M. Mercedes Blanco, Isabel A. Perillo* and Celia B. Schapira

Departamento de Química Orgánica, Facultad de Farmacia y Bioquímica, Universidad de Buenos Aires, Junín 956 (1113), Buenos Aires, República Argentina

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A number of 8-hydroxy-6-methyl-1,6-naphthyridin-5(6*H*)-one-7-carboxylic acid alkyl esters **3** and the isomeric 5-hydroxy-7-methyl-1,7-naphthyridin-8(7*H*)-one-6-carboxylic acid alkyl esters **4** were synthesized from acyclic precursors obtained starting from quinolinic anhydride **5**. Thus, methanolysis of **5** afforded the hemiester **6** which treated with oxalyl chloride and sarcosine ethyl ester gave 3-(*N*-ethoxycarbonylmethyl-*N*-methylcarbamoyl)pyridine-2-carboxylic acid methyl ester **8**. Compound **8** was cyclized to naphthyridines **3a-e** with sodium alkoxides. The isomeric naphthyridines **4a-c** were obtained by cyclization of the open intermediary 2-(*N*-ethoxycarbonylmethyl-*N*-methylcarbamoyl)pyridine-3-carboxylic acid methyl ester **9** obtained by a route that involves treatment of **5** with sarcosine ethyl ester and esterification with diazomethane. Spectroscopic properties (^{1}H nmr, uv, ir) of compounds **3** and **4** are discussed and confirmed the proposed structures.

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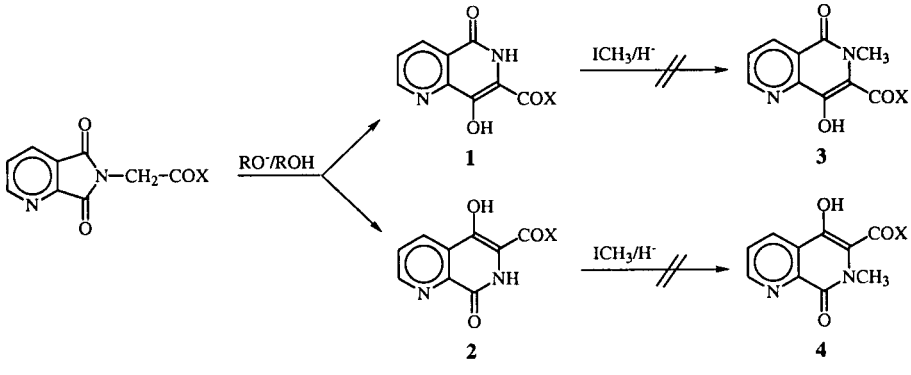
Introduction.

In previous work [1], we reported the preparation of 7-substituted 8-hydroxy-1,6-naphthyridin-5(6*H*)-ones **1** and of 6-substituted 5-hydroxy-1,7-naphthyridin-8(7*H*)-ones **2** by expansion reactions induced by alkoxides of quinolinimidoacetic acid derivatives (Scheme I). Given our interest in these types of compounds, we then focused our attention on the synthesis of *N*-methyl analogues **3** and **4**, in order to obtain more stable products that were less hydrophilic in character. Initially, *N*-methylation of naphthyridines **1** and **2** seemed to be the most direct route for their preparation. However, treatment of these compounds with methyl iodide under diverse conditions afforded a complex mixture of unidentified products. These results, together with the difficult separation of the isomeric naphthyridines obtained by rearrangement of quinolinimides [1], led us to outline a strategy that involved the synthesis and ring closure of open products obtained starting from quinolinic anhydride **5**.

