

Physical Sciences Section

Pakistan J. Sci. Ind., Res. Vol. 22, Nos. 1-2, February-April 1978

SYNTHESIS AND REACTIONS OF 3-CINNAMOYL PYRIDONES AND AMINONICOTINONITRILES

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(Received April 5, 1977; revised July 1, 1978)

Abstract. As regards study of some of the reactions of 3-cinnamoyl pyridones (II), they were allowed to react with hydrazine hydrate, hydroxylamine hydrochloride, semicarbazide hydrochloride, phenylhydrazine, urea, thiourea, ethyl acetoacetate, ethyl malonate, and acetyl acetone, maleic anhydride, *N*-phenyl and *N-p*-tolylmaleimide. 2-Amino -4, 6-diaryl nicotinitriles (XIa, c and e) on hydrolysis with alcoholic potash gave (XIIa-c). (XIIa-c) were hydrolyzed with KOH in glycerol to (XIIIa-c). (XIIIa-c) on heating with acetic anhydride gave (XIV a-c) which on condensation with anisaldehyde gave (XVa-c). Treatment of (XIVa) with butylamine gave (IV).

Introduction

Structure - activity relationship of some pyridine derivatives was studied by A. Essawy *et al.*⁹ The aim of the present investigation is to synthesize some new pyridine derivatives. Their phytotoxic and stimulant actions were studied.⁹

Results and Discussion

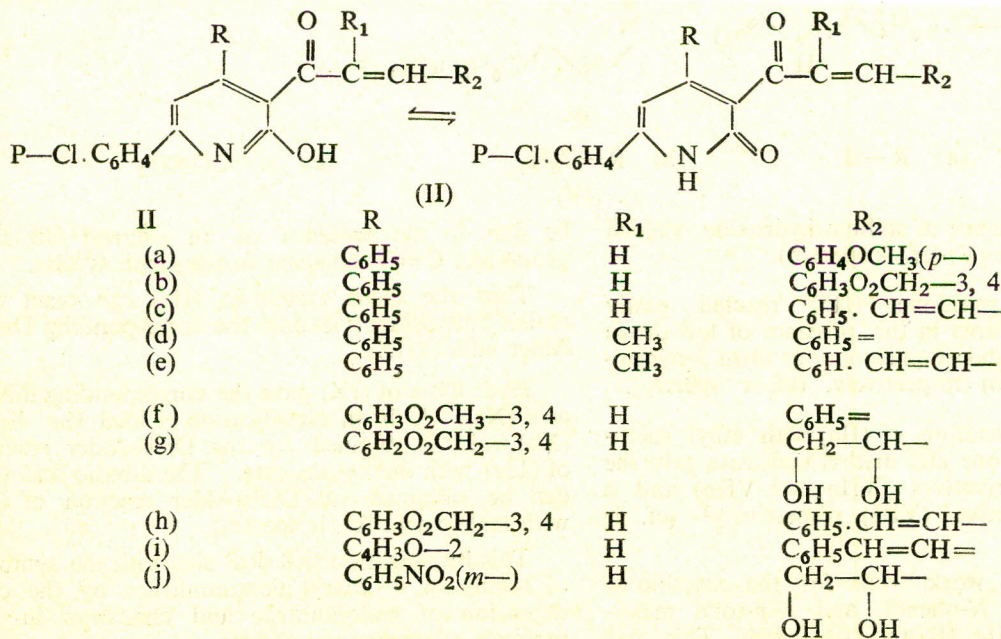
The 3-cinnamoyl pyridone derivatives (IIa-j) were prepared by Claisen - Schmidt condensation of an appropriate aldehyde with the methyl and ethyl ketones (1 a-e) using ethanol as solvent and potassium hydroxide or piperidine as base.^{1,2}

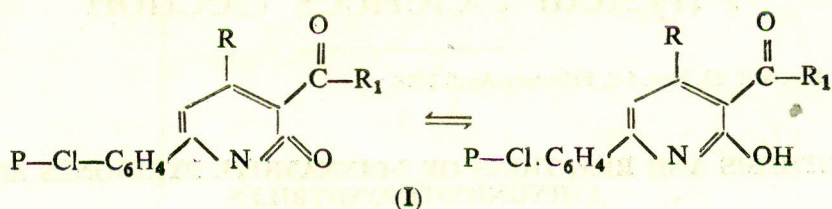
Also the compounds (1a and d) were fused with ketones and diketones namely, diethylketone, ninhydrine and diacetyl at 200° in the presence of anhydrous ZnCl₂ and gave (III a-c).

3-Cinnamoyl pyridones (II) condensed with hydrazine and semicarbazide hydrochloride and gave (IV a-c) respectively (for ir spectra cf. Table 1).

