Synthesis and characterization of chromogenic fluoran compounds containing 4-ketoquinazolinone moieties

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Abstract: Chromogenic fluoran compounds containing 4-ketoquinazolinone were synthesized by reacting 2-(4-diethylamino-2-hydroxybenzoyl)benzoic acid with various substituted 4-ketoquinazolinones in the presence of sulfuric acid. The 4-ketoquinazolinones were obtained by reacting various substituted benzoxazin-4-ones with 4-aminophenol or 2-nitro-p-anisidine. All the synthesized derivatives were identified by conventional methods, such as melting points, elemental analysis, IR, 1H-NMR, and UV-visible spectroscopy in organic solvent and 95 % acetic acid. All the fluoran compounds develop colour on contact with acidic or electron-accepting compounds.

Keywords: synthesis, fluoran compounds, 4-ketoquinazolinone, colour change.

INTRODUCTION

Fluoran, spiro[isobenzofuran-1,9'xanthen]-3-one, has been used in a variety of fields, such as thermoindicator, textile finishing, sublimation transfer printing, printing
circuit writing materials etc., though recording papers, i.e., carbonless copying paper and thermo sensitive recording paper, are extraordinarily large in volume.\textsuperscript{1}

Various fluoran compounds containing heterocyclic moieties, such as quinolines,\textsuperscript{2} indole,\textsuperscript{3} benzothiazole,\textsuperscript{4} triazine,\textsuperscript{5} pyridone,\textsuperscript{6} pyrimidine\textsuperscript{7} and pyrrole\textsuperscript{8} are reported as colourless or nearly colourless chromogenic compounds. These compounds produce colour when in contact with an acidic, colour-activating substance, such as organic acid, acid clay, activated clay, phenol-formaldehyde resin, metal salts of aromatic carboxylic acids and bisphenol-A, due to the opening of the lactone ring and conversion to their quinone form.

In the present investigation, chromogenic compounds having the structural formula shown in Fig. 1 have been prepared.

**EXPERIMENTAL**

All melting points (m.p.) are uncorrected and expressed in °C. IR spectra of all the compounds were recorded using a Nicolet Impact-400D FT-IR spectrophotometer as KBr pellets. The \textsuperscript{1}H-NMR spectra were recorded using a Hitachi R-1500 instrument, with TMS as the internal standard. Chemical shifts are given in δ (ppm). Absorption spectra of the compounds in toluene and 95 % acetic acid were recorded using a Shimadzu UV-240 instrument.

**Synthesis of various substituted benzoxazine-4-ones I**

The various substituted benzoxazine-4-ones \textbf{I}, were synthesized according to the method reported in the literature.\textsuperscript{9–14} The benzoxazine-4-ones used were 2-methyl-4\textsubscript{H}-3,1-benzoxazin-4-one (\textbf{I\textsubscript{a}}) (R\textsubscript{1} = CH\textsubscript{3}, R\textsubscript{2} = H, R\textsubscript{3} = H),\textsuperscript{9} 2-phenyl-4\textsubscript{H}-3,1-benzoxazin-4-one (\textbf{I\textsubscript{b}}) (R\textsubscript{1} = Ph, R\textsubscript{2} = R\textsubscript{3} = H),\textsuperscript{10} 2-(chloromethyl)-4\textsubscript{H}-3,1-benzoxazin-4-one (\textbf{I\textsubscript{c}}) (R\textsubscript{1} = CH\textsubscript{2}Cl, R\textsubscript{2} = R\textsubscript{3} = H),\textsuperscript{11} 2-benzyl-4\textsubscript{H}-3,1-benzoxazin-4-one (\textbf{I\textsubscript{d}}) (R\textsubscript{1} = CH\textsubscript{2}Ph, R\textsubscript{2} = R\textsubscript{3} = H),\textsuperscript{12} 2-methyl-6-nitro-4\textsubscript{H}-3,1-benzoxazin-4-one (\textbf{I\textsubscript{e}}) (R\textsubscript{1} = CH\textsubscript{3}, R\textsubscript{2} = NO\textsubscript{2}, R\textsubscript{3} = H),\textsuperscript{13} and 7-chloro-2-methyl-6-nitro-4\textsubscript{H}-3,1-benzoxazin-4-one (\textbf{I\textsubscript{f}}) (R\textsubscript{1} = CH\textsubscript{3}, R\textsubscript{2} = NO\textsubscript{2}, R\textsubscript{3} = Cl).\textsuperscript{14}