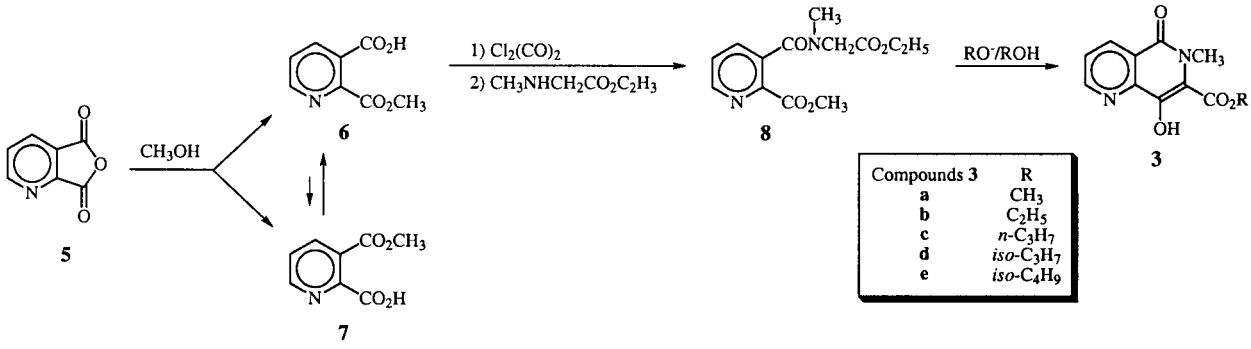
Scheme I



Results and Discussion.

The first alternative synthesis that was designed used quinolinic acid hemiesters **6** and **7** as intermediaries. The 2,3-pyridinedicarboxylic acid 2-methyl ester **6** (stable isomer) was obtained pure without difficulty by methanolysis of **5** [2]. Successive treatment of **6** with oxalyl chloride and sarcosine ethyl ester gave the expected 3-(*N*-ethoxycarbonylmethyl-*N*-methylcarbamoyl)pyridine-2-carboxylic acid methyl ester **8** which was cyclized to 8-hydroxy-6-methyl-1,6-naphthyridin-5(6*H*)-one-7-carboxylic acid ethyl ester **3b** with sodium ethoxide (Scheme II). If the ring closure reaction is carried out with sodium methoxide, total transesterification is observed, with methyl ester **3a** as the only product. As already observed in similar reactions [1,3], transesterification almost surely takes place in a step prior to cyclization, since prolonged heating of **3b** with sodium methoxide provides only small quantities of the transesterified product. With alkoxides of alcohols having greater molecular weight (*n*-propyl, isopropyl or isobutyl alcohol), transesterification is partial, affording a mixture of the corresponding compounds **3c-e** as major products and small quantities of the ethyl ester **3b**, which were separated by chromatographic methods in every case.

Scheme II



With regard to the preparation of 2,3-pyridinedicarboxylic acid 3-methyl ester **7** (labile isomer) by methanolysis of **5**, the literature is confusing [2,4,5]. In our hands, this reaction gave a mixture of **7** as the major product with variable quantities of **6**, while attempts to obtain the pure compound proved fruitless. A fast **7** → **6** transformation upon standing was also observed, which provides evidence of the dissimilar stability of the two isomers. Besides, treatment of a pure sample of **7** recently obtained starting from 2,3-pyridinedicarboxylic acid dimethyl ester [6], under the same conditions as those described for **6**, led exclusively to naphthyridine **3b**, indicating that an isomerization process took place prior to the reaction with oxalyl chloride (Scheme II).

Accordingly, it was decided to attempt the synthesis of the open chain intermediate **9** by means of an alternative route that involves aminolysis of the quinolinic anhydride **5** with sarcosine ethyl ester and later esterification with diazomethane (Scheme III). As expected, given the dissimilar reactivity of the carbonyl groups, the main product was 2-(*N*-ethoxycarbonylmethyl-*N*-methylcarbamoyl)pyridine-3-carboxylic acid methyl ester **9**, together with variable quantities of isomer **8** [7]. The reaction mixture treated with sodium ethoxide led to a mixture of 5-hydroxy-7-methyl-1,7-naphthyridin-8(7*H*)-one-6-carboxylic acid ethyl ester **4b** as the major and 1,6-naphthyridine **3b** as the minor product. Likewise, the reaction with sodium methoxide or *n*-propoxide allowed compounds **4a** and **4c**, respectively, to be obtained. In all cases compounds **3** and **4** were isolated by centrifugally accelerated radial chromatography. Elemental analyses and spectroscopic properties (as discussed below) are presented in Tables I and II and confirm the proposed structures. On the whole, the 1,6-naphthyridines **3** have lower Rf, giving a green coloration with ferric chloride and forming chelate structures with ferrous sulphate, confirming the presence of a hydroxyl group *peri* to a ring nitrogen atom [8]. On the other hand, the 1,7-naphthyridines **4** have higher Rf, give a brown coloration with ferric chloride and fail to form chelates.