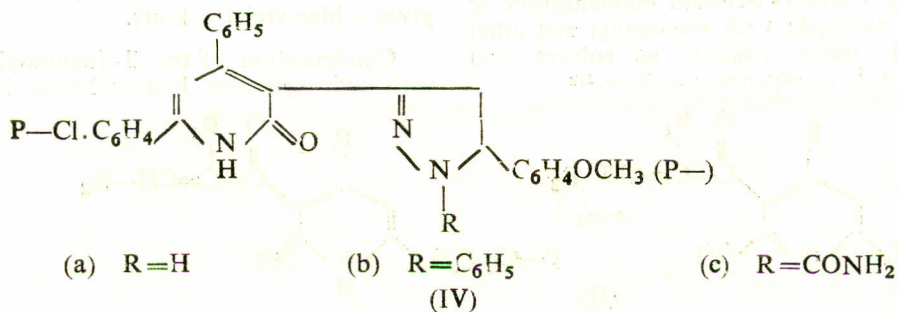
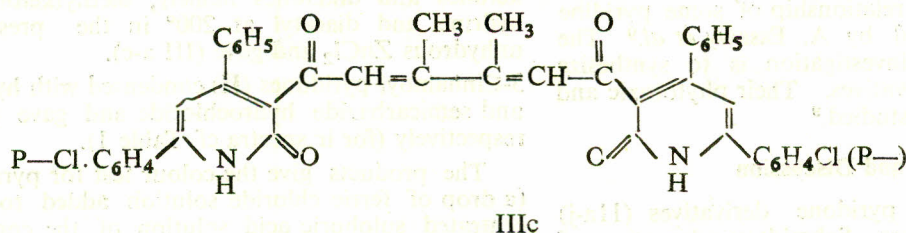
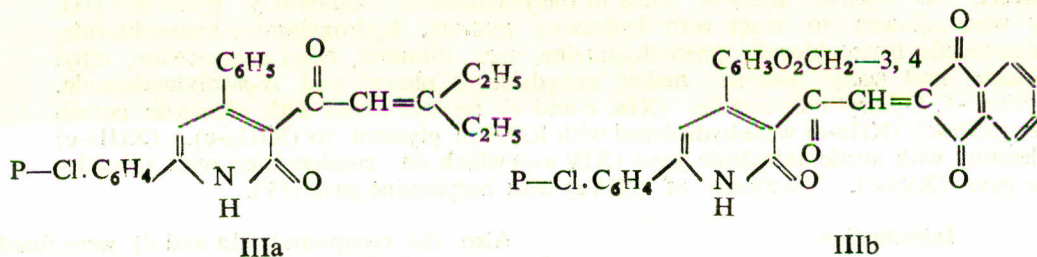
The products give the colour test for pyrazolines (a drop of ferric chloride solution added to a concentrated sulphuric acid solution of the compound gives a blue-violet colour).

Condensation of the 3-cinnamoyl pyridone (II a) with hydroxylamine hydrochloride in boiling ethyl





I	R	R ₁
(a)	C ₆ H ₅	CH ₃
(b)	C ₆ H ₅	C ₂ H ₅
(c)	C ₄ H ₃ O-2	CH ₃
(d)	C ₆ H ₃ O ₂ CH ₂ -3, 4	CH ₃
(e)	C ₆ H ₄ NO ₂ (<i>m</i> -)	CH ₃



alcohol in the presence of sodium hydroxide yielded isoxalone derivative (V) (cf. ir spectra).

3-Cinnamoyl pyridone (IIa) reacted easily with urea and thiourea in the presence of few drops of sulphuric acid and ethyl alcohol to form 3-pyrimidone (VIa and VIb) respectively. (cf. ir spectra).

Michael condensation of (IIa) with ethyl acetate, acetyl acetone and diethyl malonate gave the cyclohexenone derivatives (VIIa and VIIb) and a pyranopyran derivative (VIII) respectively³ (cf. ir and uv spectra).