**General procedure for quinazolinone A**

A mixture of substituted benzoxazine-4-ones \textbf{I\textsubscript{a}–f} (0.01 mol) and p-aminophenol or 2-nitro-p-anisidine (0.01 mol) was maintained at the temperature given in Table I for 3 h. The reaction mixture was then cooled to 30 °C and 20 ml absolute alcohol added. The solid precipitates were collected by filtration and recrystallized from absolute alcohol. Melting points and yields are presented in Table I.

**TABLE I. Physical data for compounds A**

<table>
<thead>
<tr>
<th>Comp. No.</th>
<th>Reaction temperature/°C</th>
<th>Yield/%</th>
<th>M.p./°C</th>
<th>Molecular weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{A}_1 )</td>
<td>H CH\textsubscript{3} H H</td>
<td>170–75</td>
<td>80</td>
<td>295–97</td>
</tr>
<tr>
<td>( \text{A}_2 )</td>
<td>H Ph H H</td>
<td>170–75</td>
<td>81</td>
<td>229–33</td>
</tr>
<tr>
<td>( \text{A}_3 )</td>
<td>H CH\textsubscript{2}Cl H H</td>
<td>170–75</td>
<td>80</td>
<td>170–73</td>
</tr>
<tr>
<td>( \text{A}_4 )</td>
<td>H CH\textsubscript{2}Ph H H</td>
<td>170–75</td>
<td>79</td>
<td>164–68</td>
</tr>
<tr>
<td>( \text{A}_5 )</td>
<td>H CH\textsubscript{3} NO\textsubscript{2} Cl</td>
<td>170–75</td>
<td>81</td>
<td>260–62</td>
</tr>
<tr>
<td>( \text{A}_6 )</td>
<td>NO\textsubscript{2} CH\textsubscript{3} H H</td>
<td>120–30</td>
<td>70</td>
<td>125–38</td>
</tr>
</tbody>
</table>
IR (KBr) and $^1$H-NMR (DMSO-$d_6$) data for compounds A

3-(4-Hydroxyphenyl)-2-methyl-4(3H)-quinazolinone (A1). IR: 3200–3400 cm$^{-1}$ (OH), 3157 and 1313 cm$^{-1}$ (CH$_3$), 1696 cm$^{-1}$ (C=O), 1596 cm$^{-1}$ (C=N), and 836 cm$^{-1}$ for a p-disubstituted benzene. $^1$H-NMR: 8.79 (1H, s, OH); 6.90–8.37 (13H, m, Ar–H).

2-Chloromethyl-3-(4-hydroxyphenyl)-4(3H)-quinazolinone (A2). IR: 3200–3400 cm$^{-1}$ (OH), 3086 cm$^{-1}$ (CH$_2$Cl), 1693 cm$^{-1}$ (C=O), 1609 cm$^{-1}$ (C=N), 780 cm$^{-1}$ (Cl) and 822 cm$^{-1}$ for a p-disubstituted benzene. $^1$H-NMR: 8.79 (1H, s, OH); 6.90–8.37 (8H, m, Ar–H), 4.23 (2H, s, Ar–CH$_2$Cl).

2-Benzyl-3-(4-hydroxyphenyl)-4(3H)-quinazolinone (A3). IR: 3200–3400 cm$^{-1}$ (OH), 3089 cm$^{-1}$ (CH$_2$), 1696 cm$^{-1}$ (C=O), 1595 cm$^{-1}$ (C=N) and 820 cm$^{-1}$ for a p-disubstituted benzene. 1H-NMR: 8.82 (1H, s, OH); 6.90–8.36 (13H, m, Ar–H); 4.08 (2H, s, Ar–CH$_2$Ph).