Spectroscopic Features of Naphthyridines **3** and **4**.

In the ^1H nmr spectra, the chemical shift of the pyridine ring hydrogens (a, b and c) in dimethyl-d_6 sulfoxide presents the same pattern as that of the analogue naphthyridines **1** and **2** [1]. Thus, the 1,6-naphthyridines **3** present greater chemical shifts for Ha and Hc, but a lower shift for Hb, *versus* the corresponding protons in the 1,7-naphthyridines **4**. These results may be interpreted as a consequence of the contribution of resonant structures **A** and **B** for compounds **3** and of **C** for **4**.

Scheme III

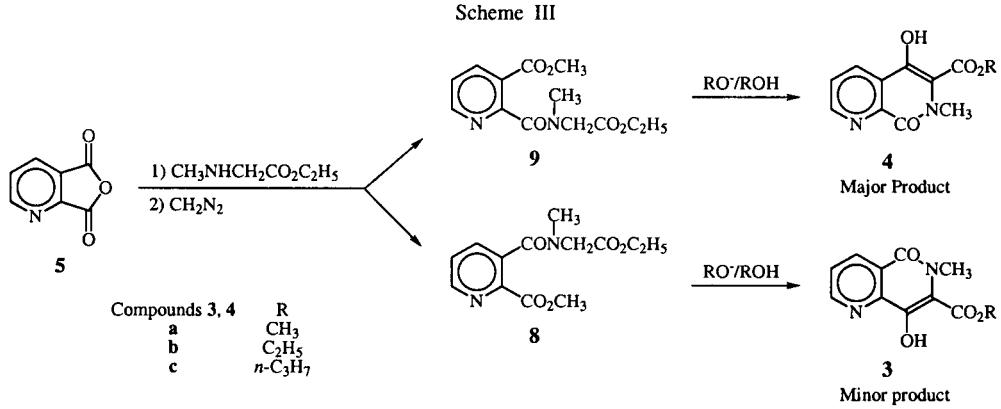
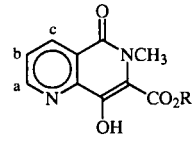


Table I
8-Hydroxy-6-methyl-1,6-naphthyridin-5(6*H*)-one-7-carboxylic Acid Alkyl Esters **3a-e**



Compound No.	Mp (°C)	Yield (%)	Formula	Analyses (Calcd./Found) %C	%H	%N	Mass M+· (%)	IR ν (cm-1)	UV [a] 0.1*N* HCl λ max (nm)	ethanol λ max (nm)	0.1*N* NaOH λ max (nm)	δ (ppm)	1H-NMR Multiplicity	Assignment
3a	120	50	$C_{11}H_{10}N_2O_4$	56.41	4.30	11.96	234	3280	335	331	382	9.40	bs [c]	OH
	[b]			56.57	4.25	12.08	(47.2)	2975	260 [d]	256	268	9.00	dd [e]	Ha
								1710	242		231	8.70	dd [e]	Hc
								1658				7.61	dd [e]	Hb
								1615				4.00	s	$O\text{-}CH_3$
								1580				3.60	s	$N\text{-}CH_3$
								1260						
3b	95	43	$C_{12}H_{12}N_2O_4$	58.06	4.87	11.28	248	3308	333	331	380	9.30	bs [c]	OH
	[b]			58.26	4.93	11.02	(39.0)	2970	259 [d]	256	268	9.00	dd [f]	Ha
								1719	246		230	8.70	dd [f]	Hc
								1648				7.60	dd [f]	Hb
								1620				4.51	c	CH_2
								1590				3.60	s	$N\text{-}CH_3$
								1270				1.51	t	$C\text{-}CH_3$
3c	90	64	$C_{13}H_{14}N_2O_4$	59.54	5.38	10.68	262	3280	333	330	382	9.52	bs [c]	OH
				59.36	5.45	10.80	(39.8)	2964	258 [d]	257	268	9.01	dd [g]	Ha
								1718	256		236	8.70	dd [g]	Hc
								1650				7.59	dd [g]	Hb
								1610				4.40	t	$O\text{-}CH_2$
								1590				3.60	s	$N\text{-}CH_3$
								1258				1.85	m	$C\text{-}CH_2$
								1099				1.10	t	$C\text{-}CH_3$
3d	124	59	$C_{13}H_{14}N_2O_4$	59.54	5.38	10.68	262	3300	332	330	385	9.40	bs [c]	OH
				59.41	5.30	10.79	(37.5)	2981	259 [d]	255	270	9.05	dd [h]	Ha
								2935	256		242	8.72	dd [h]	Hc
								1720				7.60	dd [h]	Hb
								1662				5.41	m	CH
								1625				3.60	s	$N\text{-}CH_3$
								1585				1.42	d	$C\text{-}CH_3$
3e	[i]	25	$C_{14}H_{16}N_2O_4$	60.86	5.84	10.14	276	3097	333	330	382	9.50	bs [c]	OH
				61.00	5.92	9.99	(41.3)	1718	258 [d]	258	267	9.00	dd [j]	Ha
								1660	250		235	8.70	dd [j]	Hc
								1622				7.62	dd [j]	Hb
								1591				4.20	d	OCH_2
								1097				3.70	s	$N\text{-}CH_3$
												2.15	m	CH
												1.10	d	$C\text{-}CH_3$

