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KETO ACIDS IN THE PYRROLE SERIES

V. N. Eraksina, T. M. Ivanova,
T. A. Babushkina, A. M. Vasil'ev,
and N. N. Suvorov

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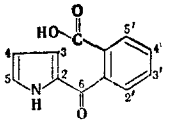
The spectral properties of o-(2-pyrrolylketo)benzoic acid and its N-methyl and N-benzyl analogs were investigated in order to detect ring-chain tautomerism. It is shown that the investigated acids exist in the open keto form. The corresponding derivatives involving the carbonyl group were obtained. The preparation of derivatives of the cyclic lactol form of the 2-pyrrolylketobenzoic acids is described.

The previously described [1] o-(2-pyrrolylketo)benzoic acid is of interest as a model for the study of various types of tautomeric transformations [2-4]. We have used it for the study of ring-chain tautomerism, in analogy with acetophenone-o-carboxylic acid [3, 4].

Keto acid I was obtained by reaction of pyrrolylmagnesium iodide with phthalic anhydride in anisole [1]. However, the reaction was complicated by considerable resinification and the production of cyclic lactam II; this was evidently a consequence of carrying out the reaction at high temperature. The yield of acid I was only 30%. We found that the yield can be increased to 63% if the reaction is terminated immediately after the addition of a hot solution of phthalic anhydride to the pyrrolylmagnesium iodide; in this case the presence of lactam II is not detected even by chromatography. Nevertheless, the reaction product has a melting point that is 40°C lower than that of pure acid I and, according to chromatography, is a mixture of two compounds. To separate them we used column chromatography on silica gel. The principal reaction product is actually acid I, which was isolated in a ratio of 9:1 relative to the second compound, which also has acid character (Ia). On the basis of an examination of the UV (Fig. 1) and IR spectra we assumed that we are dealing with structural isomers: It was shown by mass spectrometry that the molecular ion at m/e 215 is the maximum peak in the spectra of both compounds and that the fragmentation pathways of the two substances coincide. An analysis of the PMR spectra (Table 2), which we will discuss below, confirms the assumption made above.



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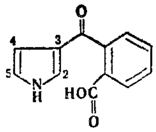

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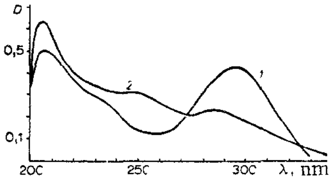


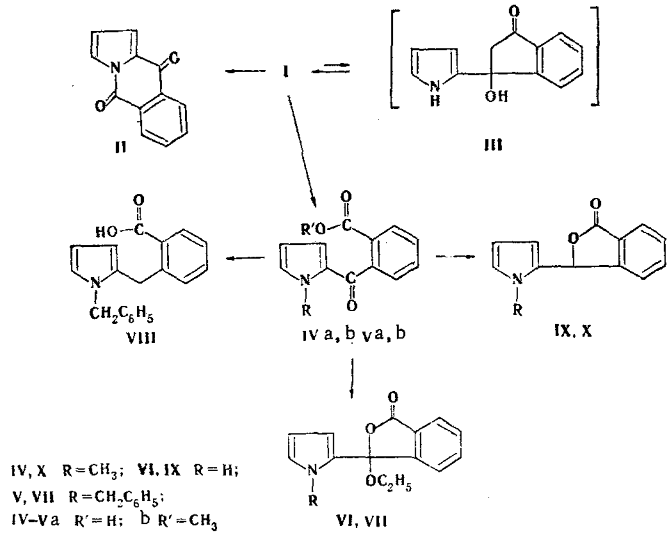
Fig. 1. UV spectra of 2-pyrrolylketo- and 3-pyrrolylketobenzoic acids: 1) o-(2-pyrrolylketo)benzoic acid (I); 2) o-(3-pyrrolylketo)benzoic acid (Ia).