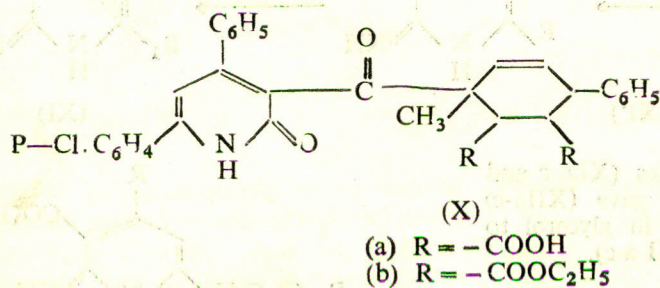
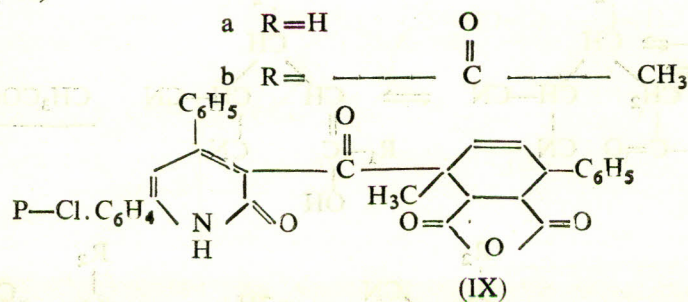
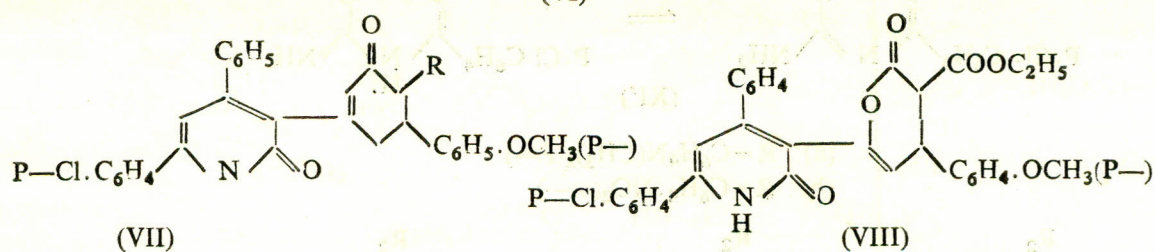
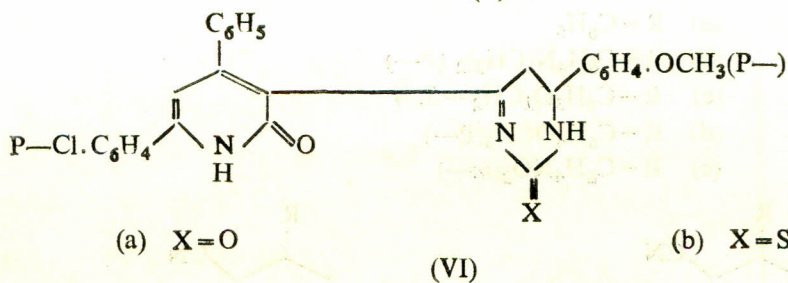
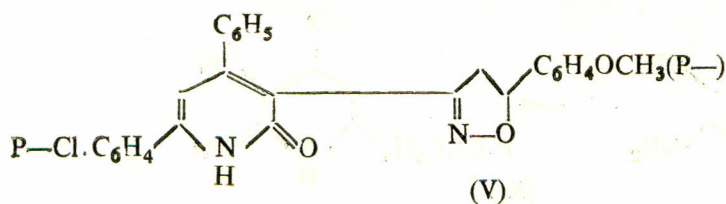
In the present work trials for the reaction of maleic anhydride, *N*-phenyl, and *N*-*p*-tolyl maleimides^{6, 7} with (IIc, IIh and Ili) failed. This may

be due to the presence of an electron attracting group like C=O adjacent to the diene system.

Thus the diene system in (IIe) can react with maleic anhydride, yielding the corresponding Diels-Alder adduct (IX).

Hydrolysis of (IX) gave the corresponding dibasic acid (Xa), which on esterification yielded the diester (Xb) which is obtained by the Diels-Alder reaction of (IIe) with diethyl maleate. The dibasic acid (Xa) can be obtained via Diels-Alder reaction of (IIe) with maleic acid (cf. ir spectra).

This investigation will deal also with the synthesis of 2-amino-4, 6-diaryl nicotinonitriles by the condensation of malononitrile and chalcones in the presence of ammonium acetate.



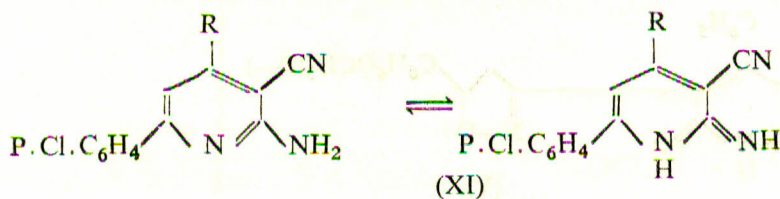
The reaction between malononitrile and chalcones was found to be vigorous and instantaneous. Thus, the condensation was carried out by heating a mixture of malononitrile, chalcone, and ammonium acetate (molar ratio 1:1:0.8) at 150° for 6 hr. Treatment of the reaction mixture with ethanol yielded two parts.

The ethanol insoluble part afforded 2-amino-4, 6, diaryl - nicotinonitriles (XIa-e) and the ethanol soluble part afforded 2-amino 4, 6 - diaryl 1, 2, 3, 4-

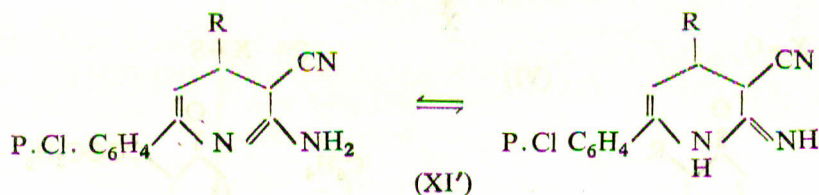
tetrahyronicotinonitriles (XIa-b). (cf. ir spectra).