7-Chloro-3-(4-hydroxyphenyl)-2-methyl-6-nitro-4(3H)-quinazolinone (A4). IR: 3200–3400 cm$^{-1}$ (OH), 3167 and 1308 cm$^{-1}$ (–CH$_3$), 1694 cm$^{-1}$ (C=O), 1608 cm$^{-1}$ (C=N), 1520 cm$^{-1}$ and 1354 cm$^{-1}$ (NO$_2$), 812 cm$^{-1}$ (Cl) and 818 cm$^{-1}$ for a p-disubstituted benzene. 1H-NMR: 8.99 (1H, s, OH); 6.90–8.76 (6H, m, Ar–H), 2.25 (3H, s, Ar–CH$_3$).

3-(4-Methoxy-2-nitrophenyl)-2-methyl-4(3H)-quinazolinone (A5). IR: 3032 cm$^{-1}$ and 1320 cm$^{-1}$ (Ar–CH$_3$), 1693 cm$^{-1}$ (C=O), 1351 cm$^{-1}$ (NO$_2$) and 860–900 cm$^{-1}$, for a 1,2,4-trisubstituted benzene. 1H-NMR: 7.10–8.35 (7H, m, Ar–H), 3.85 (3H, s, Ar–OMe), 2.27 (3H, s, Ar–CH$_3$).

3-(4-Methoxy-2-nitrophenyl)-2-phenyl-4(3H)-quinazolinone (A6). IR: 1696 cm$^{-1}$ (C=O), 1602 cm$^{-1}$ (C=N), 1535 and 1350 cm$^{-1}$ (NO$_2$) and 860–900 cm$^{-1}$ for a 1,2,4-trisubstituted benzene. 1H-NMR: 7.10–8.35 (7H, m, Ar–H), 3.83 (3H, s, Ar–OMe), 2.27 (3H, s, Ar–CH$_3$).

2-(4-Diethylamino-2-hydroxybenzoyl)benzoic acid B

2-(4-Diethylamino-2-hydroxybenzoyl)benzoic acid was synthesized by refluxing equimolar amounts of 3-diethylaminophenol and phthalic anhydride in toluene, as reported in the literature.$^1$
m.p. 205–207 °C. IR (KBr): 3294 cm⁻¹ (OH), 2992, 2891, 2856, 1488 cm⁻¹ (N–Et), 1702 cm⁻¹ (CO; Ar–COOH), 1669 cm⁻¹ (CO), 1608, 1427, 1320, 1300, 1055, 1011, 944, 809, 802, 708, 541 cm⁻¹.

1H-NMR (DMSO-d₆): 12.59 (2H, s, –COOH, OH), 6.77–7.97 (7H, m, Ar–H), 3.16–3.55 (4H, q, N–(CH₂–CH₃)₂), 1.06–1.28 (6H, t, N–(CH₂–CH₃)₂).

General procedure for fluoran compounds C

Compounds A₁–10 (0.01 mol) and B (0.01 mol) were dissolved in 10 ml of concentrated sulfuric acid, at 40 °C and thereafter stirred at room temperature for 48 h. After completion of the reaction, the reaction mixture was poured into a mixture of 50 g ice and 50 ml cold water and the solid substance which precipitated was collected by filtration. The solid substance after disintegration in water and adjustment of the pH to 10 by addition of a 10 % solution of sodium hydroxide was filtered and dried. The dried substance was then recrystallized from n-butanol.

Solutions of these compounds in toluene were almost colourless while in contact with silica gel a colour formed instantaneously. The colour which formed are given in Table II.

Elemental analysis; IR (KBr) and ¹H-NMR (acetone) data for compounds C

6-Diethylamino-2-(2’-methyl-4’-oxo(3’,4’-dihydroquinazolin-3’-yl))fluoran (C₁). Calculated for C₃₃H₂₇N₃O₄: C, 74.84; H, 5.14; N, 7.93 %. Found: C, 73.94; H, 5.21; N, 7.96 %. IR: 3073 and 1313 cm⁻¹ (CH₃), 2965, 2938, 2865, 1467 and 1347 cm⁻¹ (N–Et), 1783 cm⁻¹ (C=O group of lactone ring) and 1696 cm⁻¹ (C=O group of quinazolinone), 1595 cm⁻¹ (C=N), 1600, 1525, 1501, 1286, 1105, 876, 762, 695 and 534 cm⁻¹. ¹H-NMR: 6.46–8.32 (14H, m, Ar–H), 3.20–3.53 (4H, q, N–(CH₂–CH₃)₂), 2.31 (3H, s, Ar–CH₃), 1.06–1.28 (6H, t, N–(CH₂–CH₃)₂).