D. I. Mendeleev Moscow Institute of Chemical Technology, Moscow 125047. Institute of Biophysics of the Ministry of Public Health of the USSR, Moscow 123182. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 7, pp. 916-922, July, 1979. Original article submitted July 25, 1978.

TABLE 1. Spectral Characteristics of the Keto Acids

Compound	Solvent, mole/liter	IR Spectrum, cm^{-1}		UV Spectrum	
		C=O	COOH	λ_{max}, nm	$\varepsilon \cdot 10^3$
I	KBr	1575 i 1595, 1630	1685, 1700, 1750 d 1710 i, 1715, 1745	203 295	18,7 11,2
	$CHCl_3$, $5 \cdot 10^{-3}$	1605, 1637 d	1715, 1735, 1750 d		
	$CHCl_3$, $5 \cdot 10^{-5}$	1640	1700 i, 1715, 1730 wi.		
	DMSO	1640	1717, 1725, 1755, 1765 i		
	CCl_4	1650	1735		
	CH_3CN	1645			
IVa	KBr	1590	1715	203	21,0
	$CHCl_3$, $2 \cdot 10^{-2}$	1640	1710, 1735 i	296	16,1
	$CHCl_3$, $2 \cdot 10^{-4}$	1645	1715 i, 1740		
Va	KBr	1650	1680, 1690, 1697, 1720 i, 1740 i	205 296	36,7 15,5
	$CHCl_3$, $3 \cdot 10^{-2}$	1643	1695, 1705, 1740 i		
	$CHCl_3$, $3 \cdot 10^{-4}$	1643	1715 i, 1745		
IVb	KBr	1590 int 1645	1695, 1715	206	15,2
	$CHCl_3$, $2 \cdot 10^{-2}$	1640	1700 vs, 1720, 1730	298	15,2
	$CHCl_3$, $2 \cdot 10^{-4}$	1640	1700 w, 1732		
VII	$CHCl_3$		1715, 1777	207	19,5
	DMSO			230	18,0
VI	Mineral oil	1605	1760, 3380	205 230	16,1 15,0
X	$CHCl_3$	1610	1770, 3340, 3540	204 226	17,2 20,0

The use of succinic acid in the Grignard reaction led to γ-(2-pyrrolylketo)butyric acid. The keto acids obtained are alkylated in alkaline media by dimethyl sulfate and benzyl chloride at the nitrogen atom of the pyrrole ring. Under the methylation conditions that we used, N-methyl-2-pyrrolylketobenzoic acid was isolated in the form its methyl ester (IVb). Methyl ester Vb was obtained by methylation of Va with a solution of diazomethane. It has been previously shown [1] that keto acid I in acidic media undergoes internal acylation to give lactam II; the same lactam was also obtained by the action of a solution of diazomethane on keto acid I. In the case of benzylation the yield of pure keto Va is 50%; o-Phthalic acid was also isolated by means of column chromatography. Another hydrolytic cleavage product, viz., benzoic acid, is obtained if treatment of the reaction mixture with steam is used for the isolation of keto acid Va. However, we were unable to isolate pure products with pyrrole character from the resin formed in this case.



IV, X R=CH$_3$; VI, IX R=H;
V, VII R=CH$_2$C$_6$H$_5$;
IV–Va R′=H; b R′=CH$_3$

The presence of two electron-donor and two electron-acceptor groups in keto acid I creates possibilities for different types of intramolecular interactions and for the formation of various types of hydrogen bonds. The absorption of the α-carbonyl group in the IR spectrum of a KBr pellet of keto acid I shows up in the form of several bands at 1580-1630 cm^{-1}, while the absorption of the carboxy group shows up in the form of a broad intense band at ~1700 cm^{-1}. These values provide evidence that I exists in the crystalline state in the open