The mechanism of formation of (XI) and (XI') is summarized in the scheme given below.

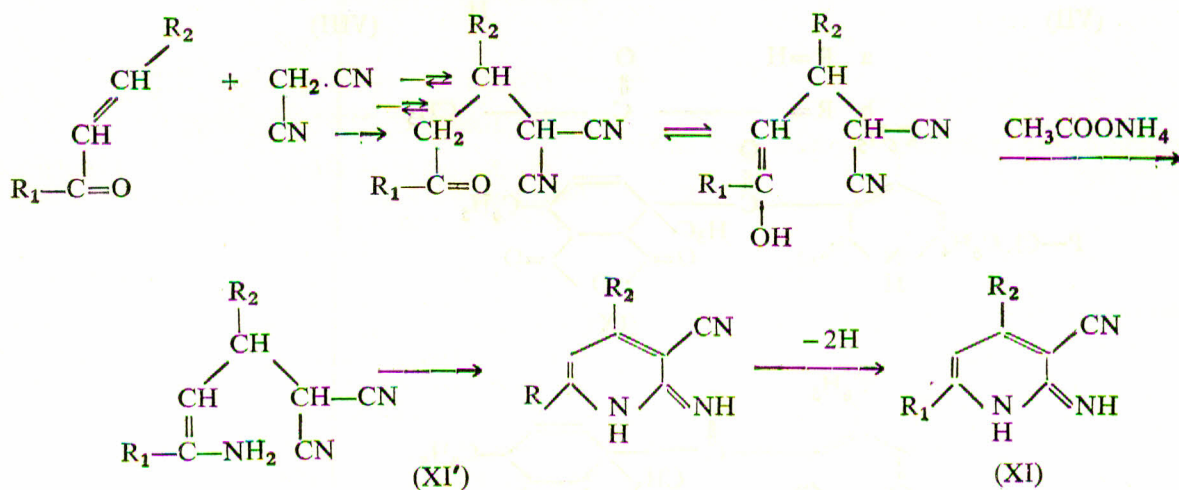
At different conditions, condensation of chalcone with malononitrile in the presence of ammonium acetate at 170° for 15 hr. yielded one product (XI') through the intermediate (XI) which on dehydrogenation yielded the compound (XI'), and this proves the above mechanism.



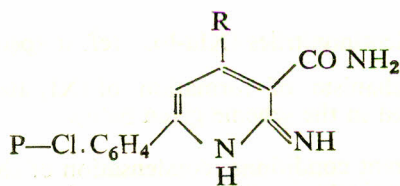
- (a) $\text{R} = \text{C}_6\text{H}_5$
 (b) $\text{R} = \text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2$ (P-)
 (c) $\text{R} = \text{C}_6\text{H}_3\text{O}_2\text{CH}_2$ -3, 4
 (d) $\text{R} = \text{C}_6\text{H}_4\text{OCH}_3$ (P-)
 (e) $\text{R} = \text{C}_6\text{H}_4\text{NO}_2$ (*m*-)



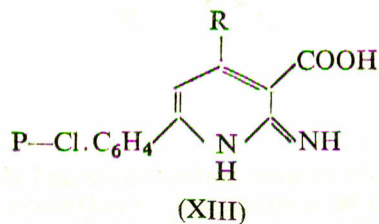
- (a) $\text{R} = \text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2$ (P-)
 (b) $\text{R} = \text{C}_6\text{H}_4\text{NO}_2$ (*m*-)



2-Amino-4, 6-diaryl nicotinonitriles (XIa, c and e) on hydrolysis with alcoholic potash gave (XIIa-c) which were hydrolyzed with KOH in glycerol to 2-amino-4, 6-diarylnicotinic acids (XIII a-c).

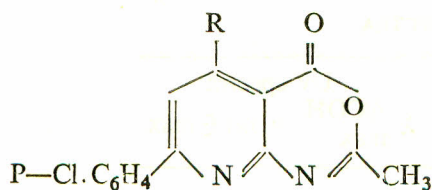


- (a) $\text{R} = \text{C}_6\text{H}_5$
 (b) $\text{R} = \text{C}_6\text{H}_3\text{O}_2\text{CH}_2$ -3, 4
 (c) $\text{R} = \text{C}_6\text{H}_4\text{NO}_2$ (*m*-)



- (a) $\text{R} = \text{C}_6\text{H}_5$
 (b) $\text{R} = \text{C}_6\text{H}_3\text{O}_2\text{CH}_2$ -3, 4
 (c) $\text{R} = \text{C}_6\text{H}_4\text{NO}_2$ (*m*-)

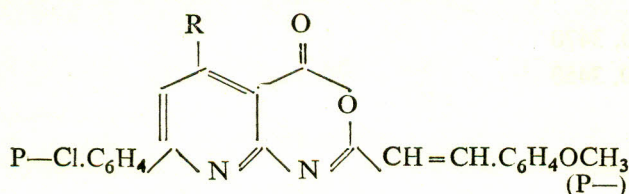
Heating 2-amino-4, 6-diarylnicotinic acids (XIIIa, b and c) with acetic anhydride gave (XIV)⁸ (cf. ir spectra).



