with a stirrer and a drying tube was immersed in an ice bath and a solution of 0.03 mol of anhydrous dimethylamine in 100 mL of dry benzene was added. Then 0.01 mol of compound II was added very slowly to the vigorously stirred solution in the flask. The mixture was then stirred at room temperature overnight. Water (150 mL) was added, the layers were separated, and the aqueous phase was extracted with two 100-mL portions of diethyl ether. The ether extracts were washed with saturated sodium chloride solution and dried over anhydrous magnesium sulfate. The solvent was evaporated on a rotary evaporator and an oily liquid was collected but not purified.

***Preparation of Quaternary Ammonium Salts. Compounds 1-6.*** A solution of 0.01 mol of the tertiary amine, 0.03 mol of dimethyl sulfate, and 5 g of potassium carbonate in 60 mL of acetone was refluxed for 2 h. The hot mixture was filtered and the filtrate evaporated to dryness on a rotary evaporator. The crude methosulfate salt was collected and redissolved in a minimum amount of water. Solid potassium iodide was added to the aqueous solution of the crude methosulfate salt followed by stirring with a glass rod. The iodide salt was immediately formed and filtered. The residue was dried and recrystallized from ethanol-water mixtures.

***Compounds 7-10.*** The tertiary amine (0.01 mol) was dissolved in a minimal amount of methylene chloride. Addition of 0.01 mol of methyl trifluoromethanesulfonate to the stirred solution of the amine formed the trifluoromethanesulfonate salt immediately. The salt was filtered, dried, and recrystallized from 95% ethanol.

**Registry No.** **1**, 108472-52-2; **1** (amine), 108472-66-8; **2**, 108472-53-3; **2** (amine), 108472-67-9; **3**, 108472-54-4; **3** (amine), 47502-97-6; **4**, 108472-55-5; **4** (amine), 108472-68-0; **5**, 108472-56-6; **5** (amine), 108472-69-1; **6**, 108472-57-7; **6** (amine), 108472-70-4; **7**, 108472-59-9; **7** (amine), 21132-48-9; **8**, 108472-61-3; **8** (amine), 21132-46-7; **9**, 108472-63-5; **9** (amine), 6582-06-5; **10**, 108472-65-7; I, 108472-71-5; II, 108472-72-6; III, 108472-73-7; 3,4,5-$(CH_3O)_3C_6H_2CH_2CN$, 13338-63-1; 3,4-$(CH_3O)_2C_6H_3CH_2CN$, 93-17-4; 4-$CH_3OC_6H_4CH_2CN$, 104-47-2; $C_6H_5CH_2CN$, 140-29-4; 4-$OHCC_6H_4O(CH_2)_2N(C_2H_5)_2$, 15182-94-2; 3,4,5-$(CH_3O)_2C_6H_3CHO$, 86-81-7; 3,4-$(CH_3O)_2C_6H_3CHO$, 120-14-9; 4-$NCCH_2C_6H_4O(CH_2)_2N(C_2H_5)_2$, 92373-69-8; 4-$OHCC_6H_4N(CH_3)_2$, 100-10-7; 4-$OHCC_6H_4CH_3$, 104-87-0; $(CH_3)_2NH$, 124-40-3.

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# 2-Aryl-4(3*H*)-quinazolinone-5-carboxylic Acids

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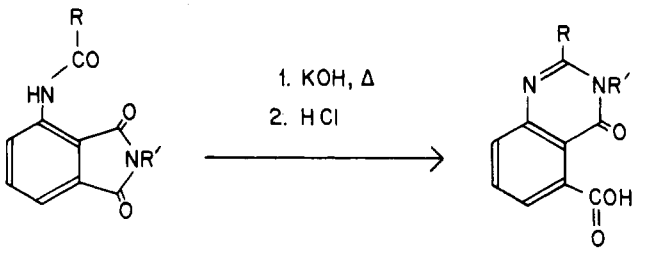
**Twelve 2-aryl-4(3*H*)-quinazolinone-5-carboxylic acids have been prepared by the base-catalyzed rearrangement of 3-(aroylamino)phthalimides. This rearrangement reaction is inhibited by an ortho substituent on the aroyl group.**

Bogert and Jouard (*1*) in 1909 reported that 3-acetamidophthalimide (I, R = $CH_3$, R′ = H) undergoes isomerization to form 2-methyl-4(3*H*)-quinazolinone-5-carboxylic acid (II, R = $CH_3$, R′ = H) when dissolved in hot, aqueous base and then acidified. This reaction was rediscovered in 1961 by Arcoria (*2*, *3*), who showed (*4*, *5*) that it is also applicable to cases in which R is aryl and R′ is methyl or phenyl (Scheme I).