TABLE 2. Parameters of the PMR Spectra

Compound	Solvent	δ, ppm				J, Hz						δ, ppm						J, Hz	
		NR	5-H	4-H	3-H	1,3	1,5	1,4	4,5	3,5	3,4	5'-H	3',4'-H	2'-H	COOH	CH_2	CH_3	CH_2-CH_3	CH_A-CH_B
I	$CDCl_3$	10,02	7,10	6,21	6,48	2,5		2,3	2,7	1,25	4,0	7,98	7,52						
	DMSO	11,91	7,07	6,09	6,23	2,6	2,8	2,0	2,4	1,3	3,8	7,80	7,54	7,38					
IVa	DMSO	CH_3 3,90	7,11	5,97	6,09		CH_3, H^2 0,3		2,4	1,8	4,0	7,82	7,55	7,35	12,78				
Va	DMSO	NCH_2 5,63 C_6H_5 7,07—7,35	7,22	6,06	6,20				2,4	1,7	4,0	7,83	7,53	7,28	12,83				
VI	$CDCl_3$	8,72	6,76	6,08	5,99	2,8	2,8	2,5	2,6	1,4	3,6	7,82	7,56			3,25 3,49	1,14	7,0	8,0
	DMSO	11,18	6,72	5,95								7,85	7,72			3,22 3,39	1,08	6,8	8,8
VII	$CDCl_3$	NCH_2 5,55; 5,28 $J=15,4$ Hz C_6H_5 6,8—7,2	6,56	5,91	5,72		CH_2, H^2 0,3		2,8	1,9	3,8	7,85	7,64			3,32 3,46	0,94	6,8	8,0
	DMSO	NCH_2 5,39 C_6H_5 6,9—7,3	6,78	5,89	5,64				2,9	1,75	4,0		7,75			3,19 3,23	0,88	7,0	9,5
VIII	DMSO	NCH_2 4,94 C_6H_5 6,8—7,4	6,65	5,88	5,52				2,7	1,7	3,0	7,46	7,15		12,71				

		δ, ppm				J, Hz					
			5-H	4-H	2-H	1,2	1,5	1,4	2,3	5,4	4,2
Ia	DMSO		6,79	6,34	6,97	3,0	2,3	2,6	3,0	1,8	1,6

keto form and that strong intermolecular interactions are present. However, one cannot exclude the possibility that the presence of the cyclic lactol form is also possible in a solution of keto acid I. A shoulder at 1740 cm^{-1} appears on the intense band at 1710 cm^{-1} in the spectra of concentrated solutions of I, IVa, and Va in chloroform (Table 1); when the solution is diluted gradually, the intensity of this shoulder increases, and the component at 1745 cm^{-1} with a weak shoulder at 1710 cm^{-1} dominates in the case of a concentration of $5 \cdot 10^{-5}$ mole/liter. Thus, keto acids I, IVa, and Va form dimers involving the carboxy group in the condensed phase and in concentrated $CHCl_3$ solutions. Judging from the difference in the frequencies of the monomer and dimer ($\Delta\nu = 35$ cm^{-1}), these dimers can be assumed to be cyclic [5]. The carboxy group shows up in the spectra of solutions of I in dimethyl sulfoxide (DMSO) and acetonitrile in the form of a single band with frequencies of 1715 and 1735 cm^{-1} (Table 1). The absence of absorption bands above 1750 cm^{-1} constitutes evidence that keto acids I, IVa, and Va do not produce a lactol form in any of the investigated solvents.*

It should be noted that not only the carboxy group but also the α-keto group, which at a concentration of $5 \cdot 10^{-3}$ mole/liter has the form of a doublet at 1606 and 1637 cm^{-1}, the low-frequency component of which becomes weaker when the solution is diluted, forms strong intermolecular hydrogen bonds in dilute solutions of keto acid I in chloroform. Since this doublet character of the carbonyl group is not present in the spectra of keto acids IVa and Va, it may be concluded that the pyrrole hydrogen atom participates in this intermolecular hydrogen bond. This conclusion is also confirmed by the low-field position of the signal of the proton of the NH group in the PMR spectrum of I in chloroform (Table 2).