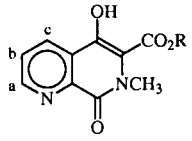
[a] Only absorptions above 230 nm are considered. [b] Recrystallized from methanol. [c] Exchangeable. [d] Shoulder. [e] $J_{Ha\text{-}Hb}$ = 4.52 Hz, $J_{Ha\text{-}Hc}$ = 1.38 Hz, $J_{Hb\text{-}Hc}$ = 8.10 Hz. [f] $J_{Ha\text{-}Hb}$ = 4.00 Hz, $J_{Ha\text{-}Hc}$ = 1.80 Hz, $J_{Hb\text{-}Hc}$ = 8.01 Hz. [g] $J_{Ha\text{-}Hb}$ = 4.59 Hz, $J_{Ha\text{-}Hc}$ = 1.33 Hz, $J_{Hb\text{-}Hc}$ = 8.05 Hz. [h] $J_{Ha\text{-}Hb}$ = 4.59 Hz, $J_{Ha\text{-}Hc}$ = 1.30 Hz, $J_{Hb\text{-}Hc}$ = 7.41 Hz. [i] This compound could not be crystallized. [j] $J_{Ha\text{-}Hb}$ = 4.41 Hz, $J_{Ha\text{-}Hc}$ = 1.47 Hz, $J_{Hb\text{-}Hc}$ = 8.09 Hz.

Characteristically, the ir spectra of compounds **4** present in potassium bromide, the ester carbonyl stretching at *ca.* 1660 cm^{-1} similar to that of related benzothiazines [3], and compatible with a structure having an intramolecular hydrogen bond **D** [9]. Similar structures were proposed for structurally related isoquinolones [10,11].

On the other hand, in compounds **3** the ester carbonyl absorption occurs at *ca.* 1710 cm^{-1}, similar to the frequency in benzoic and α,β-unsaturated esters [9]. This observation indicates that the ester carbonyl moiety is not involved in an intramolecular hydrogen bond with the enolic hydroxyl, whence the possibility is inferred of the existence in the solid state of a zwitterion structure **E** (R' = CH_3) or alternatively a hydrogen bond between the hydroxyl and the pyridine nitrogen (**F**), similar to that of 8-hydroxyquinoline [12,13]. However, the absence of absorption bands between 2500 and 2325 cm^{-1} rules out the presence of the protonated pyridine nitrogen [14] and lends further support to the second option.

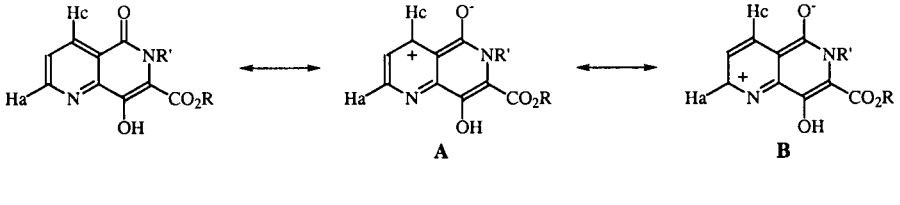
Table II

5-Hydroxy-7-methyl-1,7-naphthyridin-8(7*H*)-one-6-carboxylic Acid Alkyl Esters **4a-c**