(XIV)

- (a) $R = C_6H_5$
 (b) $R = C_6H_3O_2CH_2-3, 4$
 (c) $R = C_6H_4NO_2 (m-)$

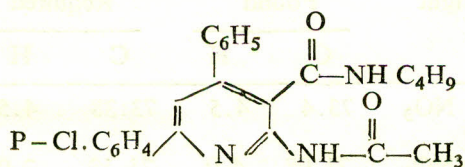
Condensation of (XIVa, b and c) with anisaldehyde by fusion at 170° in the presence of anhydrous zinc chloride yielded (XVa-c) respectively.



(XV)

- (a) $R = C_6H_5$
 (b) $R = C_6H_3O_2-3, 4$
 (c) $R = C_6H_4NO_2 (m-)$

Treatment of (XIVa) with butylamine gave (XVI) (cf. ir spectra).



(XVI)

Experimental

The ir absorption spectra were determined with a Unicam Sp-1200 Spectrometer using KBr wafer technique. The uv absorption spectra were determined by a Unicam Sp - 8000 ultraviolet recording spectrophotometer.

All the melting points reported in the paper are uncorrected.

Condensation of 2-hydroxy-4, 6-diaryl-3-pyridyl alkyl-ketones with aldehydes: Formation of (II). A mixture of each of (I) (1g.), the appropriate aldehyde (0.5 ml), and a few drops of piperidine and ethanol (30 ml) was refluxed for 3 hr. The crystals which separated upon cooling and dilution of the reaction mixture with water were filtered off and crystallized from the suitable solvent to give (II). The details of these products are summarized in Table 2.

Condensation of 2-hydroxy-4, 6-diaryl-3-pyridyl alkyl ketones with ketones and diketones: Formation of (III). A mixture of (IIa and Ig) (0.01 mole), the ketone or diketone (0.015 mole) and anhydrous zinc chloride (0.5 g) was heated on an oil-bath at $200-30^\circ$ for 6 hr. After cooling the reaction mixture was washed with water and crystallized from a suitable solvent to give (IIIa-c) as coloured crystals with a yield of 60-70%. The results are listed in Table 3.

Action of hydrazines on (II a): Formation of (IV a-c). A solution of (II a) (0.01 mole), phenyl hydrazine, hydrazine hydrate or semicarbazide hydrochloride (0.01 mole) in alcohol (30 ml) was refluxed for 4 hr. The products obtained after concentration and cooling were crystallized from ethanol to give the pyrazoline derivatives (IVa-c) (cf. Table 4).

Action of hydroxylamine hydrochloride on (II a) for the formation of (V). A mixture of (II a) (0.01 mole), hydroxylamine hydrochloride (0.012 mole) and pyridine (20 ml) was refluxed for 4 hr. The cooled reaction mixture was poured into 10% cold acetic acid and the product obtained was crystallized from ethyl alcohol to give the oxazole derivative (V), m.p. 290° ; yellow crystals.

Analysis

(Found: C, 70.9; H, 4.9; $C_{27}H_{21}ClN_3O_3$ 456.5

Required: C, 70.97; H, 4.60).

Condensation of (IIa) with urea and/or thiourea: Formation of (VIa and VIb). A cold solution of (IIa) (0.01 mole) and urea/or thiourea (0.01 mole) in absolute ethanol (20 ml) was treated with concentrated sulphuric acid (2 ml). The mixture was set aside at room temperature for 24 hr. The reaction mixture was neutralized with sodium bicarbonate solution. The product separated was crystallized from ethyl alcohol to give (VI) as yellow crystals; yield, 65% (cf. Table 5).

Reaction of (IIa) with ethyl acetoacetate, acetyl acetone and/or diethylmalonate: Formation of (VIIa and VIIb) and (VIII). **Procedure (A).** To a boiling solution of (II a) (0.02 mole) in absolute ethyl alcohol (80 ml) was added a solution of sodium ethoxide (0.02 mole). The solution was cooled to room temperature and, while stirring, ethyl acetoacetate, acetyl acetone and/or diethyl malonate (0.02 mole) were added. Stirring was continued at 100° for one hr.; then the mixture was refluxed for 7 hr. during which the colour of the solution faded. The pale yellow alcoholic solution was concentrated to half its volume, cooled to room temperature and acidified with dilute hydrochloric acid. The crystals formed were crystallized from ethyl alcohol to give (VIIa and VIIb) and (VIII) (yield 70%) (cf. Table 6).