Cyclic pyridazinones are formed in the reaction of acids I and Va and γ-(2-pyrrolylketo)butyric acid with hydrazine hydrate.

The reduction of acid Va with zinc dust in ammonia leads to o-(N-benzyl-2-pyrrolylmethyl)benzoic acid (VIII).

We were able to isolate derivatives (VI and VII) of hydroxy lactone form III of the acids by reaction of keto acids I and Va with ethyl chlorocarbonate in the presence of cyclizing agents, viz., zinc chloride and boron trifluoride etherate. In addition, the reduction of keto acids I and IVa with sodium borohydride leads to 2-pyrrolylbenzolactones IX and X, which were isolated by elution with silica gel. All of the compounds obtained are stable white crystalline substances, the UV spectra of which differ considerably from the spectra of keto acids I and IVa: In addition to a maximum at 206 nm, a broad diffuse maximum with approximately the same intensity is observed at 230-235 nm. The IR spectra of these compounds contain a strong band at 1760-1775 cm^{-1}. The data from the PMR spectra of VI and VII and a comparison of them with the spectra of the corresponding keto acids (Table 2) confirm the structures proposed for these compounds.

We made the assignments of the signals to the protons of the pyrrole ring from the spin–spin coupling constants (SSCC), assuming that $J_{3,4} > J_{2,3} > J_{2,4}$ [6]. The assignment of the signals to the protons of the benzene ring was made with allowance for the change in the hybridization of the carbon atom of the carbonyl group from sp^2 in keto acids I, IVa, and Va to sp^3 in the cyclic lactol form of VI-VIII. In the case of sp^3 hybridization of the bridge C_6 atom only the 5'-H proton will experience additional deshielding as a consequence of the anisotropy of the magnetic susceptibility of the $C=O$ bond. The signals of the protons of the benzene ring in the spectra of VI-VIII therefore form two multiplets with an intensity ratio of 1:3 (we did not carry out a detailed analysis of these multiplets, and only their centers are indicated in Table 2). In the case of the spectra of I, IVa, and Va in solution in d_6-DMSO the protons under discussion give three multiplets with an intensity ratio of 1:2:1. A distinctive feature of the spectra of VI and VII is the complexity of the signals of the protons of the ethoxy group, which in this case is an ABX_3 spin system with $J_{AX} = J_{BX}$, while the $N-CH_2$ signals of the benzyl group for VII in $CHCl_3$ form an AB quartet with J = 15.4 Hz. This is probably associated with the magnetic nonequivalence of the hydroxymethylene protons of the ethoxy group, which is specifically oriented relative to the plane of the aromatic ring bonded to the chiral center, as observed for the methyl groups in 2-aryl-3-methyl-2-butanols [7].

A comparison of the chemical shifts of the protons for Ia and the absence of the $J_{3,4}$ constant (4 Hz) that is characteristic for 2-acylpyrroles provide a basis for the assertion that the side product in the reaction between pyrrolylmagnesium iodide and phthalic anhydride is actually o-(3-pyrrolylketo)benzole acid (Ia).

EXPERIMENTAL

The IR spectra were recorded with a UR-20 spectrometer: The spectra of concentrated solutions were obtained in standard KBr and CaF_2 cuvettes ($l = 0.1$ mm), while the spectra of dilute solutions were obtained in

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* Measurements in CCl_4 were difficult because of the low solubilities of the keto acids.

dismountable cylindrical cuvettes (l =10 mm). The UV spectra of solutions of the compounds in ethanol were recorded with a Specord spectrophotometer. The PMR spectra were recorded with a Varian HA-100D spectrometer with hexamethyldisiloxane as the internal standard.

