which darken at 270° and do not melt at 310°. Heating this compound, 10 mg. in 2 ml. of 2 *N* sodium hydroxide, gives a product with an ultraviolet absorption spectrum identical with that previously found for 3-mercapto-5-hydroxy-6-methyl-1,2,4-triazine.

Anal. Calcd. for $C_4H_6N_4S$: C, 33.8; H, 4.22; N, 39.4. Found: C, 33.94; H, 4.44; N, 39.13.

5-Amino-3-hydroxy-6-methyl-1,2,4-triazine (6-Aza-5-methylcytosine) (XIII).—Ten grams of the above compound was dissolved in 500 ml. of 0.02 *N* sodium hydroxide. To this was added slowly 12.5 g. of potassium permanganate dissolved in 250 ml. of water. The manganese dioxide was removed, washed well with water and neutralized by the addition of glacial acetic acid. The neutral solution was taken to dryness *in vacuo*. The residue was recrystallized from about 50 ml. of water, to give 5.7 g. of a colorless material XII. To 500 mg. of this white crystalline material was added 11.5 ml. of 0.4 *N* hydrochloric acid and the mixture was allowed to stand at room temperature for two days. An odor of SO_2 was detected and white crystals slowly deposited. Those (200 mg.) were collected and washed with a few ml. of water. The compound was recrystallized from 5 ml. of water and melted with decomposition at 327°.

Anal. Calcd. for $C_4H_6N_4O$: C, 38.1; H, 4.76; N, 44.4. Found: C, 38.6; H, 4.76; N, 44.87.

5-Amino-6-methyl-3-methylmercapto-1,2,4-triazine (XIV).—To 5 g. of 5-amino-3-mercapto-6-methyl-1,2,4-triazine in 100 ml. of 0.35 *N* alkali there was added with shaking 3.4 ml. of dimethyl sulfate over a period of 30 minutes. The precipitate which formed was removed and crystallized from methanol–ether–petroleum ether to form colorless plates melting at 164–165°.

Anal. Calcd. for $C_5H_8N_4S$: C, 38.5; H, 5.1. Found: C, 38.1; H, 4.92.

Treatment of 5-Amino-6-methyl-3-methylmercapto-1,2,4-triazine with 0.1 *N* Sodium Hydroxide.—To 500 mg. of the above compound there was added 20 ml. of 0.1 *N* sodium hydroxide and this mixture was boiled for 90 minutes at reflux temperature (until the evolution of ammonia ceased). A pale yellow crystalline compound was obtained which melted at 224–225°. The ultraviolet absorption spectrum is identical with that of the compound given below.

5-Hydroxy-6-methyl-3-methylmercapto-1,2,4-triazine (XV).—The 5-hydroxy-3-mercapto-6-methyl-1,2,4-triazine (20 g.) was added to 261.5 ml. of 0.114 *N* NaOH and methyl iodide (19.9 g.) was added portionwise with shaking, over one hour. After standing an additional hour the reaction mixture was adjusted to *p*H 5 by the addition of acetic acid. The precipitate which formed on cooling (13.3 g.) was recrystallized from boiling water; m.p. 222–223° dec.

Anal. Calcd. for $C_6H_7N_3OS$: C, 38.2; H, 4.45. Found: C, 37.9; H, 4.8.

TUCKAHOE, N. Y.

[CONTRIBUTION FROM LOS ALAMOS SCIENTIFIC LABORATORY, UNIVERSITY OF CALIFORNIA]

Oxazole Quaternary Salts[1]

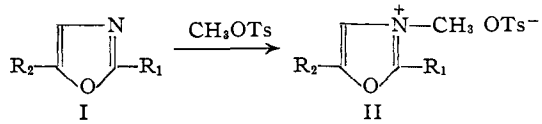
BY DONALD G. OTT, F. NEWTON HAYES AND VERNON N. KERR

RECEIVED SEPTEMBER 29, 1955

2,5-Disubstituted-3-methyloxazolium salts have been prepared for pharmacological investigations of their notable inhibition of thermoregulation in animals. General procedures are described for the anions tosylate, perchlorate, iodide and chloride. Hydrolytic studies on the oxazolium ring ion have been carried out. Ultraviolet absorption maxima and representative infrared absorption data are given.