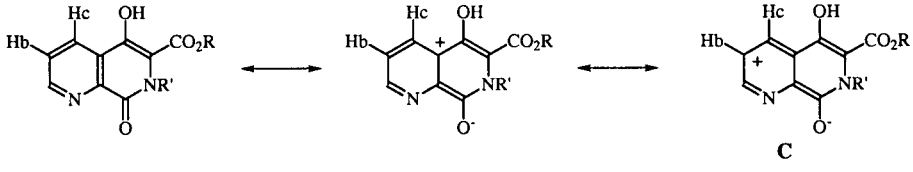


Compound No.	Mp (°C)	Yield (%)	Formula	Analyses Calcd./Found) %C	%H	%N	Mass M+• (%)	IR ν (cm-1)	0.1*N* HCl λ max (nm)	UV [a] ethanol λ max (nm)	0.1*N* NaOH λ max (nm)	δ (ppm)	^{1}H-NMR multiplicity	Assignment
4a	129	46	$C_{11}H_{10}N_2O_4$	56.41	4.30	11.96	234	3420	346	344	380	11.1	bs [c]	OH
	[b]			56.59	4.39	12.10	(78.3)	2960	265	258	276 [d]	9.10	dd [e]	Ha
								1650	239	230	269	8.49	dd [e]	Hc
								1638			230	7.70	dd [e]	Hb
								1585				4.10	s	CH_3
								1328				3.70	s	N-CH_3
								1250						
4b	148	52	$C_{12}H_{12}N_2O_4$	58.06	4.87	11.28	248	3415	345	344	380	11.20	bs [c]	OH
	[b]			58.29	5.82	11.36	(69.0)	2970	266	258	277[d]	9.10	dd [f]	Ha
								1660	239	230	269	8.50	dd [f]	Hc
								1588			230	7.71	dd [f]	Hb
								1320				4.50	c	CH_2
								1265				3.70	s	N-CH_3
												1.45	t	CH_3
5c	142	60	$C_{13}H_{14}N_2O_4$	59.54	5.38	10.68	262	3423	343	340	383	11.21	bs [c]	OH
				59.40	5.39	10.79	(76.8)	2964	267	256	270 [d]	9.10	dd [g]	Ha
								2881	236	230	268	8.50	dd [g]	Hc
								1654			235	7.70	dd [g]	Hb
								1583				4.40	t	O-CH_2
								1408				3.70	s	N-CH_3
								1330				1.85	m	C-CH_2
								1259				1.05	t	C-CH_3

[a] Only absorptions above 230 nm are considered. [b] Recrystallized from ethanol. [c] Exchangeable. [d] Shoulder. [e] $J_{Ha\text{-}Hb} = 4.40$; $J_{Ha\text{-}Hc} = 1.47$; $J_{Hb\text{-}Hc} = 8.21$. [f] $J_{Ha\text{-}Hb} = 4.70$; $J_{Ha\text{-}Hc} = 1.46$; $J_{Hb\text{-}Hc} = 7.93$; [g] $J_{Ha\text{-}Hb} = 4.48$; $J_{Ha\text{-}Hc} = 1.66$; $J_{Hb\text{-}Hc} = 8.19$.

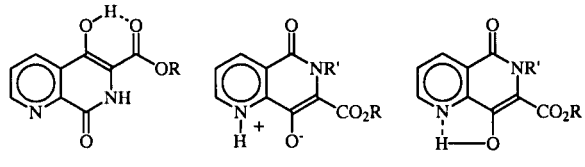


A **B**



C

The uv spectra of naphthyridines **3** and **4** show close similarity in alcoholic solution and in acid medium, presenting two strong bands at 330-335 and 255-259 nm for compounds **3** displaced to higher wavelength in compounds **4**. In all cases a strong bathochromic effect in basic solution is observed (enolate anion). These results indicate the predominance of a normal covalent structure, **3** and **4**, in alcoholic solution and highlight the difference between compounds **3** and their 6-unsustituted analogues **1**, which present a major contribution of a zwitterion structure (**E**, R' = H) under the same conditions [1].