2- and 3-Pyrrolylketobenzoic Acids (I and Ia). A hot solution of 14.8 g (0.01 mole) of phthalic anhydride in 100 ml of anisole was added with vigorous stirring to a solution of pyrrolylmagnesium iodide obtained by the method in [1] from 6.7 g (0.01 mole) of pyrrole, after which stirring was continued until the mixture had cooled completely to room temperature. The mixture was decomposed in the usual way, and the aqueous solution was extracted several times with ether. The combined ether extracts and the anisole layer were evaporated to a small volume, and the resulting precipitate was removed by filtration to give 14 g (63%) of a product with mp 138-140°C. A 10-g sample of the mixture obtained was applied to a column filled with L 100/250 μ silica gel and eluted with ether–petroleum ether (1:1). Workup of the eluate gave 8 g of o-(2-pyrrolylketo)benzoic acid (I), with mp 184-185°C, which was identical to the compound described in [1]. Subsequent elution with benzene–ethyl acetate (4:1) gave 0.8 g of o-(3-pyrrolylketo)benzoic acid (Ia) with mp 167°C (from water). IR spectrum: 3190, 1703, and 1605 cm^{-1} (mineral oil). UV spectrum, λ_{max} ($\varepsilon \cdot 10^3$): 206 (16.3), i 250 (10.4), and shoulder at 290 nm (5.2). Found: C 65.7; H 4.6; N 6.1%. $C_{12}H_7NO_2$. Calculated: C 67.0; H 4.2; N 6.5%.

γ-(2-Pyrrolylketo)butyric Acid. The reaction of 3.35 g (5 mmole) of pyrrole and 5 g (5 mmole) of succinic anhydride by the method described in [2] gave 3.05 g (35%) of a product with mp 140-141°C (from benzene–petroleum ether) after purification by reprecipitation from sodium carbonate solution. IR spectrum (mineral oil): 3320, 1710, and 1640 cm^{-1}. UV spectrum, λ_{max} ($\varepsilon \cdot 10^3$): 203 (0.63), sh 256 (0.47), and 287 nm (15.2). Found: C 58.0; H 5.8; N 8.0%. $C_8H_9NO_3$. Calculated: C 57.8; H 5.4; N 8.4%.

o-(N-Methyl-2-pyrrolylketo)benzoic Acid (IVa). A 16-ml (0.017 mole) sample of freshly distilled dimethyl sulfate was added with heating and stirring to a mixture consisting of 2.15 g (0.01 mole) of keto acid I in 200 ml of acetone, 20 g of KOH, and 50 ml of water, after which the mixture was heated for another 20 min. The solvent was then removed by vacuum distillation, and the aqueous solution was extracted with ether. The solvent was removed by distillation, and the residue was applied to a column filled with silica gel. Elution with ether–petroleum ether (1:1) gave 1.48 g (70%) of methyl ester IVb with mp 67°C. IR spectrum (mineral oil): 1735 and 1640 cm^{-1}. Refluxing with 10% aqueous NaOH solution and subsequent acidification gave IVa, with mp 183°C (from aqueous isopropyl alcohol), in 75% yield. The data from the IR and UV spectra are presented in Table 1. Found: C 68.2; H 5.3; N 6.5%. $C_{13}H_{11}NO_3$. Calculated: C 68.1; H 4.8; N 6.1%.

γ-(N-Methyl-2-pyrrolylketo)butyric Acid. The methylation of 1.47 g (8.7 mmole) of γ-(2-pyrrolylketo)-butyric acid with dimethyl sulfate under the conditions described above gave 0.8 g (50%) of the N-methylated acid with mp 107°C (from benzene–petroleum ether). IR spectrum (mineral oil): 1700 and 1650 cm^{-1}. UV spectrum, λ_{max} ($\varepsilon \cdot 10^3$): 203 (0.69), sh 256 (0.60), and 287 nm (15.9). Found: C 58.9; H 6.0; N 7.4%. $C_9H_{11}NO_3$. Calculated: C 59.5; H 6.1; N 7.7%.