The original interest in 3-methyloxazolium salts in the Biomedical Research Group of this Laboratory arose in connection with an extensive study of liquid solution scintillators. Observations[2] that certain of these compounds seemed to produce specific inhibition of thermoregulation in animals led to the synthesis of a number of additional salts. It appears that a new, previously overlooked series of compounds has been opened to pharmacological research.

In Table I are listed the new oxazolium salts, as well as 2,5-diphenyl-3-methyloxazolium iodide[3] and those given in an earlier communication.[4] The methyl *p*-toluenesulfonate salts (tosylates) are readily formed by heating the oxazole with an excess of methyl tosylate. Salts containing other anions were prepared by taking advantage of solubility characteristics. Oxazolium perchlorates precipitate when an aqueous solution of the tosylate is treated with perchloric acid. The perchlorate salts are soluble in acetone, and therefore the metathetical reaction with sodium iodide may be used to produce the acetone-insoluble iodides. Chloride salts may be prepared from the iodides through consideration of the difference in solubility of silver iodide ($K_{sp} = 1.5 \times 10^{-16}$) and silver chloride ($K_{sp} = 1.0 \times 10^{-10}$); an aqueous solution of the iodide salt is stirred with a slurry of silver chloride which precipitates silver iodide. The chloride salt is isolated by evaporation of the filtered reaction mixture. Another method which provides methyl chloride salts of amines without the necessity of using sealed reaction vessels and methyl chloride employs an ion exchange resin. An aqueous solution of the tosylate salt is passed through a column of anion exchange resin in the chloride form; the chloride salt is isolated by evaporation of the eluate. Presumably, this procedure is applicable to amines in general.



I II

In acidic solution the oxazolium salts are quite stable, being only slightly decomposed by refluxing for 8 hours with 48% hydrobromic acid. In dilute base, however, they are rapidly and quantitatively converted at room temperature to the N-methyl-α-acylamido ketone by hydrolytic ring

$$\text{II} \xrightarrow[(HO^-)]{H_2O} R_2COCH_2N(CH_3)COR_1$$

IV

(1) Work performed under the auspices of the U. S. Atomic Energy Commission.

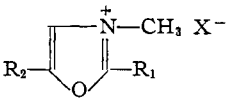
(2) C. C. Lushbaugh, F. N. Hayes, W. H. Langham, D. G. Ott and P. C. Sanders, *J. Pharm. Exptl. Therap.*, **116**, February, (1956).

(3) E. Fischer, *Ber.*, **29**, 208 (1896).

(4) F. N. Hayes, B. S. Rogers and D. G. Ott, THIS JOURNAL, **77**, 1850 (1955).

