The following compounds were similarly obtained: p-propylphenyl 2-furyl ketone with bp 139-141°C (5 mm) and mp 48°C (mp 48°C [2]), p-isopropylphenyl 2-furyl ketone with bp 140-141°C (5 mm) and mp 50°C (mp 50-51°C [4]), p-tert-butylphenyl 2-furyl ketone with bp 146-148°C (5 mm) and mp 59°C (mp 58-59°C [4]), and p-butylphenyl 2-furyl ketone with bp 147-149°C (5 mm) and mp 65°C. Found: C 78.8; H 6.9%; $C_{15}H_{16}O_2$. Calculated: C 78.9; H 7.0%.

The competitive reactions were carried out by the following method. A mixture of 0.05 mole of anisole (or mesitylene), 0.05 mole of 2-furoyl chloride, 0.05 mole of benzoyl chloride, and 0.1 g of ferric chloride was heated at 155°C for 3 h, after which 10 ml of alkali was added to tie up the acyl chloride residues, and the mixture was extracted with benzene. The solvent was removed by distillation, and the residue was distilled in vacuo. A fraction with bp 145-155°C (3 mm) was isolated in the experiment with mesitylene, and a fraction with bp 160-190°C (3 mm) was isolated in the experiment with anisole.

The yields of the products were determined by using p-furoylanisole and 2,4,6-trimethylphenyl furyl ketone as internal standards.

LITERATURE CITED

1. A. Dunlop and E. Peters, The Furans, Reinhold Publ. Corp., New York (1953). p. 498.
2. Yu. K. Yur'ev, Z. V. Belyakov, and V. P. Volkov, Zh. Obshch. Khim., 28, 2372 (1958).
3. G. G. Galust'yan and I. P. Tsukervanik, Zh. Org. Khim., 3, 1259 (1967).
4. L. D. Smirnov, V. I. Kuz'min, L. N. Mikhailova, V. P. Levina, and K. M. Dyumaev, Izv. Akad. Nauk SSSR, Ser. Khim., No. 8, 1845 (1970).
5. I. K. Fel'dman and A. I. Sizov, Zh. Obshch. Khim., 23, 441 (1953).
6. Kh. Yu. Yuldashev, Zh. Org. Khim., 13, 2369 (1977).
7. N. V. Veber, I. P. Tsukervanik, and N. G. Sidorova, Zh. Org. Khim., 6, 529 (1970).
8. I. N. Zemzina and I. P. Tsukervanik, Zh. Org. Khim., 2, 697 (1966).
9. N. V. Veber and I. P. Tsukervanik, Zh. Org. Khim., 5, 116 (1969).

SYNTHESIS OF 4-OXO-1,3-BENZ- AND NAPHTHOXAZINIUM SALTS FROM o-HYDROXYARYLAMIDES

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The acid-catalyzed acylation of primary and secondary o-hydroxyarylamides, the probable reaction scheme, and the possibility of the formation of 4-oxo-1,3-arenooxazinium salts and analogs in the reaction of amides of salicylic, coumaric, and 1-hydroxy- and 3-hydroxynaphthoic acids and their N-alkyl(aryl)derivatives with excess amounts of aliphatic acid anhydrides and 70% perchloric acid, as well as by the acid cyclization of O- and N-mono- and diacyl derivatives of these amides, are examined. Twelve previously unknown 2-alkyl- and 2-styryl-4-oxo-1,3-naphth[3,2-e]- and -[1,2-e]oxazinium perchlorates were synthesized. N-Methyl- and N-benzylamides of 8-acetoxy-1-naphthoic acid are formed instead of the expected peri-cyclic analogs of oxazinium salts in the reaction of 8-hydroxy-1-naphthoic acid anilides with acetic anhydride and perchloric acid.