6-Diethylamino-2-(2’-phenyl-4’-oxo(3’,4-dihydroquinazolin-3’-yl))fluoran (C₂). Calculated for C₃₈H₂₉N₄O₆: C, 77.14; H, 4.94; N, 7.10 %. Found: C, 76.28; H, 4.90; N, 7.01 %. IR: 2932, 2885, 2832, 1481 and 1355 cm⁻¹ (N–Et), 1763 cm⁻¹ (C=O group of lactone ring) and 1692 cm⁻¹ (C=O group of quinazolinone), 1629 cm⁻¹ (C=N), 1602, 1552, 1521, 1111, 1064, 1017, 762, 695 and 534 cm⁻¹. ¹H-NMR: 6.47–8.37 (19H, m, Ar–H), 3.20–3.42 (4H, q, N–(CH₂–CH₃)₂), 1.06–1.28 (6H, t, N–(CH₂–CH₃)₂).

6-Diethylamino-2-(2’-(chloromethyl)-4’-oxo(3’,4-dihydroquinazolin-3’-yl))fluoran (C₃). Calculated for C₃₃H₂₆ClN₃O₄: C, 70.27; H, 4.64; N, 7.45 %. Found: C, 70.79; H, 4.52; N, 7.46 %. IR: 3068 cm⁻¹ (CH₂), 2959, 2918, 2859, 1481 and 1347 cm⁻¹ (N–Et), 1776 cm⁻¹ (C=O group of lactone ring) and 1693 cm⁻¹ (C=O group of quinazolinone), 1629 cm⁻¹ (C=N), 1608 cm⁻¹ (C=N), 789 cm⁻¹ (Cl), 1600, 1279, 1118, 876, 774, 708, 628 and 541 cm⁻¹. ¹H-NMR: 6.41–8.38 (14H, m, Ar–H), 4.22 (2H, s, CH₂Cl), 3.22 to 3.44 (4H, q, N–(CH₂–CH₃)₂), 1.08–1.27 (6H, t, N–(CH₂–CH₃)₂).

6-Diethylamino-2-(2’-benzyl-4’-oxo(3’,4’-dihydroquinazolin-3’-yl))fluoran (C₄). Calculated for C₃₉H₃₁N₃O₄: C, 77.34; H, 5.16; N, 6.93 %. Found: C, 76.05; H, 5.08; N, 6.94 %. IR: 3100 cm⁻¹ (CH₂), 2965, 2943, 2865, 1481, 1367 cm⁻¹ (N–Et), 1770 cm⁻¹ (C=O group of lactone ring) and 1689 cm⁻¹ (C=O group of quinazolinone), 1625 cm⁻¹ (C=N), 1602, 1525, 1501, 1111, 1038, 883, 823, 762, 708 and 460 cm⁻¹. ¹H-NMR: 6.42–8.35 (29H, m, Ar–H), 4.08 (2H, s, –CH₂Ph), 3.22–3.45 (4H, q, N–(CH₂–CH₃)₂), 1.07–1.29 (6H, t, N–(CH₂–CH₃)₂).

6-Diethylamino-2-(7’-chloro-2’-methyl-6’-nitro-4’-oxo(3’,4’-dihydroquinazolin-3’-yl))fluoran (C₅). Calculated for C₃₃H₂₅ClN₄O₆: C, 65.08; H, 4.14; N, 9.20 %. Found: C, 64.95; H, 4.01; N, 9.31 %. IR: 3053, 1323 cm⁻¹, 2979, 2891, 1484, 1347 cm⁻¹ (N–Et), 1756 cm⁻¹ (C=O group of lactone ring) and 1693 cm⁻¹ (C=O group of quinazolinone), 1625 cm⁻¹ (C=N), 1520 and 1533 cm⁻¹ (NO₂), 787 cm⁻¹ (Cl), 1602, 1508, 1405, 1275, 1192, 1152, 1105, 1038, 823, 776, 695 and 581 cm⁻¹. ¹H-NMR: 6.48–8.73 (12H, m, Ar–H), 3.22–3.54 (4H, q, N–(CH₂–CH₃)₂), 2.19 (3H, s, Ar–CH₃), 1.08–1.28 (6H, t, N–(CH₂–CH₃)₂).