TABLE I

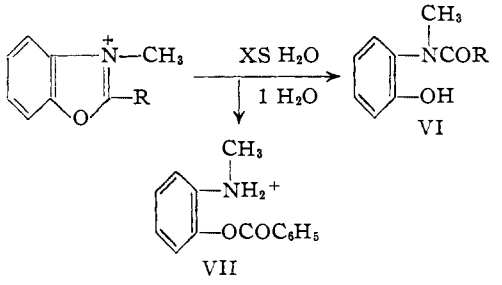
3-METHYLOXAZOLIUM SALTS



R_1	R_2	X	Formula	M.p., °C.	Analyses, % sulfur or halogen Calcd.	Found	Ultraviolet absorption maxima[a] λ_1, mμ	$\epsilon_1 \times 10^{-4}$	λ_2, mμ	$\epsilon_2 \times 10^{-4}$
C_6H_5	C_6H_5	OTs^b	[c]	.....	...	...	298	2.00	241	1.30
C_6H_5	C_6H_5	ClO_4	[c]	.....	...	...	299	1.93	242	1.24
C_6H_5	C_6H_5	I	[d]	200 d.	...	...	299	2.04	228	2.23
C_6H_5	C_6H_5	Cl	$C_{16}H_{14}ClNO$	180–183	13.05	13.39	298	1.84	241	1.20
C_6H_5	4-$C_6H_5C_6H_4$	OTs	$C_{29}H_{25}NO_4S$	199–201	6.63	6.50	316	2.87	273sh	1.7
1-$C_{10}H_7$	C_6H_5	OTs	[c]	.....	...	...	321	1.30	270	1.17
1-$C_{10}H_7$	C_6H_5	ClO_4	$C_{20}H_{16}ClNO_5$	196–197	9.19	8.93	321	1.32	271	1.18
1-$C_{10}H_7$	C_6H_5	I	$C_{20}H_{16}INO$	195–196 d.	30.71	30.81	321	1.35	271	1.20
1-$C_{10}H_7$	C_6H_5	Cl	$C_{20}H_{16}ClNO$	206 d.	11.02	11.23	321	1.14	270	1.02
1-$C_{10}H_7$	1-$C_{10}H_7$	OTs	$C_{31}H_{25}NO_4S$	156–157	7.76	7.67	330	1.69	<235	>4
1-$C_{10}H_7$	CH_3	OTs	$C_{22}H_{21}NO_4S$	153–154	8.11	8.00	308	0.86	238	1.77
2-$C_{10}H_7$	C_6H_5	OTs	$C_{27}H_{23}NO_4S$	161–162	7.01	6.98	312	1.86	252	0.74
4-$C_6H_5C_6H_4$	4-$C_6H_5C_6H_4$	OTs	[c]	.....	...	...	338	3.30	284	2.51*
4-$C_6H_5C_6H_4$	C_6H_5	OTs	$C_{29}H_{25}NO_4S$	196–197	6.63	6.49	322	2.80	262	1.05
$C_6H_5CH{=}CH$	C_6H_5	OTs	$C_{25}H_{23}NO_4S$	246–247	7.40	7.26	277	1.03	250sh	0.88
C_6H_{11}	C_6H_5	OTs	$C_{23}H_{27}NO_4S$	156–157	7.76	7.67	263	2.13	228	1.52
4-$CH_3C_6H_4$	C_6H_5	OTs	$C_{24}H_{23}NO_4S$	174–175	7.61	7.40	304	1.25	246	1.60
3-$CH_3OC_6H_4$	C_6H_5	OTs	$C_{24}H_{23}NO_5S$	162–163	7.33	7.28	302	1.39	243	0.89
4-$CH_3OC_6H_4$	C_6H_5	OTs	$C_{24}H_{23}NO_5S$	177–178	7.33	7.03	316	2.28	253	1.49
3,4-$OCH_2OC_6H_3$	C_6H_5	OTs	$C_{24}H_{21}NO_6S$	213–215	7.10	6.89	331	1.47	254	1.31
2-ClC_6H_4	C_6H_5	OTs	$C_{23}H_{20}ClNO_4S$	148–150	7.26	7.19	286	1.58	242	1.22
3,4-$Cl_2C_6H_3$	C_6H_5	OTs	$C_{23}H_{19}Cl_2NO_4S$	215–217	6.73	6.51	305	0.34	246	1.84
2-Furyl	C_6H_5	OTs	$C_{21}H_{19}NO_5S$	194–196	8.07	8.09	326	1.17	253	1.99
2-Thienyl	C_6H_5	OTs	$C_{21}H_{19}NO_4S$	205–206	15.51	15.57	331	0.88	250	1.86
1,4-Di-[2-(3-methyl-5-phenyloxazoliumyl)]-benzene ditosylate			$C_{40}H_{36}N_2O_6S_2$	342–344	8.70	8.62	305	1.48	247	2.84
2,5-Diphenyl-3-methylthiazolium tosylate			$C_{23}H_{22}NO_3S_2$	230–233	15.12	15.00	320	1.52	270sh	1.5
2-(1-Naphthyl)-3-methylbenzoxazolium tosylate			$C_{25}H_{21}NO_4S$	182–185	7.43	7.22				
2-Phenyl-3-methylbenzoxazolium tosylate			$C_{21}H_{19}NO_4S$	120–123	8.41	8.48				