The reaction of salicylamide and N-substituted salicylamides with alkanecarboxylic acids and perchloric acid leads to the production of 4-oxo-1,3-benzoxazinium perchlorates [1]. To ascertain the effect of benzo annelation on the stabilities and properties of 4-oxo-1,3-oxazinium cations in the present research we used the same method for the preparation of a number of previously unknown 2-alkyl-4-oxo-1,3-naphth[3,2-e]- and -[1,2-e]oxazinium salts (I, II). The synthesized naphthoxazinium perchlorates (I-II) are less stable than

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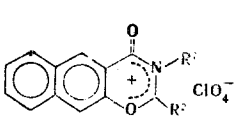
TABLE 1. 4-Oxo-1,3-benz- and Naphthoxazinium Perchlorates

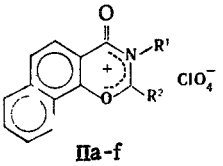
Compound	mp, °C	Found, %				Empirical formula	Calculated, %				Yield, %
		C	H	Cl	N		C	H	Cl	N	
I a	228[a]	49,8	3,6	10,9	4,1	$C_{13}H_{10}ClNO_6$	50,1	3,2	11,4	4,5	62
I b	210	51,1	4,0	11,5	4,0	$C_{14}H_{12}ClNO_6$	51,6	3,7	11,0	4,3	50
I c	192—193	58,4	4,0	9,7	3,3	$C_{19}H_{14}ClNO_6$	58,8	3,6	9,4	3,6	80
I d	140	59,4	4,1	9,2	3,1	$C_{20}H_{18}ClNO_6$	59,5	4,5	8,8	3,5	82
I e	234—236[a]	60,3	3,5	8,4	3,1	$C_{20}H_{14}ClNO_6$	60,1	3,5	8,9	3,5	75
I f	Dec. >98	60,5	4,2	8,4	3,7	$C_{21}H_{16}ClNO_6$	60,9	3,9	8,6	3,5	40
II a	211[a]	50,3	3,6	11,0	4,1	$C_{13}H_{10}ClNO_6$	50,1	3,2	11,4	4,5	90
II b	Dec. >103	58,4	3,9	9,7	3,3	$C_{19}H_{14}ClNO_6$	58,8	3,6	9,4	3,6	55
II c	Dec. >72	60,1	4,8	8,2	3,0	$C_{20}H_{18}ClNO_6$	59,5	4,5	8,8	3,5	42
II d	167—169	52,1	4,1	10,7	4,1	$C_{14}H_{12}ClNO_6$	51,6	3,7	11,0	4,3	51
II e	181	53,6	4,5	10,0	3,7	$C_{15}H_{14}ClNO_6$	53,1	4,1	10,5	4,1	47
II f	Dec. >230[a]	61,1	3,7	8,4	3,6	$C_{21}H_{16}ClNO_6$	60,9	3,9	8,6	3,4	81
III a	200—203	53,2	3,8	11,0	4,0	$C_{15}H_{12}ClNO_6$	53,4	3,6	10,5	4,2	93
III b[b]	Dec. >100	47,8	4,9	11,3	4,8	$C_{12}H_{14}ClNO_6$	47,4	4,6	11,7	4,6	55
III c	Dec. >160	51,8	4,5	10,1	7,1	$C_{16}H_{15}ClN_2O_6$	52,4	4,1	9,7	7,6	35
III d	240—241[c]	46,6	2,7	20,2	3,6	$C_{14}H_9Cl_2NO_6$	46,9	2,5	19,8	4,0	90

[a]Crystallization from glacial acetic acid and nitromethane with the addition of two to three drops of 70% $HClO_4$. [b]Perchlorates IIIb-d were obtained by cyclization of the corresponding N-acylsalicylamides [6]. [c]From nitromethane.

their benzo analogs, and most of them undergo hydrolytic cleavage, which is often accompanied by deacylation, during attempts to recrystallize them.



I a-f



IIa-f

I **a** R^1=H, R^2=CH_3; **b** R^1=H, R^2=C_2H_5; **c** R^1=C_6H_5, R^2=CH_3; **d** R^1=C_6H_5, R^2=n-C_3H_7; **e** R^1=H, R^2=CH=CHC_6H_5; **f** R^1=H, R^2=C(=CH_2)-CHC_6H_5; II **a** R^1=H, R^2=CH_3; **b** R^1=C_6H_5, R^2=CH_3; **c** R^1=C_6H_5, R^2=n-C_3H_7; **d** R^1=CH_3, R^2=CH_3; **e** R^1=CH_3, R^2=C_2H_5; **f** R^1=CH_3, R^2=CH=CHC_6H_5