Methyl o-(N-Benzyl-2-pyrrolylketo)benzoate (Vb). An ether solution of diazomethane obtained from 2.5 g of N-nitrosomethylurea was added to an ether solution of 0.5 g (2.4 mmole) of keto acid Va in 20 ml of absolute ether, after which the solvent was removed by distillation, and the residue was eluted with benzene–petroleum ether (1:1) in a column filled with Al_2O_3. Workup of the eluate gave 0.25 g (48%) of methyl ester Vb in the form of an oil. IR spectrum (mineral oil): 1735 and 1640 cm^{-1}. Found: N 4.12%. $C_{20}H_{17}NO_3$. Calculated: N 4.38%.

1-(2-Pyrrolyl)benz[5,6]isoxazol-4-one. A mixture of 1.1 g (5 mmole) of keto acid I, 3.5 g of hydroxylamine, and 30 ml of dry pyridine was refluxed for 6 h, after which the solution was poured over a mixture of ice and hydrochloric acid, and unchanged acid I was separated. The aqueous solution was extracted with ether, and the ether extract was washed with sodium carbonate solution and water and dried with $MgSO_4$. Evaporation of the solvent gave 0.9 g of 1-(2-pyrrolyl)benz[5,6]isoxazol-4-one with mp 203-204°C (from aqueous methanol). IR spectrum (mineral oil): 3180-3270, 1710, and 1660 cm^{-1}. UV spectrum, λ_{max} ($\varepsilon \cdot 10^3$): 234 (4.57) and 269 nm (4.17). Found: C 68.3; H 4.3; N 13.8%. $C_{12}H_8N_2O_2$. Calculated: C 68.0; H 3.7; N 13.2%.

1-(2-Pyrrolyl)benzo[5,6]pyridazin-4-one. A mixture of 0.53 g (2.4 mmole) of I, 0.5 g of hydrazine hydrate, and 15 ml of ethanol was refluxed for 6 h, after which it was cooled, and 0.5 g of 1-(2-pyrrolyl)-benzo[5,6]pyridazin-4-one, with mp 228-229°C (from alcohol), was separated. IR spectrum (mineral oil): 3430, 3360, 1670-1680, and 1590 cm^{-1}. Found: C 68.0; H 4.5; N 19.6%. $C_{12}H_9N_3O$. Calculated: C 68.2; H 4.3; N 19.9%.

1-(N-Benzyl-2-pyrrolyl)benzo[5,6]pyridazin-4-one. A total of 0.40 g (40%) of 1-N-benzyl-2-pyrrolyl)-benzo[5,6]pyridazin-4-one, with mp 263-264°C (from alcohol), was obtained by refluxing 1 g (4.9 mmole) of keto acid Va in 3 ml of hydrazine hydrate. IR spectrum ($CHCl_3$): 3420, 1675, and 1590 cm^{-1}. Found: C 73.1; H 4.8; N 13.3%. $C_{19}H_{15}N_3O \cdot 1/2\ H_2O$. Calculated: C 73.5; H 5.2; N 13.5%.

1-(2-Pyrrolyl)pyridazin-4-one. The reaction of 0.38 g (2.2 mmole) of a γ-(2-pyrrolylketo)butyric acid and 1.5 g of hydrazine hydrate in 5 ml of ethanol by the usual method gave 0.68 g (68%) of 1-(2-pyrrolyl)pyridazin-4-one with mp 226°C (dec., from methanol). IR spectrum (mineral oil): 3320 and 1660 cm^{-1}. Found: C 59.1; H 6.1; N 26.0%. $C_8H_9N_3O$. Calculated: C 58.9; H 5.6; N 25.8%.