Procedure (B): (Fusion method). A mixture of (IIa) (0.01 mole) and ethyl acetoacetate acetyl-acetone and/or diethylmalonate (0.015 mole) and

TABLE 1. IR AND UV SPECTRA

Compound	Ir spectra (KBr, cm^{-1})	Uv spectra	
		λ_{max}	ϵ_{max}
IVa	1625, 1660, 1500, 3030		
V	1625, 1650, 3030		
VIb	1280, 1470, 1540 1610, 3350, 3440		
VIIa	1625, 1660, 3030	308 246	8820 19800
VIIb	1618, 1625, 1640, 3030		
VIII	1640, 1740	306 262	17860 29708
IX	1625, 1760, 3030		
Xa	1630, 1725, 1700, 3150, 3470		
Xb	1635, 1725, 1705, 3180, 3450		
XI	1640, 2220, 3470		
XIV	1615, 1715		
XV	1605, 1650, 1718		
XVI	1670, 3270		

TABLE 2

Compound	M. P. ($^{\circ}\text{C}$)	Colour	Solvent of crystalliza- tion	M. F. Mol. Weight	Analysis			
					Found		Required	
					C	H	C	H
IIa	241	Orange	E	$\text{C}_{27}\text{H}_{20}\text{Cl NO}_3$ (441.5)	73.4	4.5	73.38	4.53
IIb	178	Orange	Ac	$\text{C}_{27}\text{H}_{18}\text{Cl NO}_4$ (455.5)	71.08	4.0	71.13	3.95
IIc	146	Yellow	E	$\text{C}_{28}\text{H}_{20}\text{Cl NO}_2$ (437.5)	76.9	4.5	76.8	4.57
IId	271	Yellow	DMF	$\text{C}_{27}\text{H}_{20}\text{Cl NO}_2$ (425.5)	76.1	4.7	76.1	4.70
IIe	110	Yellow	E	$\text{C}_{29}\text{H}_{22}\text{Cl NO}_2$ (451.5)	77.1	4.8	77.07	4.87
IIf	250	Yellow	E	$\text{C}_{27}\text{H}_{18}\text{Cl NO}_4$ (417.5)	71.08	4.0	71.13	3.95
IIg	164	Buff	E/A	$\text{C}_{22}\text{H}_{18}\text{Cl NO}_6$ (439.5)	62.3	4.2	62.79	4.09
IIh	300	Yellow	DMF	$\text{C}_{29}\text{H}_{20}\text{Cl NO}_4$ (481.5)	72.2	4.2	72.27	4.15
IIi	146	Yellow	Ac	$\text{C}_{26}\text{H}_{18}\text{Cl NO}_3$ (427.5)	72.9	4.1	72.98	4.21
IIj	151	Brown	E	$\text{C}_{22}\text{H}_{17}\text{Cl N}_2\text{O}_6$ (440.5)	59.7	4.0	59.93	3.85

D.M.F. = dimethyl formamide, E = ethanol, A = acetone, Ac = acetic acid.

TABLE 3

Compound No.	M. P. (°C)	Colour	Solvent for crystallization	M. F. Mol. weight	Analysis %			
					Found		Required	
					C	H	C	H
IIIa	242	Yellow	A	C ₂₄ H ₂₂ Cl NO ₂ (391.5)	73.5	5.6	73.56	5.6
IIIb	215	Brownish-black	A	C ₂₉ H ₁₆ Cl NO ₆ (509.5)	68.6	2.9	68.30	3.14
IIIc	>300	Yellow	E	C ₄₂ H ₃₀ Cl ₂ N ₂ O ₄ (697)	472.3	4.3	72.3	4.3

TABLE 4

Compound No.	M. P. (°C)	Colour	Yield %	M. F. Mol. weight	Analysis %			
					Found		Required	
					C	H	C	H
IVa	210	White	60	C ₂₇ H ₂₂ Cl NO ₂ (455.5)	71.20	4.90	71.13	4.82
IVb	>300	Yellow	70	C ₃₃ H ₂₆ Cl NO ₂ (531.5)	74.46	4.95	74.50	4.89
IVc	>300	White	65	C ₂₈ H ₂₃ Cl N ₄ O ₃ (498.5)	67.35	4.52	67.40	4.61

TABLE 5

Compound No.	M. P.	M. F. Mol. weight	Analysis %			
			Found		Required	
VIa	110	C ₂₈ H ₂₂ Cl N ₃ O ₃ (483.5)	C	69.50	69.49	
			H	5.58	4.55	
VIb	134	C ₂₈ H ₂₂ Cl N ₃ O ₂ S (499.5)	C	67.16	67.26	
			H	4.42	4.40	
			S	6.5	6.40	