Absorption bands of the C=O group that are characteristic for the oxooxazinium cation [1] are present in the IR spectra of salts I and II at 1770 cm^{-1}. The 8a and 8b absorption bands (in the Wilson numbering system [2]) of the symmetrical and asymmetrical stretching vibrations of the aromatic rings in the spectra of I are shifted ∿ 15 cm^{-1} to the high-frequency region as compared with benzoxazinium salts. The same bands in the spectra of salts II experience the somewhat greater shift (up to 25 cm^{-1}) that is characteristic for o-substituted benzenes [3] and naphthalenes that contain electron-acceptor substituents in the 1 and 2 positions [4]. Two bands of medium intensity, which are probably due to the skeletal vibrations of the oxazinium cation, are present in the spectra of the nephth- and benzoxazinium salts at 1510-1580 cm^{-1}.

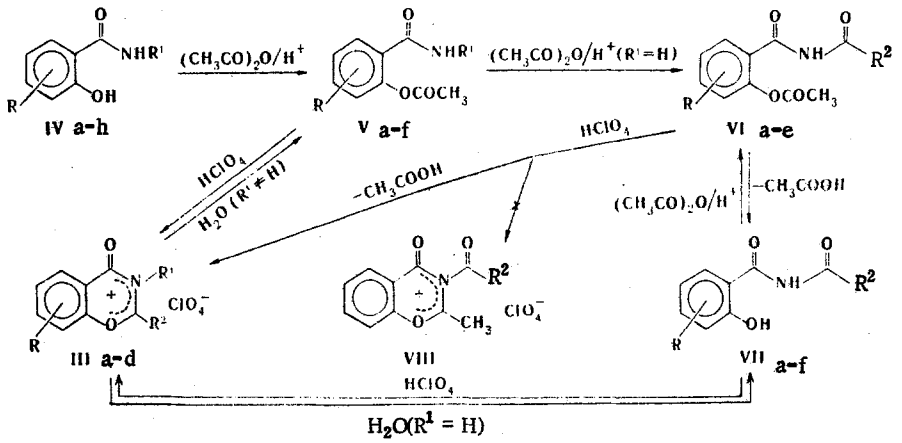
Naphthoxazinium perchlorates I and II display CH-acid properties of the methyl (methylene) group in the 2 position and react with benzaldehyde to give styryl derivatives (Ie, f and IIf, respectively).

In the present research we also examined the possibility of the formation of 4-oxo-1, 3-arenooxazinium salts (III) from the corresponding o-hydroxyarylamides and their acyl derivatives.

It was established that the phenolic or naphtholic hydroxy group is acylated in the reaction of N-alkyl(aryl) and N-acyl derivatives (IV and VII) of salicylamide and of anilides of hydroxynaphthoic acids with aliphatic acid anhydrides in the presence of catalytic

amounts of perchloric acid at room temperature. However, the amino groups also undergo acylation in the case of primary amides. For example, salicylamide forms a mixture of O-acetyl and O,N-diacetyl derivatives (Va and VIa) in a ratio of 3:1 under these conditions, and O,N-diacylamides VI were obtained as the principal products in the acid-catalyzed acylation of primary amides of naphtholcarboxylic acids.

O-Acyl derivatives V undergo cyclization in glacial acetic acid under the influence of equimolar amounts of anhydrous [5] perchloric acid to give the corresponding oxooxazinium salts III. O-Aroyl derivatives of secondary amides remain unchanged under similar conditions. These compounds undergo transacylation in excess acetic anhydride to give 2-methyl-oxooxazinium perchlorates. Thus, the heretofore unknown 2-aryl-4-oxo-1,3-arenooxazinium salts III with substituents in the 3 position cannot be obtained by this method.



