filtration and washed with hot acetic acid, and the acetic acid solution was vacuum evaporated. Water was added to the residue, and the precipitated acetanilide was removed by filtration. The aqueous solution was vacuum evaporated, chloroform was added to the residual light-brown

TABLE 3. Substituted 2-Amino-3-Cyanopyrroles

Compound	R	R′	R″	mp, °C*	Empirical formula	Found, %			Calc., %			IR spectra, cm^{-1}					UV spectra		Yield, %
						C	H	N	C	H	N	$\nu_{NH_2,NH}$	ν_{CN}	ν_{NH}	$\nu_{C=N}$	ν_{CO}	λ_{max}, nm	lg ε	
VIa	CH_3	CH_3	H	163—164†	$C_7H_9N_3$	—	—	—	—	—	—	3360, 3220	2180	1620	1345	—	268	3,83	90
VIb	C_2H_5	CH_3	H	147—149	$C_8H_{11}N_3$	64,5	7,5	28,2	64,4	7,4	28,2	3350, 3230	2190	1620	1350	—	268	3,85	60
VIc	CH_3	C_2H_5	H	160—161	$C_8H_{11}N_3$	64,6	7,6	28,5	64,4	7,4	28,2	3370, 3200	2180	1640	1330	—	266	3,93	89
VId	CH_3	C_3H_7	H	120—121	$C_9H_{13}N_3$	66,2	8,2	25,7	66,2	8,0	25,7	3370, 3140	2200	1620	1345	—	268	3,83	71
VIe	C_3H_7	C_2H_5	H	124—125	$C_{10}H_{15}N_3$	67,9	8,5	23,7	67,7	8,5	23,7	3360, 3240	2190	1620	1345	—	268	2,92	67
VIf	C_6H_5	H		172—173†	$C_9H_{11}N_3$	—	—	—	—	—	—	3390, 3225	2200	1640	1350	—	268	3,85	68
VII	CH_3	CH_3	$COCH_3$	283—284 (dec.)	$C_9H_{11}N_3O$	61,1	6,1	23,8	60,0	6,2	23,7	— 3300 3230 3160	2210	1670	1310	1670	284	3,93	97
VIIb	C_2H_5	CH_3	$COCH_3$	229—230 (dec.)	$C_{10}H_{13}N_3O$	63,1	6,8	22,0	62,8	6,8	21,9	— 3310 3220 3160	2210	1640	1320	1670	284	4,25	93
VIIc	CH_3	C_2H_5	$COCH_3$	201—202 (dec.)	$C_{10}H_{13}N_3O$	63,1	7,0	22,0	62,8	6,8	21,9	— 3310 3260 3100	2200	1640	1320	1670	284	3,89	95
VIId	CH_3	C_3H_7	$COCH_3$	209—210 (dec.)	$C_{11}H_{15}N_3O$	64,5	7,3	20,6	64,4	7,4	20,5	— 3320 3240 3100	2200	1640	1320	1670	287	3,95	87
VIIe	C_3H_7	C_2H_5	$COCH_3$	187—189	$C_{12}H_{17}N_3O$	65,7	7,9	19,0	65,7	7,8	19,1	— 3310 3260 3160	2220	1640	1320	1665	286	4,01	86
VIIf	C_6H_5	H	$COCH_3$	272—273 (dec.)	$C_{13}H_{11}N_3O$	69,6	4,9	18,9	69,3	4,9	18,6	— 3300 3210 3125	2200	1640		1655	225 286	4,62 4,48	97

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* Recrystallized from alcohol.

†According to [7], VIa has mp 163° and VIf has mp 173°.

TABLE 4. 5-Aminoalkyl Derivatives of 2-Acylamino-3-cyano-4-phenylpyrrole

Compound	mp, °C*, base (hydrochloride	Empirical formula	Found, %				Calc., %				Yield, %
			C	H	Cl	N	C	H	Cl	N	
IXa	166—167	$C_{16}H_{18}N_4O$	68,4	6,2	—	19,6	68,1	6,4	—	19,8	82
	(213—214)	$C_{16}H_{18}N_4O \cdot HCl$	—	—	10,8	17,7	—	—	11,1	17,6	
IXb	176—178	$C_{18}H_{22}N_4O$	69,4	7,0	—	18,5	69,6	7,1	—	18,1	82
	(180—181)	$C_{18}H_{22}N_4O \cdot HCl$	—	—	9,7	16,5	—	—	10,0	16,2	
IXc	130—131	$C_{22}H_{30}N_4O$	71,8	8,0	—	15,7	72,1	8,2	—	15,4	69
	(146—150)	$C_{22}H_{30}N_4O \cdot HCl$	—	—	8,7	13,9	—	—	8,8	13,9	
IXd	204—205	$C_{18}H_{20}N_4O_2$	66,6	6,5	—	17,2	66,6	6,2	—	17,3	82
	(190—191)	$C_{18}H_{20}N_4O_2 \cdot HCl$	—	—	9,7	15,7	—	—	9,8	15,5	
IXe	198—199	$C_{19}H_{22}N_4O$	70,8	6,8	—	17,3	70,9	6,9	—	17,4	64
	(190—191)	$C_{19}H_{22}N_4O \cdot HCl$	—	—	9,8	15,7	—	—	9,9	15,6	

*The bases were recrystallized from alcohol, and the hydrochlorides were recrystallized from acetone–methanol–ether.

syrup, and the zinc acetate was removed by filtration. The chloroform was evaporated, and the residue was vacuum distilled. Data on the N-acetyl-α-amino ketones are presented in Table 2.