6-Diethylamino-2-(2’-methyl-6’-nitro-4’-oxo(3’,4’-dihydroquinazolin-3’-yl))fluoran (C₆). Calculated for C₃₃H₂₆N₄O₆: C, 68.98; H, 4.56; N, 9.75 %. Found: C, 68.68; H, 4.32; N, 9.64 %. IR: 3076 and 1313 cm⁻¹ (Ar–CH₃), 2992, 2892, 1481, 1347 cm⁻¹ (N–Et), 1770 cm⁻¹ (C=O group of lactone ring) and 1696 cm⁻¹ (C=O group of quinazolinone), 1595 cm⁻¹ (C=N), 1521 and 1353 cm⁻¹.
(NO₂). 1H-NMR: 6.46–8.32 (13H, m, Ar–H), 3.20–3.53 (4H, q, N–(CH₂–CH₃)₂), 2.31 (3H, s, Ar–CH₃), 1.06–1.28 (6H, t, N–(CH₂–CH₃)₂).

6-Diethylamino-2-(2’-phenyl-4’-oxo(3’,4’-dihydroquinazolin-3’-yl)-3-nitrofluoran (C⁷). Calculated for C₃₈H₂₈N₄O₆: C, 71.69; H, 4.43; N, 8.80 %. Found: C, 71.21; H, 4.55; N, 8.87 %.

IR: 2979, 2918, 2870, 1467, 1347 cm⁻¹ (N–Et), 1750 cm⁻¹ (C=O group of lactone ring) and 1688 cm⁻¹ (C=O group of quinazolinone), 1622 cm⁻¹ (C=N), 1514 and 1350 cm⁻¹ (NO₂).


6-Diethylamino-2-(2’-(chloromethyl)-4’-oxo(3’,4’-dihydroquinazolin-3’-yl)-3-nitrofluoran (C⁸). Calculated for C₃₃H₂₅ClN₄O₆: C, 65.08; H, 4.14; N, 9.20 %.

IR: 3104 cm⁻¹ (CH₂Cl), 2959, 2918, 2850, 1464, 1347 cm⁻¹ (N–Et), 1789 cm⁻¹ (C=O group of lactone ring) and 1693 cm⁻¹ (C=O group of quinazolinone), 1615 cm⁻¹ (C=N), 1525 and 1355 cm⁻¹ (NO₂).

1H-NMR: 6.41–8.38 (13H, m, Ar–H), 4.22 (2H, s, CH₂Cl), 3.22 to 3.44 (4H, q, N–(CH₂–CH₃)₂), 1.08 to 1.27 (6H, t, N–(CH₂–CH₃)₂).

6-Diethylamino-2-(2’-benzyl-4’-oxo(3’,4’-dihydroquinazolin-3’-yl)-3-nitrofluoran (C⁹). Calculated for C₃₉H₃₀N₄O₆: C, 71.99; H, 4.65; N, 8.61 %.

IR: 3092 cm⁻¹ (CH₂Ph), 2985, 2960, 2887, 1462, 1347 cm⁻¹ (C=O group of lactone ring) and 1683 cm⁻¹ (C=O group of quinazolinone), 1592 cm⁻¹ (C=N), 1514 and 1357 cm⁻¹ (NO₂).

1H-NMR: 6.42–8.35 (28H, m, Ar–H), 4.08 (2H, s, –CH₂Ph), 3.22–3.44 (4H, q, N–(CH₂–CH₃)₂), 1.07–1.29 (6H, t, N–(CH₂–CH₃)₂).