III **a-d** R=H; a $R^1=C_6H_5$, $R^2=CH_3$; **b** R^1=H, R^2= iso-C_4H_9; **c** R^1=H, R^2=4-$(CH_3)_2NC_6H_4$; **d** R^1=H, R^2=4-ClC_6H_4; IV a R=R^1=H; **b** R=H, $R^1=CH_3$; **c** R=H, $R^1=C_6H_5$; **d** R=3,4-**benz**, R^1=H; **e** R=4,5-**benz**, R^1=H; **f** R=3,4-**benz**, $R^1=CH_3$; **g** R=3,4 **benz**, $R^1=C_6H_5$; **h** R=4,5-**benz**, $R^1=C_6H_5$; V **a** R=R^1=H; **b** R=H, $R^1=CH_3$; **c** R=H, $R^1=C_6H_5$; **d** R=3,4-**benz**, $R^1=CH_3$; **e** R=3,4-**benz**, $R^1=C_6H_5$; **f** R=4,5-**benz**, $R^1=C_6H_5$; VI **a** R=H, $R^2=CH_3$; **b** R=H, $R^2=C_6H_5$; **c** R=H; R^2=4-$CH_3OC_6H_4$; **d** R=3,4-**benz**, $R^2=CH_3$; **e** R=4,5-**benz**, $R^2=CH_3$; VII **a** R^1=H, $R^2=CH_3$; **b** R=H, $R^2=C_6H_5$; **c** R=H, R^2=4-$CH_3OC_6H_4$; **d** R=3,4-**benz**, $R^2=CH_3$; **e** R=4,5-**benz**, $R^2=CH_3$; **f** R=4,5-**benz**, $R^2=C_2H_5$

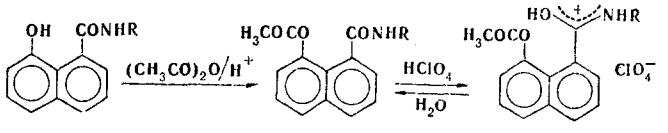
Thus, the assumption of the formation of N-acylamides [1] as intermediates in the synthesis of 4-oxo-1,3-benzoxazinium salts along with O-acylamides is evidently valid only for primary o-hydroxyarylamides. This is promoted by the formation of O,N-diacyl derivatives in the acid-catalyzed acylation of primary o-hydroxyarylamides and the ease of conversion of the latter to N-acylamides. Thus, for example, hydroxynaphthoic acid O,N-diacylamides VId,e split out an O-acyl residue and are converted to the corresponding N-acylamides VII when they are heated in organic solvents (during attempts to recrystallize them), while O,N-diacetylsalicylamide (VIa) undergoes this transformation on standing in solution.

Salts III, which do not contain acyl substituents in the 3 position of the heteroring, were obtained from O,N-diacyl derivatives VI. Cyclization in this case is accompanied by deacylation of the N-acyl residue (in the form of benzoic or anisic acid from VIb,c), and the formation of N-acyl-substituted salts VIII cannot be established. Attempts to synthesize these compounds by direct acylation of 2-phenyl-4-oxo-1,3-benzoxazine with acetyl and benzoyl perchlorates were also unsuccessful. The perchlorate of the starting oxazinone was isolated instead of the expected VIII.

The cyclization of N-acyl- and particularly N-aroylsalicylamides VII in a mixture of acetic acid and anhydride under the influence of perchloric acid is a convenient method and sometimes the only method for the preparation of salts III. However, in this case one should take into account the possibility of acylation of the phenolic hydroxy group with the subsequent formation of 2-methyl salts and should avoid the use of excess anhydride.

Thus, the facts noted above constitute evidence that the more probable step in the synthesis of salts III from o-hydroxyarylamides is the cyclization of their O-acyl derivatives.

We also made attempts to synthesize peri-cyclic analogs of oxazinium salts III from 8-hydroxy-1-naphthoic acid amides IXa, b. However, stable perchlorates XI have noncyclic structures, as indicated by the doublet splitting of the signals of the methyl and methylene protons in their PMR spectra. Under the influence of water, perchlorates XI are converted to the corresponding 8-acetoxy-1-naphthoic acid N-alkylamides X, which are also obtained by the acid-catalyzed acetylation of hydroxy amides IX. The starting amides were isolated in the case of treatment of salts XI with concentrated ammonium hydroxide.



IX X XI

IX-XI a R = CH_3; b R = $CH_2C_6H_5$

We were also unable to obtain vinyl analogs of the oxazinium salts from coumaric acid amide. Under catalytic acetylation conditions we obtained 2-acetoxycinnamic acid N-acetylamide, which is converted by the action of equimolar amounts of perchloric acid to the oily perchlorate, which is slowly hydrolyzed to the starting coumaric acid amide in water.

EXPERIMENTAL

The PMR spectra of solutions of IX and XI in trifluoroacetic acid and of X in deuterchloroform were recorded with a Tesla BS 467 spectrometer (60 MHz) at 20°C with hexamethyldisiloxane as the internal standard. The IR spectra of mineral oil suspensions were recorded with a UR-20 spectrometer.