1-Acetyl-2-amino-3-cyano-4,5-dimethylpyrrole (V). A solution of 1.38 g (0.023 mole) of malononitrile in 15 ml of absolute methanol was added with stirring to 4 g (0.023 mole) of amino ketone IVa in 5 ml of absolute ethanol and 0.01 ml of piperidine, during which the temperature of the mixture rose to 30°. The resulting precipitate was removed by filtration and washed with a small amount of methanol to give 4.1 g (74%) of pyrrole V with mp 166-168° (from dioxane). IR spectrum: 3420, 3300, 3140, 1640 (NH_2); 2200 (CN); 1700 (CO); and 1310 cm^{-1} (C–N). UV spectrum, λ_{max}, nm (log ε): 232 (4.08). Found: C 61.1; H 6.0; N 23.8%. $C_9H_{11}N_3O$. Calculated: C 61.0; H 6.2; N 23.7%. Compounds V and VIa were formed in 33.4 and 52.5% yields, respectively, in the case of an equimolar ratio of IVa and piperidine. 2-Amino-3-cyanopyrroles VIa-f were similarly obtained, but the reaction was carried out at 45-50°. Data on the physical constants and yields of VIIa-f are presented in Table 3.

General Method for the Preparation of Substituted 2-Acetamido-3-cyanopyrroles (VIIa-f). Acetic anhydride (16 ml) was added to 0.022 mole of aminopyrroles VIa-f and the mixture was allowed to stand at room temperature for 15 min. The resulting precipitate was removed by filtration and washed with alcohol. Data on VIIa-f are presented in Table 3.

1-Acetyl-2-diacetamido-3-cyano-4-phenylpyrrole (VIII). A mixture of 5 g (0.0027 mole) of pyrrole VIf, 10 ml of pyridine, and 3 ml (0.03 mole) of acetic anhydride was refluxed for 2 h, after which the mixture was evaporated to dryness, and the residue was washed with methanol. The yield of VIf with mp 273-274° (dec.) was 42%. The methanol mother liquor was evaporated to dryness to give VIII with mp 123-124° (from methanol) in 40% yield. IR spectrum: 2200 (CN); 1740, 1730, and 1710 cm^{-1} (CO). UV spectrum, λ_{max}, nm (log ε): 242 (4.38). PMR spectrum, ppm: 2.32, s, 6H($COCH_3$), 2.57, s, 3H($COOH_3$), 7.3, s, H(5H), and 7.3-7.5, 5H(C_6H_5). Found: C 65.6; H 5.0; N 13.3%. $C_{17}H_{15}N_3O_3$. Calculated: C 65.9; H 4.9; N 13.3%.

2-Benzamido-3-cyano-4-phenylpyrrole (VIIg). A mixture of 1.83 g (0.01 mole) of VIf, 30 ml of dry dioxane, and 3.9 g (0.028 mole) of benzoyl chloride was refluxed for 2 h, after which the solution was evaporated to dryness, and the residue was washed with methanol. The yield of VIIg with mp 267-268°C (dec., from methanol) was 2.43 g (86%). IR spectrum: 3340, 3260, 3220, 3150 (NH); 2220 (CN); and 1650 (CO). Found: C 75.5; H 4.7; N 14.3%. $C_{18}H_{13}N_3O$. Calculated: C 75.2; H 4.6; N 14.6%.

2-Tosylamino-3-cyano-4-phenylpyrrole (VIIh). A 1.05-g (0.0055 mole) sample of p-toluenesulfonyl chloride was added to 0.9 g (0.005 mole) of pyrrole VIf in 15 ml of dry pyridine, after which the mixture was allowed to stand at room temperature for 15 min. It was then poured into water, and the aqueous mixture was extracted with ether. The ether solution was washed successively with water, 10% sulfuric acid, and water and dried with $MgSO_4$. The violet solution was decolorized with charcoal, and the ether was evaporated to give 1.04 g (63%) of a product with mp 155-156° (from ethanol). IR spectrum: 2220 (CN) and 3300 cm^{-1} (NH). Found: C 64.2; H 4.5; N 9.5%. $C_{18}H_{15}N_3O_2$. Calculated: C 64.0 H 4.5; N 9.5%.

General Method for the Preparation of 5-Dialkylaminomethyl-2-acetamido-3-cyano-4-phenylpyrroles (IXa-e). A mixture of 0.01 mole of VIIf, 40 ml of dry dioxane, and 0.02 mole of bis(dialkylamino)methane derivative was refluxed in a flask equipped with a reflux condenser and fitted with Bunsen valve for 3 h. The mixture was then vacuum evaporated to dryness, and the hydrochlorides were obtained from the bases in the usual way. Data on the physical constants and yields of IXa–e are presented in Table 4. PMR spectrum (ppm) for IXa: 2.13, s, 9H($COCH_3$), $(CH_3)_2$, 3.36, s, 2H(CH_2), 9.66, s, H(NH) and 7.34, s, 5H(C_6H_5); for IXd: 2.12, s, 3H($COCH_3$), 2.35 t, 4H[$N(CH_2)_2$], 3.43, s, 2H(CH_2), 3.73, t, 4H[$O(CH_2)_2$], 9.66, s, H($NHCOCH_3$), and 10.77, s, H (NH).

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