D **E** **F**

EXPERIMENTAL

Melting points were taken on a Büchi capillary apparatus and are uncorrected. The ^1H nmr spectra were recorded on a Bruker MSL 300 MHz using deuteriochloroform as solvent. Chemical shifts are quoted in parts per million (δ) downfield from the internal tetramethylsilane reference. The presence of exchangeable protons was confirmed by the use of deuterium oxide. Proton signals are quoted as: s (singlet), bs (broad singlet), d (doublet), dd (doublet of doublet), t (triplet), c (quartet) and m (multiplet). Mass spectra were performed on a MS Shimadzu QP-1000 instrument at 70 eV. The ir spectra were recorded on a Beckman 180A spectrometer and samples were run as potassium bromide pellets. The uv spectra were recorded on a Jasco 7850 UV-VIS spectrophotometer. Analytical tlc was carried out on aluminium sheets, Silica Gel 60 F_{254}. Preparative thin layer separations (plc) were carried out by centrifugally accelerated, radial chromatography using Chromatotron model 7924T. The rotors were coated with Silica Gel 60 PF_{254} and the layer thickness was 2 mm. Chloroform and increasing percentages of methanol were used as eluents. Reagents, solvents and starting materials were purchased from standard sources and purified according to literature procedures.

Attempted *N*-Methylation of Naphthyridines **1** and **2**.

In a typical procedure, to a slurry of 0.16 g of a 53.4% mineral oil dispersion of sodium hydride (0.003 moles) in dimethylformamide (5 ml) was added a solution of 8-hydroxy-1,6-naphthyridin-5(6*H*)-one-7-carboxylic acid methyl ester (**1**, X = OCH_3) [1] (0.0015 moles) in dimethylformamide (5 ml), the temperature being maintained at 5-10° during the addition. The solution was stirred at room temperature for 1 hour and 1.5 ml of methyl iodide was added at 0-10°. After stirring for 1 hour at room temperature, the reaction mixture was poured into 10% oxalic acid (ice bath) and the solution was extracted three times with chloroform. The organic layer was washed with water, dried and concentrated *in vacuo* affording a complex mixture of products which were not further characterized.

Similar results were obtained using sodium hydride in hexamethylphosphoric triamide or 1,2-dimethoxyethane as the solvents or calcium hydride in dimethylformamide.

2,3-Pyridinedicarboxylic Acid 2-Methyl Ester **6**.

This compound was prepared following Kenyon and Thaker's procedure [2].

3-(*N*-Ethoxycarbonylmethyl-*N*-methylcarbamoyl)pyridine-2-carboxylic Acid Methyl Ester **8**.

A suspension of **6** (0.005 moles) in dry chloroform (5 ml) and oxalyl chloride (0.006 moles) was heated at 40° for 3 hours. The solvent and excess of oxalyl chloride were removed *in vacuo* and the residual oil dissolved in dry chloroform (5 ml). The solution was stirred and treated with sarcosine ethyl ester hydrochloride (0.005 moles) and then triethylamine (0.008 moles) was added dropwise. The reaction mixture was refluxed for 4 hours, evaporated *in vacuo* and dry benzene (5 ml) added twice evaporating each time to dryness affording **8** as an oil which was used in the next step without purification.

An analytical sample of **8** was achieved by plc; ms: m/z 280 ($M^{+\bullet}$); ^1H nmr: δ 8.64 (dd, 1H, H6), 7.70 (dd, 1H, H4), 7.48 (dd, 1H, H5), 4.22 (s, 2H, N-CH_2), 4.02 (c, 2H, O-CH_2), 3.86 (s, 3H, O-CH_3), 3.07 (s, 3H, N-CH_3) and 1.09 (t, 3H, CH_2-CH_3).

Anal. Calcd. for $C_{13}H_{16}N_2O_5$: C, 55.71; H, 5.75; N, 9.99. Found: C, 55.60; H, 5.69; N, 10.05.

8-Hydroxy-6-methyl-1,6-naphthyridin-5(6*H*)-one-7-carboxylic Acid Alkyl Esters **3**.

In a typical procedure, 1.1 g of the crude product **8** was dissolved in refluxing ethanol (5 ml) and 3 ml of 2*M* sodium ethoxide was added and refluxed for 15 minutes. The orange yellow suspension was poured into ice-acetic acid and extracted three times with chloroform. After washing with water the organic solution was dried, concentrated *in vacuo* and purified affording **3b**.