2-Methyl-4-oxo-1,3-naphth[3,2-e]oxazinium Perchlorate (Ia). A 1-ml sample of 70% perchloric acid was added to a suspension of 1.87 g (0.01 mole) of 3-hydroxy-2-naphthoic acid amide in 6 ml of acetic anhydride, and the mixture was heated carefully to the boiling point. It was then cooled and diluted gradually with a threefold amount of ether, and the resulting precipitate was removed by filtration, washed successively with a small amount of acetic acid (or ethyl acetate) and four times with ether to give 1.93 g (62%) of a product with mp 228°C (dec.).

Perchlorates Ib-d, IIa-e, and IIIa were synthesized under similar conditions (Table 1).

2-Methyl-4-oxo-1,3-naphth[1,2-e]oxazinium Perchlorate (IIa). This compound was obtained by the method in [7]. A 0.5-ml sample of 70% perchloric acid was added to a mixture of 0.85 g (5 mmole) of α-hydroxynaphthoic acid nitrile in 4 ml of acetic anhydride, and the mixture was heated to the boiling point. It was then cooled and gradually diluted with a threefold amount of ether. The resulting precipitate was removed by filtration and washed four times with ether to give 1.14 g (73%) of the salt with mp 211°C (dec., from glacial acetic acid).

2-Styryl-4-oxo-1,3-naphth[3,2-e]oxazinium Perchlorate (Ie). A suspension of 1.56 g (5 mmole) of perchlorate Ia, 0.64 g (6 mmole) of benzaldehyde, 3 ml of acetic anhydride, and 5 ml of glacial acetic acid was heated rapidly to the boiling point, after which the mixture was cooled and diluted with a small amount of ether. The product was removed by filtration and washed three times with the minimum amount of glacial acetic acid and four times with ether to give 1.56 g (78%) of the salt with mp 234-236°C (dec.).

Perchlorates If and IIf were similarly obtained.

O-Acetylsalicylamide (Va). Three drops of 70% perchloric acid were added to a suspension of 4.11 g (0.03 mole) of salicylamide in 15-20 ml of acetic anhydride, and the mixture, which began to crystallize after 30 min, was diluted with water. The resulting precipitate was removed by filtration and dried to give 3.22 g (60%) of the amide with mp 138°C (from ethyl acetate) (mp 138°C [8]). IR spectrum: 3422 and 3190 (NH_2), 1760 ($OCOCH_3$), and 1690 and 1640 cm^{-1} (amide I and II). The filtrate was extracted with ether, and the extract was worked up to give 1.33 g (20%) of O,N-diacetylsalicylamide (VIa) with mp 67-68°C (from ether) [9].

The corresponding amide acetates Vd-f, VI, and X were similarly obtained in 50-95% yields in the acid-catalyzed mono- and diacylation of 1-hydroxy- and 3-hydroxy-2-naphthoic and