Recently we reported (*6*) the results of our studies of the spectral properties of some 3-benzamidophthalimide (I, R = aryl, R′ = H). The isomerization of a few of these compounds to 2-aryl-4(3*H*)-quinazolinone-5-carboxylic acids (II, R = aryl, R′ = H) had been described by Arcoria, together with the ultraviolet absorption spectra of basic aqueous solutions of the products (*4*, *5*). Acoria's work included no examples of cases in which R possessed an ortho substituent. Since the necessary starting materials were at hand, further studies of this rearrangement reaction were possible.

Table I summarizes the results of these syntheses. The products were white powders with high melting points and low solubilities in all solvents except aqueous bases. The attempted rearrangement of 3-benzamidophthalimdes with ortho substituents on the benzamide moiety yielded the expected product only in the case in which R was 2-methoxyphenyl, and then in only 34% yield. For the cases in which R was 2-nitrophenyl or 2-chlorophenyl, the original 3-benzamidophthalimide was recovered unchanged upon acidification of the reaction mixture.

Scheme I




I II

**Table I. 2-Aryl-4(3*H*)-quinazolinone-5-carboxylic Acids (II, R′ = H)**

| compound II, R = | yield, % | melting point, °C found | melting point, °C reported[a] |
|---|---|---|---|
| phenyl | 72 | 305-307 | 299 |
| 4-methoxyphenyl | 21 | 308-311 | 310 |
| 2-methoxyphenyl | 34 | 255-258 | |
| 4-methylphenyl | 85 | 308-309 | 310 |
| 3-methylphenyl | 52 | 266-267 | 272-273 |
| 4-chlorophenyl | 60 | *b* | >280[c] |
| 3-chlorophenyl | 89 | 329-330 | |
| 4-fluorophenyl | 51 | 340-341 | |
| 3-fluorophenyl | 67 | 315-316 | |
| 3-nitrophenyl | 71 | 335-336 | 334-335 |
| 3,5-dinitrophenyl | 65 | 283-285 | |
| 2-furyl | 70 | 299-300 | |

[a] Reference 5. [b] Sublimes >335 °C. [c] Reference 9.

In the case where R was 2-fluorophenyl an analytically pure product could not be obtained. When R was 2-methylphenyl, the final acidification failed to precipitate a product.

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## Experimental Section

***General.*** Melting points were determined, in °C, with an Electrothermal apparatus. Microanalyses were done by Atlantic Microlab Inc., Atlanta, GA. Infrared absorption spectra were determined with a Perkin-Elmer 225 infrared spectrophotometer, for KBr pellets with weight proportion of sample to KBr of 1 to 300.

***3-(Aroylamino)phthalimides*** (I, R′ = H). The synthesis of the 3-benzamidophthalimides is described elsewhere (*6*). The following two additional 3-aroylaminophthalimides were synthesized by the same method. Both were recrystallized from ethanol.

3-(3,5-Dinitrobenzamido)phthalimide (R = 3,5-dinitrophenyl), 60% yield, mp 271–272 °C. Anal. Calcd for $C_{15}H_8N_4O_7$: C, 50.57; H, 2.26; N, 15.73. Found: C, 50.64; H, 2.31; N, 15.71.

3-(2-Furamido)phthalimide (R = 2-furyl), 65% yield, mp 307–308 °C. Anal. Calcd for $C_{13}H_8N_2O_4$: C, 60.94; H, 3.15; N, 10.93. Found: C, 60.81; H, 3.19; N, 10.94.

***2-Aryl-4(3H)-quinazolinone-5-carboxylic Acids (II).*** To 15 mL of 1 N KOH solution and a stirring bar in a 50-mL beaker was added 0.005 mol of I. The mixture was warmed to 50 °C with stirring and maintained at this temperature for 30 min after complete solution was achieved. The mixture was allowed to cool to room temperature and acidified with 1 N HCl. The resulting white precipitate was removed by suction filtration, washed several times on the filter with cold water to remove inorganic impurities, and air-dried. The crude product was recrystallized from ethanol. The yields reported in Table I are those of the purified products. Elemental analyses were submitted for review.