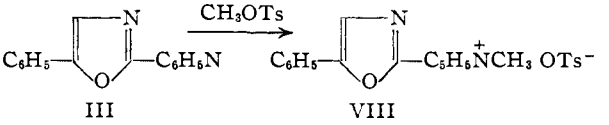
[a] An asterisk signifies 95% ethanol as solvent; otherwise, the solvent was water. [b] OTs = *p*-toluenesulfonate. [c] Previously reported in ref. 4. [d] Previously reported in ref. 3, m.p. 201° dec.; prepared here from the perchlorate.

cleavage. The amides can be further hydrolyzed with acid to N-methyl-α-aminoketones. The oxazoles themselves are not subject to such facile hydrolysis. The two benzoxazolium salts listed in Table I hydrolyze even more readily; within a few minutes after being dissolved in water (neutral solution), precipitation of the amidophenol VI takes place. 2-Phenyl-3-methylbenzoxazolium tosylate also showed a second mode of cleavage. Several months after a sample had been prepared, an



VII

infrared spectrum was recorded and found to be inconsistent with the assigned structure. Chemical evidence and analysis supported the spectrum in showing that cleavage had occurred to form an ester VII, rather than an amide. The necessary water had apparently entered through a poorly sealed cap on the vial. An analogous reaction was not pursued with the α-naphthyl derivative.



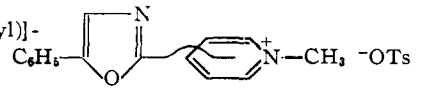
III VIII

The three isomeric 2-(*x*-pyridyl)-5-phenyloxazoles when treated with methyl tosylate gave only the *mono*tosylate salts (Table II), even when a large excess of the ester was employed. This behavior is in contrast with that of 1,4-di-[2-(5-phenyloxazolyl)]-benzene (POPOP), from which only the *di*tosylate could be obtained. It would be expected, considering the relative basicities of the oxazole and pyridine systems, that pyridinium salt formation would be preferred, and this supposition is supported by spectral data. Figure 1 shows the ultraviolet absorption spectra of 2-(4-pyridyl)-5-phenyloxazole in 95% ethanol, in 95% ethanol which is 0.1 *N* in hydrochloric acid, and of

the methyl tosylate salt. The agreement between spectra of the hydrochloride and the tosylate and the observation that the spectrum of 2,5-diphenyloxazole is not thus affected by the presence of acid show that the tosylate is of the pyridine rather than the oxazole moiety. Similar spectra were obtained from the 2- and 3-isomers, although the former was not as readily affected by hydrochloric acid. Further observations which also support a structure other than oxazolium are that the 2- and 4-isomers have an intense yellow-green fluorescence (a wave length longer than encountered with the 3-isomer or with oxazolium salts), and that they are not decomposed in basic solution.

TABLE II

[2-(5-Phenyloxazoyl)]-1-methylpyridinium Tosylates



Isomer		2	3	4
Formula		$C_{22}H_{20}N_2O_4S$	$C_{22}H_{20}N_2O_4S$	$C_{22}H_{20}N_2O_4S$
M.p., °C.		214–216	195–196	216–217
Sulfur, %	Calcd.	7.85	7.85	7.85
	Found	7.82	7.80	7.82
Ultraviolet	λ_1, mμ	360	326	371
absorpt.	$\epsilon_1 \times 10^{-4}$	2.22	1.73	2.19
max. in	λ_2, mμ	249	256	248
water	$\epsilon_2 \times 10^{-4}$	1.25	1.31	1.40

