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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Received for review November 18, 1986. Accepted March 30, 1987.

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

Dhoaib Al-Sammerrai*

Petroleum Research Centre, Jadiriyah, Baghdad, Iraq

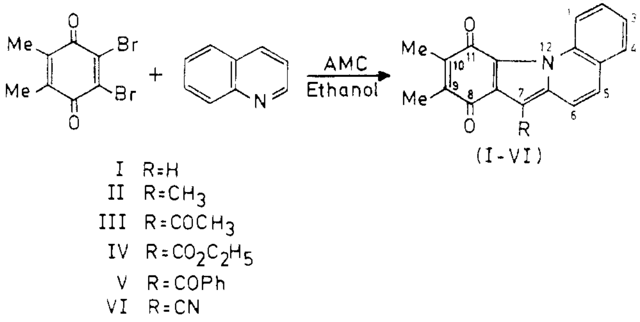
Zuhair S. Salih

Chemistry Department, College of Science, Baghdad University, Baghdad, Iraq

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

0021-9568/87/1732-0390$01.50/0

J. Chem. Eng. Data **1987**, *32*, 391–393

Table I. Characterization Data of 7-Substituted 9,10-Dimethyldibenz[*b*,*e*]indolizine-8,11-diones

compd[a,b]	AMC used	yield, %	mp, °C	^{1}H NMR δ[c]
I	CH_3NO_2	11	>300 decomp	2.2 (d, 6 H), 7.75 (m, 7 H)
II	$C_2H_5NO_2$	19	290 decomp	2.0 (d, 6 H), 2.85 (s, 3 H), 7.5 (m, 5 H), 8.9 (d, 1 H)
III	$CH_3COCH_2COCH_3$	56	286	2.1 (d, 6 H), 2.9 (s, 3 H), 7.5 (m, 4 H), 8.4 (m, 1 H), 9.0 (d, 1 H)
IV	$CH_3COCH_2CO_2C_2H_5$	68	>300 decomp	1.65 (t, 3 H), 2.2 (s, 6 H), 4.8 (q, 2 H), 7.7 (m, 4 H), 8.45 (m, 1 H), 9.3 (d, 1 H)
V	CH_3COCH_2COPh	39	>280 decomp	2.25 (d, 6 H), 7.75 (m, 10 H), 9.35 (d, 1 H)
VI	$NCCH_2CO_2C_2H_5$	40	>320 decomp	2.3 (s, 6 H), 7.8 (m, 5 H), 9.35 (d, 1 H)

[a] Compounds I to V were obtained as deep-red crystals and compound VI as an orange powder. [b] The IR spectra of I to VI exhibited ν(C=O) at 1630–1700 cm^{-1} besides the characteristic peaks. Compound VI also exhibited ν(C≡N) at 2215 cm^{-1}. [c] The ^{1}H NMR spectra were all recorded in CF_3COOH.

Experimental Section

Melting points were determined by using a Gallenkamp heated block apparatus.

IR spectra were recorded on a Perkin-Elmer 337 spectrometer using KBr disks.

PMR were recorded on a Jeol C-60 HL high-resolution NMR spectrometer using Me_4Si as internal standard.

Microanalyses were carried out by the Analytical Department at the Iraqi National Oil Co. Elemental analyses (C, H, N) were obtained and submitted for review and were within ∓0.4% of the theoretical value (Table I).

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

General Method. A mixture of 2,3-dibromo-5,6-dimethylbenzo-1,4-quinone (0.0017 mol) which was prepared following the method of Smith and Austin (*4*), excess quinoline (6 cm^3), and the active methylene compound (AMC) (3.5 cm^3) was heated under reflux in ethanol (25 cm^3) for 3 h. The reaction mixture was cooled and the dark precipitate filtered off, washed well with ethanol, dried, and then crystallized from acetone.

Registry No. I, 108594-48-5; II, 108562-24-9; III, 108562-25-0; IV, 108562-26-1; V, 108562-27-2; VI, 108562-28-3; $MeNO_2$, 75-52-5; $EtNO_2$, 79-24-3; $CH_3C(O)CH_2C(O)CH_3$, 123-54-6; $CH_3C(O)CH_2C(O)OEt$, 141-97-9; $CH_3C(O)CH_2C(O)Ph$, 93-91-4; $NCCH_2C(O)OEt$, 105-56-6; 2,3-dibromo-5,6-dimethyl-2,5-cyclohexadiene-1,4-dione, 38969-08-3; quinoline, 91-22-5.

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Received for review October 7, 1986. Revised manuscript received March 16, 1987. Accepted April 24, 1987.

Synthesis and Spectroscopic Properties of Some New Substituted Saccharin Anils

Salman R. Salman,* Amjad K. Subber, F. A. Hussein,[†] and S. K. Shubber[‡]

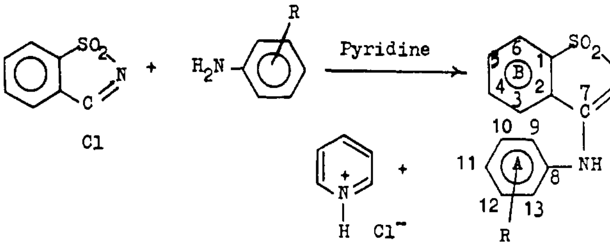
Chemistry Department, College of Science, University of Baghdad, Jadiriyah, Baghdad, Iraq

Fourteen new substituted saccharin anils have been synthesized by the reaction of pseudosaccharin chloride with various substituted anilines. The IR, proton NMR, and carbon-13 NMR spectral properties are presented and discussed.

Introduction

A large number of saccharin derivatives were synthesized (*1–5*) and were reported to show antibacterial activity (*6–12*). On the other hand only saccharin aniline (*13*) was reported in the literature. In this work 14 new saccharin derivative anils are prepared (Table I). The compounds were prepared by the reaction of chlorosaccharin with various substituted aromatic amines. The structures and physical properties of these compounds are given in Tables I and II. The IR, ^{1}H NMR, and carbon-13 NMR spectral data are presented in Tables II and III.

Scheme I



[†] Chemistry Department, College of Pharmacy, University of Baghdad, Baghdad, Iraq.

[‡] Chemistry department, College of Education, University of Baghdad, Baghdad, Iraq.

0021-9568/87/1732-0391$01.50/0