6-Diethylamino-2-(7’-chloro-2’-methyl-6’-nitro-4’-oxo(3’,4’-dihydroquinazolin-3’-yl)-3-nitrofluoran (C₁₀). Calculated for C₃₃H₂₄ClN₅O₈: C, 60.60; H, 3.70; N, 10.71 %.

IR: 3072 and 1317 cm⁻¹ (Ar–CH₃), 2965, 2918, 2850, 1481, 1340 cm⁻¹ (N–Et), 1750 cm⁻¹ (C=O group of lactone ring) and 1693 cm⁻¹ (C=O group of quinazolinone), 1615 cm⁻¹ (C=N), 1521–1340 cm⁻¹ (NO₂), 1323 cm⁻¹ (CH₃), 820 cm⁻¹ (Cl).

1H-NMR: 6.48–8.73 (11H, m, Ar–H), 3.22–3.54 (4H, q, N–(CH₂–CH₃)₂), 2.19 (3H, s, Ar–CH₃), 1.08–1.28 (6H, t, N–(CH₂–CH₃)₂).

RESULTS AND DISCUSSION

Various benzoxazones Iₐ-f when reacted with 4-aminophenol (II; R = H) or 2-nitro-p-anisidine (II; R = NO₂) gave the corresponding ketoquinazolinone A₁-10. The reaction between compounds A₁-10 with 2-(4-diethylamino-2-hydroxybenzoyl)benzoic acid (B) in the presence of sulfuric acid gave the corresponding fluorans C₁-10 (Scheme 1). All the fluoran compounds were characterized by elemental analysis, UV, IR and ¹H-NMR spectroscopy.

The IR spectra of all the fluoran compounds showed the disappearance of the characteristic absorption band of the OH/OMe group and the appearance of the C=O group of the lactone ring at 1745–1790 cm⁻¹ and of the C=O group of 4-keto-quinazolinone at 1680–1700 cm⁻¹ as well as other characteristic absorption bands for the rest of the molecules.

<table>
<thead>
<tr>
<th>Comp. No.</th>
<th>R</th>
<th>R₁</th>
<th>R₂</th>
<th>R₃</th>
<th>Yield %</th>
<th>M.p./%</th>
<th>λ_max in toluene/nm</th>
<th>λ_max in 95 % acetic acid/nm</th>
<th>Colour on silica gel</th>
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</table>

TABLE II. Physical data for compound C
The absorption maxima in toluene and 95 % acetic acid of all these fluoran compounds are shown in Table II. The appearance of one peak in the spectra of the fluoran compounds in toluene is due to the lactone form while the three peaks in 95 % acetic acid are due to the quinone, zwitterions and the lactone form.15,16
The solutions of these compounds in toluene were colourless but in contact with silica gel or acetic acid a colour formed instantaneously as shown in Table II.

CONCLUSION

These chromogenic fluoran compounds are soluble in organic solvents forming colourless solutions but show a spontaneous colour-forming property in aqueous acid solutions or acidic colour-activating substances such as silica gel. Nitro substitution at the 3-position of the fluoran compound gave a more bathochromic shift compared to the unsubstituted analogs.

IZVOD

SINTEZA I KARAKTERIZACIJA HROMOGENIH JEDIWEWA FLUORANA KOJA SADRŽE 4-KETO-HINAZOLINONE

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Hromogena jediweva fluorana koje sadrže 4-keto-hinazolinon sintetizovana su reagovaljem 2-(4-dietilamino-2-hidroksibenzoil)benzoše kiseline sa različitim supstituisanim 4-keto-hinazolinonima u prisustvu sumporne kiseline. 4-Keto-hinazolinoni su dobiveni reagovaljem različitih supstituisanih benzoksazin-4-ona sa 4-aminofenolon ili 2-nitro-p-anisidinom. Svi sintetisani derivati su identifikovani konvencionalnim metodama, kao što su tачke topqewa, elementalna analiza, IR, 1H-NMR i UV – vidqiva spektroskopija u organskom rastvaraču i 95 % sirćetnoj kiseline. Sva fluoranska jediweva pokazuju obojewe u kontaktu sa kiselim ili elektron-akceptorskim jediwevima.

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REFERENCES