TABLE 2. O- and N-Acyl Derivatives of Salicylic and Hydroxynaphthoic Acid Amides

Compound	mp, °C	Crystallization solvent	IR spectra (characteristic frequencies), cm^{-1}				Found, %			Empirical formula	Calculated, %		
			NH	$OCOCH_3$	CO—NHR for V and X; CO—NH—CO for VI and VII	Aromatic C=C	C	H	N		C	H	N
Vd	105—108		3300	1770	1650 1548	1609 (sh)	69,4	5,2	5,5	$C_{14}H_{13}NO_3$	69,1	5,4	5,8
Ve	104—107		3320	1760	1656 1542	1603 1582 (sh)	75,0	4,6	4,4	$C_{19}H_{15}NO_3$	74,7	4,9	4,6
Vf	156—157	Benzene + hexane	3380	1761	1667 1530	1607	74,9	4,8	4,3	$C_{19}H_{15}NO_3$	74,7	4,9	4,6
Xa	193[a]		3280	1773	1645 1568	1588	69,0	5,5	6,8	$C_{14}H_{13}NO_3$	69,1	5,4	6,9
Xb	130—131[b]	Alcohol	3312	1765	1639 1535	1624 1596	74,9	5,3	4,6	$C_{20}H_{17}NO_3$	75,2	5,3	4,4
VIa	67—68[c]	Ether	3270 3160	1772	1736 1688 1515	1610 1585				$C_{11}H_{11}NO_4$			
VIb	116—117[d]	Acetone + hexane	3320	1774	1730 1690 1520	1610 1588	67,8	4,8	5,1	$C_{16}H_{13}NO_4$	67,8	4,6	4,9
VIc	155—158		3340	1775	1726 1687 1496	1611 1588	64,6	4,6	4,6	$C_{17}H_{15}NO_5$	65,0	4,8	4,4
VId	140—142	Reprecipitated from toluene by the addition of petroleum ether	3310 3185	1754	1710 1655 1582	1641 1605	66,8	5,0	4,9	$C_{15}H_{13}NO_4$	66,4	4,8	5,2
VIe	153	Benzene + petroleum ether	3264 3180	1768	1732 1665 1510	1640 1605	66,9	4,6	4,8	$C_{15}H_{13}NO_4$	66,4	4,8	5,2
VIId	166	Alcohol	3320 3180		1715 1648 1500	1605 1584	68,3	5,2	6,0	$C_{13}H_{11}NO_3$	68,1	4,9	6,1
VIIe	175—177	Aqueous alcohol	3285		1728 1660 1527	1639 1605	68,4	5,2	6,3	$C_{13}H_{11}NO_3$	68,1	4,9	6,1
VIIf	163—165	Reprecipitated from dimethylformamide by the addition of water	3260 (br)		1724 1668 1530	1640 1610	69,4	5,3	5,4	$C_{14}H_{13}NO_3$	69,1	5,4	5,7

[a]PMR spectrum: 2.10 (3H, s, $OCOCH_3$), 2.78 (3H, d, J = 5 Hz, NCH_3), and 6.60-7.70 ppm (6H, m, ArH). [b]PMR spectrum: 1.88 (3H, s, $OCOCH_3$), 4.25 (2H, d, J = 6 Hz, NCH_2—), and 6.38-7.62 ppm (11H, m, ArH). [c]This compound had mp 67-68°C [9]. [d]This compound had mp 124°C [8].

8-hydroxy-1-naphthoic acid amides IVd-h and IX and N-acetyl (aroyl) salicylamides VIIa-c (see Table 2).

1-Hydroxy- and 3-hydroxy-2-naphthoic acid anilide O-acetates Ve, f were also obtained by treatment of perchlorates Ic and IIb with water. Similarly, the hydrolysis of the corresponding benzoxazinium salts leads to N-methylsalicylamide acetate (Vb, mp 92.5-94°C) and salicylanilide acetate (Vc, mp 136-137°C), to which N-acyl derivative structures were previously assigned [1]. 1-Hydroxy- and 3-hydroxy-2-naphthoic acid N-acylamides (VII) were obtained by treatment of 2-alkyl-4-oxonaphthoxazinium salts (R^1 = H) with water.

8-Hydroxy-1-naphthoic Acid N-Methylamide (IXa). This compound, with mp 168-169°C (from aqueous alcohol), was obtained in 97% yield by brief passage of dry methylamine through a benzene solution of naphtholactone [10]. IR spectrum: 3315, 3200, 1638, 1600, and 1529 cm^{-1}. PMR spectrum: 2.88 (3H, broad s, NH—CH_3) and 6.50-7.70 ppm (6H, m, ArH), PMR spectrum (in nitrobenzene): 3.1 ppm (3H, d, J = 5.25 Hz, NH—CH_3). Found: C 71.2; H 5.6; N 7.2%. $C_{12}H_{11}NO_2$. Calculated: C 71.6; H 5.5; N 7.0%.

8-Hydroxy-1-naphthoic Acid N-Benzylamide (IXb). This compound, with mp 180-181°C, was obtained in 100% yield by heating equimolar amounts of naphtholactone and benzylamine in benzene on a water bath. IR spectrum: 3290, 3190, 1620, 1583, 1555, and 1523 cm^{-1}. PMR spectrum: 4.5 (2H, broad s, NH—CH_2—) and 6.58-7.75 ppm (11H, m, ArH). Found: C 77.7; H 5.6; N 5.2%. $C_{18}H_{15}NO_2$. Calculated: C 78.0; H 5.4; N 5.0%.