The infrared absorption spectra of these products were consistent with the results of previous studies (*7*, *8*) of the infrared spectra of quinazolinones. A typical spectrum was that of 2-(3-(nitrophenyl)-4(3*H*)-quinazolinone-5-carboxylic acid (II, R = 3-nitrophenyl, R′ = H): OH–NH–CH stretching region, 3190, 3155, 3118, 3082, 3050, and 2948 $cm^{-1}$; heterocyclic ring frequencies, 1653 $cm^{-1}$ ($\bar{\nu}_{C=O}$), 1588 $cm^{-1}$ ($\bar{\nu}_{C=N}$), and 1554 $cm^{-1}$ ($\bar{\nu}_{CNH}$); carboxyl $\bar{\nu}_{C=O}$, 1708 $cm^{-1}$; CH bending region, 823 and 733 $cm^{-1}$; $\bar{\nu}_{NO_2}$, 1520 and 1340 $cm^{-1}$.

**Registry No.** I(R = $C_6H_5$, R′ = H), 70177-95-6; I(R = 4-$CH_3OC_6H_4$, R′ = H), 70178-09-5; I(R = 2-$CH_3OC_6H_4$, R′ = H), 70178-08-4; I(R = 4-$CH_3C_6H_4$, R′ = H), 70178-07-3; I(R = 3-$CH_3C_6H_4$, R′ = H), 70178-06-2; I(R = 4-$ClC_6H_4$, R′ = H), 70177-98-9; I(R = 3-$ClC_6H_4$, R′ = H), 70177-97-8; I(R = 4-$FC_6H_4$, R′ = H), 70178-01-7; I(R = 3-$FC_6H_4$, R′ = H), 70178-00-6; I(R = 3-$NO_2C_6H_4$, R′ = H), 70178-03-9; I(R = 3,5-$(NO_2)_2C_6H_3$, R′ = H), 108591-65-7; I(R = 2-furyl, R′ = H), 108591-66-8; II(R = $C_6H_5$, R′ = H), 108591-67-9; II(R = 4-$CH_3O_6H_4$, R′ = H), 108591-68-0; II(R = 2-$CH_3OC_6H_4$, R′ = H), 108591-69-1; II(R = 4-$CH_3C_6H_4$, R′ = H), 108591-70-4; II(R = 3-$CH_3C_6H_4$, R′ = H), 108591-71-5; II(R = 4-$ClC_6H_4$, R′ = H), 52171-71-8; II(R = 3-$ClC_6H_4$, R′ = H), 108591-72-6; II(R = 4-$FC_6H_4$, R′ = H), 108591-73-7; II(R = 3-$FC_6H_4$, R′ = H), 108591-74-8; II(R = 3-$NO_2C_6H_4$, R′ = H), 108591-75-9; II(R = 3,5-$(NO_2)_2C_6H_3$, R′ = H), 108591-76-0; II(R = 2-furyl, R′ = H), 108591-77-1.

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# Synthesis of Some 7-Substituted 9,10-Dimethyldibenz[*b*,*e*]indolizine-8,11-diones


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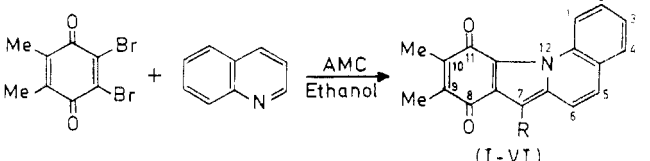
**The synthesis of some 7-substituted 9,10-dimethyldibenz[*b*,*e*]indolizine-8,11-diones using 2,3-dibromo-5,6-dimethyl-1,4-benzoquinone with active methylene compounds and quinoline is described.**


In continuation of a research program on the synthesis of heterocyclic quinones with bridgehead nitrogen atoms (*1*, *2*), which might exhibit biological activity, we report herein the synthesis of some 7-substituted 9,10-dimethyldibenz[*b*,*e*]-indolizine-8,11-diones (Scheme I). The route employed is analogous to that reported by Luckenbaugh (*3*) for synthesizing 12-substituted naphth[2,3-*b*]indolizine-6,11-diones.

**Scheme I**




(I-VI)

I R=H
II R=$CH_3$
III R=$COCH_3$
IV R=$CO_2C_2H_5$
V R=COPh
VI R=CN

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