o-(2-Pyrrolylketo)benzoic Acid Pseudoethyl Ester (VI). A 0.5-ml sample of ethyl chlorocarbonate was added to a solution of 1.07 g (5 mmole) of keto acid I in 50 ml of dry chloroform (or THF) containing 0.7 ml of triethylamine, and the mixture was stirred at room temperature for 30 min. A catalytic amount of anhydrous $ZnCl_2$ was then added, and stirring was continued at room temperature for another 2 h. The mixture was then treated with water, and the chloroform layer was washed with dilute sodium carbonate solution and water and dried with anhydrous $MgSO_4$. The solvent was removed by evaporation, and the residue was applied to a column filled with silica gel and eluted with ether–petroleum ether (1:1). The first fraction was worked up to give 10 mg of a substance that was found to be lactam II (it was identical to a genuine sample [2]). The second fraction, which gave an intense coloration with Erlich's reagent, was worked up to give 0.38 g (31%) of VI with mp 121°C (from ethanol). The data from the IR and UV spectra are presented in Table 1. Found: C 68.6; H 5.0; N 5.9%. $C_{14}H_{13}NO_3$. Calculated: C 69.1; H 5.4; N 5.8%.

o-(N-Benzyl-2-pyrrolylketo)benzoic Acid Pseudoethyl Ester (VII). Under conditions similar to those described above, 2.55 g (8.3 mmole) of keto acid Va gave 1.8 g (63%) of VII with mp 128-129°C (from alcohol). The data from the IR and UV spectra are presented in Table 1. Found: C 75.7; H 5.5; N 3.9%. $C_{21}H_{19}NO_3$. Calculated: C 75.6; H 5.7; N 4.2%.

2-(2-Pyrrolyl)benzolactone (IX). A solution of 1.1 g (5 mmole) of keto acid I in 15 ml of diglyme was added at room temperature to a solution of 1.5 g of $NaBH_4$ in 30 ml of diglyme, and the mixture was stirred for 2 h. It was then decomposed with dilute HCl and extracted with ether. The ether extract was evaporated, and the residue was applied to a column filled with silica gel. Elution with benzene yielded a fraction that gave an intense blue-violet coloration with Erlich's reagent. The solvent was removed in vacuo, and the oily residue began to crystallize after treatment with petroleum ether; the yield of lactone IX, with mp 112°C (from petroleum ether), was 0.54 g (59%). See Table 1 for the data from the IR and UV spectra. Found: C 72.5; H 5.1; N 7.0%. $C_{12}H_9NO_2$. Calculated: C 72.7; H 4.5; N 7.0%.

2-(N-Methyl-2-pyrrolyl)benzolactone (X). Similarly, 0.1 g of lactone X, with mp 88-90°C, was obtained from 0.5 g of IVa. IR spectrum ($CHCl_3$): 1760 and 1610 cm^{-1}.

o-(N-Benzyl-2-pyrrolylmethyl)benzoic Acid (VIII). A 6-g sample of zinc dust was added to a solution of 2.5 g (0.3 mmole) of keto acid Va in 100 ml of 25% NH_4OH containing a catalytic amount of copper sulfate, and the mixture was refluxed with stirring for 10 h with the addition of 50 ml of ammonium hydroxide every 2 h. The zinc dust was then removed by filtration, and the filtrate was acidified with hydrochloric acid to give 1.03 g (42%) of acid VIII with mp 140-141°C (benzene–petroleum ether). UV spectrum, λ_{max} ($\varepsilon \cdot 10^3$): 206 nm (37.0). Found: C 78.7; H 5.7; N 4.8%. $C_{19}H_{15}NO_2$. Calculated: C 78.9; H 5.2; N 4.8%.

Methyl o-(N-Benzyl-2-pyrrolylmethyl)benzoate. This compound [0.9 g (87%)], with mp 59-60°C (from petroleum ether), was obtained by the action of an ether solution of diazomethane (from 5 g of N-nitrosomethylurea) and 1 g (5 mmole) of acid VIII. UV spectrum, λ_{max} ($\varepsilon \cdot 10^3$): 212 (32.3) and sh 228 nm (12.7). IR spectrum (mineral oil): 1735 cm^{-1}. Found: C 78.6; H 6.5; N 4.1%. $C_{20}H_{19}NO_2$. Calculated: C 78.6; H 6.3; N 4.6%.

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