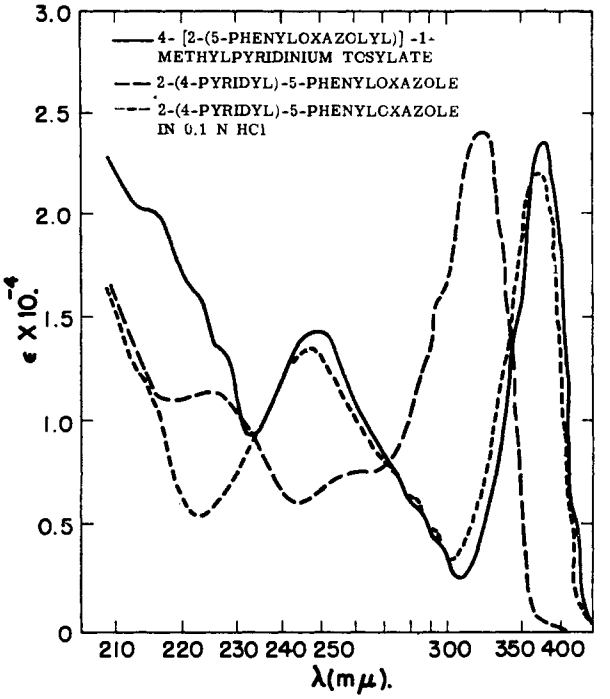
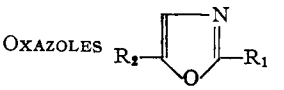


Fig. 1.—Ultraviolet absorption spectra in 95% ethanol.

TABLE III

OXAZOLES



R_1	R_2	Formula	M.p., °C.	Carbon, % Calcd.	Carbon, % Found	Hydrogen, % Calcd.	Hydrogen, % Found	Nitrogen, % Calcd.	Nitrogen, % Found	Corresponding 2-aza-1,4-diketone M.p., °C.	Corresponding 2-aza-1,4-diketone Nitrogen, % Calcd.	Corresponding 2-aza-1,4-diketone Nitrogen, % Found
2-C_5H_5N	C_6H_5	$C_{14}H_{10}N_2O$	96.5–97	75.66	75.69	4.54	4.69	12.61	12.40	126–127	11.66	11.59
3-C_5H_5N	C_6H_5	$C_{14}H_{10}N_2O$	88.5–89	75.66	75.63	4.54	4.53	12.61	12.37	144–145	11.66	11.65
4-C_5H_5N	C_6H_5	$C_{14}H_{10}N_2O$	97–97.5	75.66	75.97	4.54	4.51	12.61	12.42	152–154	11.66	11.10
1-$C_{10}H_7$	CH_3	$C_{14}H_{11}NO$	49–50	80.36	80.06	5.30	5.25	6.69	6.54	117–118	6.16	6.08

The 2,5-disubstituted oxazoles show characteristic infrared absorption about 3.35 (weak), 6.3, 6.8, 6.9, 7.45 (weak), 8.9, 10.6, and 13.0 μ. Oxazolium salt formation generally affects all of these bands, and the methyl tosylates show characteristic absorption at 3.35, 6.15, 8.3, 8.95, 9.7, 9.93, 12.3 and 14.7 μ, the 8.3 μ band being the most intense. The 6.15 μ band also appears in the spectra of the chloride, iodide and perchlorate salts; the perchlorates show in addition a broad intense band around 9.2 μ. The 3.35 μ absorption (methyl C–H) is interestingly shifted to 3.41 μ in the chloride and iodide salts, and to 3.30 μ in the perchlorates. The phenyloxazolyl-1-methylpyridinium tosylates exhibit approximately the same absorptions as the oxazolium tosylates.[5]

Experimental Part[6]

3-Methyloxazolium Tosylates (II). General Procedure.—A small amount (0.1 g. was generally used) of the oxazole was heated at 125° for approximately 5 minutes with a several-fold excess of methyl tosylate. The solution was cooled, and anhydrous ether was added to precipitate the product, which was recrystallized by dissolving in absolute alcohol and then slowly adding anhydrous ether. The yields are almost quantitative. The same procedure was used to prepare the pyridinium tosylates; attempts to prepare the *di*tosylates of the pyridyloxazoles by using a larger excess of methyl tosylate and prolonged reaction time provided only the *mono*-salts. In an attempt to prepare the *mono*tosylate salt of POPOP, equimolar quantities of the reactants were dissolved in *o*-dichlorobenzene and heated at 125° for one hour. The only salt obtained was the *di*tosylate; this compound was recrystallized from ethylene glycol.