TABLE 6

Compound No.	M. P.	Colour	M. F. Mol. weight	Analysis %			
				Found		Required	
				C	H	C	H
VIIa	161	White	C ₃₀ H ₂₄ Cl NO ₃ (481.5)	74.8	5.0	74.76	4.98
VIIb	>300	Yellow	C ₃₂ H ₂₆ Cl NO ₄ (523.5)	73.3	4.8	73.35	4.86
VIII	181	White	C ₃₂ H ₂₆ Cl NO ₆ (555.5)	69.08	4.70	69.12	4.68

sodium methoxide (1g) was heated at 150° for 15 min., then at 200° for 2 hr. The residue was acidified with dilute HCl and then extracted with ether. The products obtained were crystallized from ethyl alcohol to give (VIIa and VIIb) and (VIII). (cf. Table 6).

Diels-Alder Reaction. A mixture of (IIe) (0.0 mole), maleic anhydride (0.02 mole) and dry xylene (50 ml) was refluxed for 12 hr. After hot filtration, concentration and cooling the red solid which had separated was crystallized from dioxane to give (IX) as red crystals (yield, (50-60%), m.p., -216°.

Analysis

(Found: C, 72.0; H, 4.40; C₃₃H₂₄Cl NO₅, 549.5
Required : C, 72.06 ; H, 4.36).

Hydrolysis of (IX) : Formation of (Xa). The anhydride (IX) (2 g.) was dissolved in 50 ml ethyl alcohol containing 2 g. of sodium hydroxide and the reaction mixture was refluxed for 2 hr., then filtered while hot. The filtrate was acidified with ice-cold dilute hydrochloric acid and the separated solid was crystallized from ethyl alcohol to give the dibasic acid (Xa) as yellow crystals ; yield, 60%, m p., 290°.

Analysis

(Found: C, 69.6; H, 4.6; C₃₃H₂₆Cl NO₆
567.5

Required: C, 69.77; H, 4.58).

Preparation of an Authentic Sample of (Xa). A solution of (IIe) (2 g.), maleic acid (5g) in dry xylene (30 ml) was refluxed for 10 hr. After con-

TABLE 7

Compound No.	M. P. (°C)	Colour	M. F. Mol. weight	Analysis %					
				Found			Required		
				C	H	N	C	H	N
XIa	247	Brown	C ₁₈ H ₁₂ Cl N ₃ (305.5)	70.8	3.8	13.7	70.70	3.92	13.74
XIb	248	Brown	C ₂₀ H ₁₇ Cl N ₄ (348.5)	68.75	4.90	16.00	68.86	4.87	16.06
XIc	264	Yellow	C ₁₉ H ₁₂ Cl N ₃ O ₂ (340.5)	65.1	3.4	12.1	65.23	4.43	12.01
XId	203	Yellow	C ₁₉ H ₁₄ Cl N ₃ O (335.5)	67.9	4.1	12.5	67.95	4.17	12.51
XIe	248	Brown	C ₁₈ H ₁₁ Cl N ₄ O ₂ (350.5)	61.4	3.2	15.9	61.62	3.13	15.97
XI'a	166	Brown	C ₂₀ H ₁₀ Cl N ₄ (350.5)	68.50	5.34	16.00	68.47	5.42	15.97
XI'b	135	Brownish-yellow	C ₁₈ H ₁₅ Cl N ₄ O ₂ (352.5)	61.30	3.70	15.90	61.27	3.68	15.88

TABLE 8

Compound No.	M. P. (°C)	Colour	M. F. Mol. weight	Analysis					
				Found			Required		
				C	H	N	C	H	N
XIIa	195	Yellow	C ₁₈ H ₁₄ Cl N ₃ O (323.5)	66.8	4.4	12.9	66.76	4.32	12.98
XIIb	252	Yellow	C ₁₉ H ₁₄ Cl N ₃ O ₃ (367.5)	62.1	3.9	11.4	62.04	3.80	11.42
XIIc	288	Brown	C ₁₈ H ₁₃ Cl N ₄ O ₃ (368.5)	58.6	3.6	15.2	58.61	3.52	15.19

TABLE 9

Compound No.	M. P.	Colour	M. F. Mol. weight	Analysis %			
				Found		Required	
				C	H	C	H
XIIIa	126	Yellow	$C_{18}H_{13}Cl N_2O_2$ (324.5)	66.6	4.1	66.56	4.00
XIIIb	188	Greenish-yellow	$C_{19}H_{13}Cl N_2O_4$ (368.5)	61.8 N 7.600	3.5	61.87 N 7.598	3.52
XIIIc	210	Brown	$C_{18}H_{12}Cl N_3O_4$ (334.3)	64.8	3.6	64.7	3.59