When **8** was treated with sodium methoxide under the same conditions as above, total transesterification took place, affording only **3a**. By treatment of **8** with the sodium salts of *n*-propyl, isopropyl or isobutyl alcohol a mixture of compounds **3c**, **3d** or **3e** respectively, together with **3b** as a minor product was obtained. In each case separation of the two products was accomplished by centrifugal plc.

Melting points, recrystallization solvent, yields, elemental analyses and spectroscopic data of compounds **3a-e** are given in Table I.

When **3b** was treated with sodium methoxide under the same conditions as above, **3b** remained almost unchanged and only traces of **3a** were observed.

2,3-Pyridinedicarboxylic Acid 3-Methyl Ester **7**.

The method of Kenyon and Thaker starting from **5** [2] gave in our hands a mixture of **7** as the major product and **6** in a much smaller proportion. Several attempts to obtain pure **7** failed and rapid isomerization to the more stable isomer **6** was observed. Pure compound **7** was obtained by selective hydrolysis of 2,3-pyridinedicarboxylic acid dimethyl ester, mp 107-108°, (lit [6] 106°). Successive treatment of **7** with oxalyl chloride, sarcosine ethyl ester and sodium methoxide as for compound **6** afforded only **3b**.

2-(*N*-Ethoxycarbonylmethyl-*N*-methylcarbamoyl)pyridine-3-carboxylic Acid Methyl Ester **9**.

To a solution of **5** (0.007 moles), *p*-toluenesulfonic acid (10 mg) and sarcosine ethyl ester hydrochloride (0.008 moles) in tetrahydrofuran (10 ml) at room temperature was added dropwise and with stirring triethylamine (0.008 moles) in tetrahydrofuran (10 ml). After stirring 1 hour the reaction mixture was cooled (ice bath), filtered, and the organic solution concentrated *in vacuo*. The oily residue was dissolved in anhydrous methanol (5 ml) (ice bath) and an ethereal solution of diazomethane was added in small portions until the solution acquires a pale yellow color. After 24 hours at room temperature, the reaction mixture was concentrated *in vacuo* and used in the next step without purification. The reaction mixture shows two spots by tlc. Separation was accomplished by centrifugal plc. The first band eluted afforded **9** (50% yield); m/z 280 ($M^{+\bullet}$); ^1H nmr: δ 8.70 (dd, 1H, H6), 8.29 (dd, 1H, H4), 7.40 (dd, 1H, H5), 4.30 (s, 2H, N-CH_2), 4.10 (c, 2H, O-CH_2), 3.88 (s, 3H, O-CH_3), 3.20 (s, 3H, N-CH_3) and 1.17 (t, 3H, CH_2-CH_3).

Anal. Calcd. for $C_{13}H_{16}N_2O_5$: C, 55.71; H, 5.75; N, 9.99. Found: C, 55.80; H, 5.65; N, 10.09.

The second band eluted (10% yield) was identified as compound **8**.

When the aminolysis reaction was carried out without *p*-toluenesulfonic 35% of **9** and 15% of **8** were obtained.

5-Hydroxy-7-methyl-1,7-naphthyridin-8(7*H*)-one-6-carboxylic Acid Alkyl Esters **4**. General Procedure.

A mixture of compounds **9** and **8** obtained as above (1.1 g) was dissolved in the corresponding alcohol (5 ml) and 3 ml of 2*M* sodium alkoxide was added and refluxed for 15 minutes. The orange yellow suspension was poured into ice-acetic acid and extracted three times with chloroform. After washing with water the organic solution was dried and concentrated *in vacuo*. The crude product showed two spots by tlc (Rf *ca.* 0.4 and 0.5 in 9:1 chloroform-methanol). Separation of the two products was accomplished by centrifugal plc. The first band eluted gave the major product which was recrystallized affording compounds **4a-c**. The slower moving band afforded compounds **3a-c**. Melting points, recrystallization solvent, yields, elemental analyses and spectroscopic data of the compounds are given in Tables I and II.

Compounds **3** give a green coloration with ferric chloride and deep green chelates with ferrous sulphate [8]. Compounds **4** give brown coloration with ferric chloride and negative reaction with ferrous sulphate.

Acknowledgments.

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REFERENCES AND NOTES

[*] Author to whom correspondence should be addressed.

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