3-Methyloxazolium Perchlorates.—To 2.03 g. (4.4 millimoles) of 2-(1-naphthyl)-3-methyl-5-phenyloxazolium tosylate, dissolved in 250 ml. of water, was added an excess of 30% aqueous perchloric acid. After standing for several hours the white solid was filtered, dried and recrystallized by precipitation with anhydrous ether from an alcohol–acetone solution. The yield was 1.68 g. (97%).

3-Methyloxazolium Iodides.—To 1.50 g. (3.9 millimoles) of the above perchlorate salt dissolved in 100 ml. of acetone was added 25 ml. of a 15% solution of sodium iodide in acetone. The iodide crystallized slowly on cooling; it was filtered, washed with acetone and recrystallized from ethanol–ether to give 1.20 g. (74%) of light-yellow needles.

3-Methyloxazolium Chlorides. Method 1.—A solution of 1.00 g. (2.42 millimoles) of the above iodide salt in 50 ml. of water was stirred for 5 hours at room temperature with several grams of freshly precipitated silver chloride. The mixture was filtered from silver iodide and excess silver chloride, and the solution was evaporated to dryness. Absolute

(5) Representative infrared absorption spectra have been deposited as Document number 4714 with the ADI Auxiliary Publications Project, Photoduplication Service, Library of Congress, Washington 25, D. C. A copy may be secured by citing the Document number and by remitting in advance $2.50 for photoprints or $1.75 for 35 mm. microfilm payable to Chief, Photoduplication Service, Library of Congress.

(6) Melting points are uncorrected. Microanalyses are by Micro-Tech Laboratories, Skokie, Ill.

ethanol was added to the residue and evaporated; the solid was recrystallized from ethanol–ether to give 0.72 g. (92%) of 2-(1-naphthyl)-3-methyl-5-phenyloxazolium chloride.

Method 2.—A solution of 1.00 g. (2.46 millimoles) of 2,5-diphenyl-3-methyloxazolium tosylate in 50 ml. of water was passed through a 1 × 30 cm. column of Dowex-1 anion exchange resin in the chloride form. The column was washed with water until small test samples indicated the absence of chloride ion. The effluate was evaporated to dryness, and the product was recrystallized from ethanol–ether. The yield of 2,5-diphenyl-3-methyloxazolium chloride was 0.45 g. (67%).

2-(4-Pyridyl)-5-phenyloxazole (III).—This procedure was also followed for the 2- and 3-isomers. Twenty grams (0.16 mole) of isonicotinic acid was refluxed with thionyl chloride.[7] The crude acid chloride which remained after removal of the excess thionyl chloride at diminished pressure was dissolved in 150 ml. of pyridine and swirled while 30.0 g. (0.16 mole) of phenacylammonium chloride was added in small portions. The mixture was heated on a boiling water-bath for 2 hours and then poured into water to precipitate the product. The solid was collected, dried and twice recrystallized from toluene–hexane to give 16.5 g. (46%) of **α-isonicotinamidoacetophenone.**

A mixture of 16.5 g. (0.073 mole) of the amide, 200 ml. of acetic anhydride and 15 ml. of 90% phosphoric acid was refluxed for 1.5 hours. After cooling, the supernatant liquid was decanted from the tarry precipitate. Several milliliters of strong aqueous sodium hydroxide solution was mixed with the residue to form a thick oil which crystallized after addition of water. The solid was filtered, dried and twice recrystallized from hexane to give 8.6 g. (57%) of 2-(4-pyridyl)-5-phenyloxazole.

Hydrolysis of 3-Methyloxazolium Salts. (a) **Ring Cleavage of II.**—To an aqueous solution of 1.0 g. (2.5 millimoles) of 2,5-diphenyl-3-methyloxazolium tosylate was added dilute ammonium hydroxide until the mixture was slightly alkaline to test paper. During this time **N-methyl-α-benzamidoacetophenone** (IV, $R_1 = R_2 = C_6H_5$) separated as a white solid; the yield was 0.62 g. (96%). Recrystallization from toluene–hexane gave white needles, m.p. 64–65°, which showed infrared absorption at 3.33, 3.45, 5.89 and 6.10 μ.