8-Acetoxy-1-naphthoic Acid N-Methylamide Perchlorate (XIa). This compound, with mp 170-172°C (from glacial acetic acid), was obtained in 56% yield by treatment of amide IXa in excess acetic anhydride with 70% perchloric acid (1:7:1). The mixture was allowed to stand for 1-1.5 h, after which it was diluted with ether. IR spectrum: 3360, 1772, 1662, 1631, 1600, 1550, and 1210 cm^{-1}. PMR spectrum: 2.10 (3H, s, $OCOCH_3$), 4.25 (3H, d, J = 5 Hz, NCH_3), and 7.00-8.12 ppm (6H, m, ArH). Found: C 52.0; H 4.1; Cl 10.4%. $C_{14}H_{12}ClNO_6$. Calculated: C 51.6; H 3.7; Cl 10.9%.

8-Acetoxy-1-naphthoic Acid N-Benzylamide Perchlorate (XIb). This compound, with mp 161-161.5°C (from glacial acetic acid), was similarly obtained in 60% yield after the reaction mixture was allowed to stand overnight. IR spectrum: 3300, 1777, 1660 (sh), 1640, 1600, 1535, and 1195 cm^{-1}. PMR spectrum: 1.75 (3H, s, CH_3), 4.25 (2H, d, J = 6 Hz, NH—CH_2—), and 6.92-7.75 ppm (11H, m, ArH). Found: C 60.6; H 4.4 Cl 8.1%. $C_{20}H_{16}ClNO_6$. Calculated: C 59.8; H 4.0; Cl 8.8%.

2-Acetoxycinnamic Acid N-Acetylamide. This compound, with mp 158°C (from benzene), was obtained in 92% yield by acid-catalyzed diacetylation of coumaric acid amide [11]. IR spectrum: 3310 and 3218 (NH), 1771 ($OCOCH_3$), 1731 and 1696 (CO—NH—CO), 1663 (—CH=CH—), 1610 (sh), and 1588 cm^{-1}. PMR spectrum (in CF_3COOH): 2.10 (6H, d, $OCOCH_3$ and $NHCOCH_3$), 6.45-7.40 (5H, m, phenylene and α-vinyl protons), 7.62 (1H, d, J_{trans} = 16.5 Hz, β-CH), and 9.65 ppm (s, partially exchanged NH). Found. C 67.1; H 5.5%. $C_{13}H_{13}NO_4$. Calculated: C 67.5; H 5.6%.

In contrast to their acetates V, VI, and X, naphthols VII and IX give a greenish-blue coloration with an alcohol solution of ferric chloride.

LITERATURE CITED

1. Yu. I. Ryabukhin, V. V. Mezheritskii, and G. N. Dorofeenko, Khim. Geterotsikl. Soedin., No. 4, 460 (1975).
2. E. W. Wilson, Phys. Rev., 45, 706 (1934).
3. L. M. Sverdlov, M. A. Kovner, and E. P. Krainov, Vibrational Spectra of Polyatomic Molecules [in Russian], Nauka, Moscow (1970), p. 302.
4. I. K. Korobeinicheva and T. F. Ardyukova, Atlas of the Spectra of Aromatic and Heterocyclic Compounds [in Russian], Vol. 2, Nauka, Novosibirsk (1971), pp. 114, 115.
5. G. N. Dorofeenko, Yu. A. Zhdanov, V. I. Dulenko, and S. V. Krivun, Perchloric Acid and Its Compounds in Organic Synthesis [in Russian], Izd. Rostovsk. Univ., Rostov-on-Don (1965), p. 54.
6. Yu. I. Ryabukhin, Master's Dissertation, Rostov-on-Don (1975).
7. G. N. Dorofeenko, Yu. I. Ryabukhin, and V. V. Mezheritskii, Khim. Geterotsikl. Soedin., No. 6, 742 (1976).
8. M. Connan and A. Titherley, J. Chem. Soc., 89, 1333, 1338 (1906).
9. A. Titherley and W. Hicks, J. Chem. Soc., 99, 869 (1911).
10. A. J. Birch, M. Salahnd-Din, and D. C. C. Smith, J. Chem. Soc., C, 523 (1966).
11. R. Weermann, Rec. Trav. Chim., 37, 14 (1918).