TABLE 10

Compound No.	M. P.	Colour	Yield %	M. F. Mol. weight	Analysis %					
					Found			Required		
					C	H	N	C	H	N
XIVa	>300	White	40	$C_{20}H_{13}Cl N_2O_2$ (348.5)	68.9	4.0	8.0	68.86	3.73	8.03
XIVb	>300	Brown	50	$C_{21}H_{13}Cl N_2O_4$ (392.5)	64.1	3.3	7.12	64.04	3.30	7.11
XIVc	>300	Brown	60	$C_{20}H_{12}Cl N_3O_3$ (377.5)	63.5	3.1	11.2	63.57	3.17	11.12

TABLE 11

Compound No.	M. P. (°C)	Colour	Yield %	M. F. Mol. weight	Analysis %					
					Found			Required		
					C	H	N	C	H	N
XVa	275	Yellow	90	$C_{28}H_{19}Cl N_2O_3$ (466.5)	72.1	4.0	6.0	72.02	4.07	6.00
XVb	310	Yellow	90	$C_{29}H_{19}Cl N_2O_5$ (511.5)	68.12	3.7	5.4	68.16	7.72	5.48
XVc	210	Orange	80	$C_{28}H_{18}Cl N_2O_5$ (511.5)	65.70	3.53	8.25	65.68	3.51	8.21

centration and cooling, the reaction product was identified to be (Xa) by means of m.p. and m.m.p. determination and comparison of ir spectra (yield 70%).

Esterification of (Xa) : Formation of (Xb). The bibasic acid (Xa) (2 g) was dissolved in absolute ethyl alcohol (50 ml) and a stream of dry hydrogen chloride was passed for 2 hr. After concentration the diester was obtained. It was crystallized from ethyl alcohol to give (Xb) as yellow crystals (yield, 70%), m.p. 300°.

Analysis

(Found: C, 71.2, H, 5.5; $C_{37}H_{34}ClNO_6$ 623.5

Required: C, 71.21; H, 5.45).

Preparation of an authentic sample of (Xb). A solution of (IIe) (2 g.) and diethyl maleate (5 ml) in dry xylene (30 ml) was refluxed for 10 hr. After concentration and cooling, the reaction product was identified to (Xb) by m.p. and m.m.p. determination (yielded 60%).

Reaction of malononitrile with chalcones. A mixture of malononitrile (0.05 mole), appropriate chalcone (0.05 mole), and ammonium acetate (0.03 mole) was heated at 150° for 5 hr. After cooling a solid matter separated; this was filtered, washed with water, then treated with ethanol. It was divided into two parts: an ethanol-soluble part (A) and an ethanol insoluble precipitate (B) which was crystallized from glacial acetic acid to afford (XI) as coloured crystals (yield 60%) (cf. Table 6). The ethanol solution was concentrated. After it had stood overnight at room temperature, coloured crystals separated which after crystallization from ethanol gave (XI) (cf. Table 7).

Hydrolysis of (XI) : Formation of (XII). (XI). (2 g) was heated with 20% alcoholic potassium hydroxide (50 ml) for 5 hr. After the addition of water (30 ml) to the reaction mixture a solid was obtained which crystallized from ethanol (yield 75%) (cf. Table 8).

Hydrolysis of (XII a, b and c) : Formation of (XIII a-c). To a solution of potassium hydroxide (4.9) in glycerol (20 ml) (XIIa, b and c) (1 g.) was added and the mixture was boiled at 200° for 5 hr. After cooling the solution was neutralized with dilute hydrochloric acid; a crystalline solid separated, which was then crystallized from ethanol (yield, 70%) (cf. Table 9).

Condensation of acetic anhydride with 2-amino-4,6-diaryl nicotinic acid (XIIIa, b and c) : Formation of (XIVc). A solution of (XIIIa, b and c) (0.01 mole) in acetic anhydride (40 ml) was refluxed for 8 hr. The solid obtained after concentration and cooling was crystallized from ethyl alcohol (cf. Table 10).

Condensation of 2-Methyl-oxazone Derivatives (XIV) with Aldehydes : Formation of (XVa, b and c). A mixture of (XIV) (0.01 mole), appropriate aldehyde (0.015 mole) and anhydrous zinc chloride (0.5 g) was heated in an oil bath at 170° for 3 hr. After cooling the product was washed with water and crystallized from ethyl alcohol to give the corresponding 2-styryloxazone derivatives (XVa, b and c). (Cf. Table 11).

Reaction of 2-Methyl Oxazone Derivative (XIV a) with Butylamine : Formation of (XVI). A mixture of (XIVa) (0.01 mole), butylamine (0.01 mole) and anhydrous zinc chloride (0.5 g.) was heated in an oil bath at 170-80° for 4 hr. The solid product was cooled, washed with dilute hydrochloric acid (5%) filtered and crystallized from ethyl alcohol; (yield, 50-70%; m.p. 207°).

Analysis

Analysis. (Found: C, 68.3; H, 5.7; N, 9.9;

$C_{24}H_{24}ClN_3O_2$ (421.5)

Required: C, 68.32; H, 5.69; N, 9.96).

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