Anal. Calcd. for $C_{16}H_{15}NO_2$: C, 75.87; H, 5.97; N, 5.53. Found: C, 76.01; H, 6.05; N, 5.45.

Similarly, from 2-(1-naphthyl)-3-methyl-5-phenyloxazolium tosylate **N-methyl-α-naphthamidoacetophenone** was obtained, m.p. 102–103°; infrared absorption at 3.38, 5.85 and 6.25 μ.

Anal. Calcd. for $C_{20}H_{17}NO_2$: C, 79.19; H, 5.65; N, 4.62. Found: C, 79.27; H, 5.62; N, 4.38.

(b) **Ring Cleavage of V.**—2-Phenyl-3-methylbenzoxazolium tosylate was dissolved in water to give a neutral solution which, over a period of several hours, became quite acidic to test paper and **2-(N-methylbenzamido)-phenol** (VI, R = C_6H_5) separated in almost quantitative yield. Recrystallization from toluene–hexane gave white needles, m.p. 160–161°; infrared absorption at 3.3 (broad), 6.18, 7.35 and 7.75 μ. The compound is soluble in dilute sodium hydroxide and reprecipitated by acid.

Anal. Calcd. for $C_{14}H_{13}NO_2$: N, 6.16. Found: N, 6.01.

In a similar manner, from 2-(1-naphthyl)-3-methylbenzoxazolium tosylate, **2-(N-methyl-α-naphthamido)-phenol** was obtained, m.p. 182–183°; infrared absorption at 3.25 (broad), 6.19, 7.35 and 7.75 μ.

Anal. Calcd. for $C_{18}H_{15}NO_2$: C, 77.96; H, 5.45; N, 5.05. Found: C, 77.76; H, 5.63; N, 5.22.

A different type of cleavage occurred when a limited amount of water (apparently atmospheric moisture) was present with 2-phenyl-3-methylbenzoxazolium tosylate. An infrared absorption spectrum was recorded of a sample several months old and showed absorption at 3.4, 3.75, 4.12 (as in an amine salt), 5.75, 7.92, 8.1, 8.25, 8.95, 9.7 and 9.95 μ. The compound was soluble in water or dilute acid and insoluble in ether or cold dilute base. These properties are consistent with elemental analysis for **2-benzoxy-N-methylanilinium tosylate (VII)**, m.p. 117–118°.

Anal. Calcd. for $C_{21}H_{21}NO_5S$: S, 8.02. Found: S, 7.91, 7.92.

(c) **Complete Hydrolysis of 2,5-Diphenyl-3-methyloxazolium Tosylate.**—A solution of 1.5 g. (5.9 millimoles) of N-methyl-α-benzamidoacetophenone (from basic hydrolysis of the tosylate) in 15 ml. of ethanol and 25 ml. of concentrated hydrochloric acid was refluxed for 9 hours and then evaporated to dryness at diminished pressure. The distillate was refluxed with sodium hydroxide and then acidified to give benzoic acid, m.p. 119–120°, undepressed on admixture with an authentic sample. The solid residue was dissolved in hot absolute ethanol, and ether was added to precipitate 1.10 g. (100%) of N-methylphenacylammonium chloride, m.p. 217–218° (reported m.p. 219°),[8] after recrystallization from ethanol. The picrate derivative had m.p. 144–145° (reported m.p. 145–146°),[8] and the infrared absorption was consistent with the assigned structure.

Absorption Spectra.—Ultraviolet absorption spectra were determined at room temperature with 5×10^{-5} *M* solutions in 1-cm. silica cells using a Beckman model DK-1 recording spectrophotometer. Infrared absorption spectra were determined with a Baird Associates double-beam spectrophotometer; the samples were contained at a concentration of 0.5% in 0.5-inch diameter pressed potassium bromide disks weighing 0.15 g.

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(8) I. Heilbron, "Dictionary of Organic Compounds," Vol. 3, Oxford University Press, New York, N. Y., 1953, p. 474.