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SYNTHESIS AND OPTICAL PROPERTIES RESEARCH OF SOME NOVEL TRICHROMOPHORE COMPOUNDS CONTAINING COUMARIN, PYRAZOLINE AND NAPHTHALIMIDE GROUPS

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Abstract – A series of novel trichromophore compounds containing coumarin, pyrazoline and naphthalimide groups were synthesized and their properties were elucidated using absorption and fluorescence. In chloroform solution, their maximal absorption bands and the maximal emission bands slightly varied from 478 to 485 nm and from 559 to 565 nm, respectively.

INTRODUCTION

As an important class of organic heterocyclic dyes, coumarin derivatives exhibit unique photochemical and photophysical properties, which render them useful in a variety of applications such as optical brighteners, laser dyes, non-linear optical chromophores, solar energy collectors, fluorescent labels and probes in biology and medicine.[1-4] More importantly, coumarin dyes have also been used as blue, green and red dopants in organic light-emitting diodes (OLEDs).[5-7]

Similarly, 1, 3, 5-triaryl-2-pyrazolines, with their rigid but only partly unsaturated central pyrazoline ring, are well-known fluorescent compounds widely used for the optical brightening of textile fiber, plastics and paper.[8] Pyrazoline derivatives have not only excellent hole-transfer performance but also excellent emitting blueness property.[9, 10] The fluorescence properties of these pyrazoline derivatives were reported by studying the effect of substituents on the absorption and fluorescence properties of this class of compounds.[11,12]

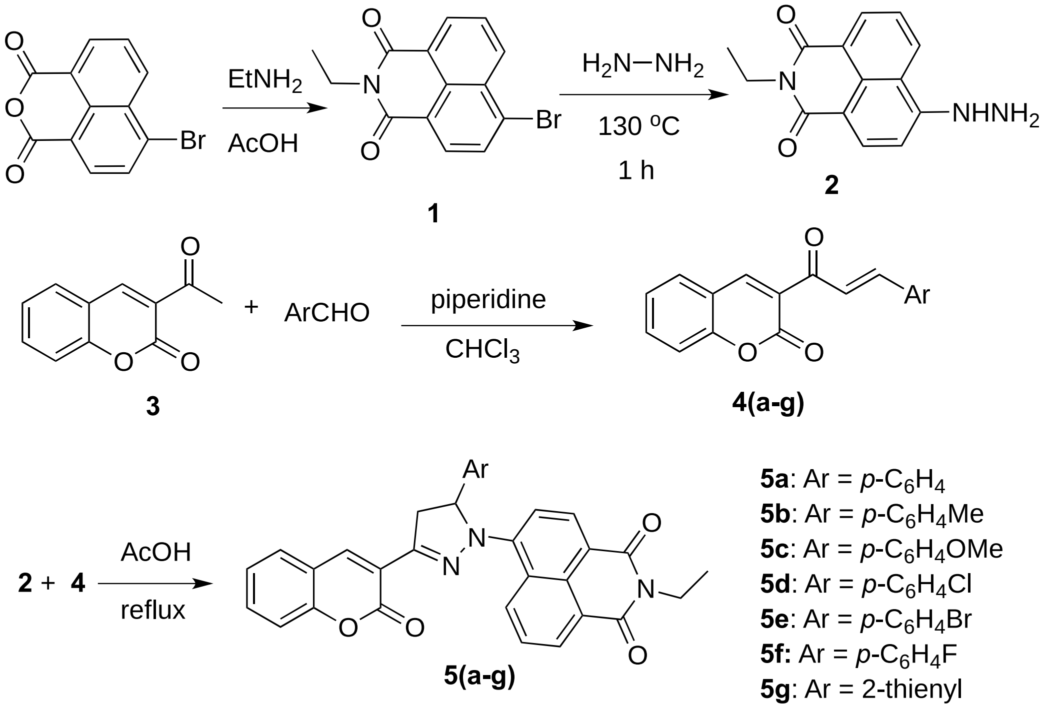
On the other hand, naphthalimide usually exhibits strong fluorescent emission on irradiation, which usually acts as supermolecular moieties for the study of photo-induced electron transfer,[13,14] fluorescence switcher,[15,16] liquid crystal displays and EL materials.[17,18]

In view of the considerable importance of these compounds work, we envisaged that compounds containing these moieties in the same molecule may show enhanced optical properties. In this paper, we synthesized a series of novel trichromophore compounds containing coumarin, pyrazoline and naphthalimide groups and researched their optical properties.

RESULTS AND DISCUSSION

The synthetic route of trichromophore compounds was shown in Scheme 1.

Compound **2** was prepared from 4-bromo-1,8-naphthalic anhydride through two-step reaction in 84% yield. Compound **4(a-g)** were synthesized from 3-acetylcoumarins and different aromatic aldehyde in the presence of piperidine in chloroform under reflux in 85-90% yields. The trichromophore compounds **5(a–g)** were obtained by the reaction of compound **2** with substituted chalcones **4(a–g)** in AcOH solution under reflux temperature in 78–85% yields. The structures of the compounds **5(a–g)** were determined by IR, NMR, EI-MS and Elemental analyses.



Scheme 1. The synthesis of trichromophore compounds **5(a-g)**

The UV-vis spectrums of naphthalimide **2** and coumarin chalcones **4(a-g)** in chloroform solution with the concentration of 1×10^{-5}M were shown in Figure 1. Their absorption wavelength maxima and molar extinction coefficient (ε) values were presented in Table 1. As shown in Table 1, compounds **2** showed

UV absorption maxima at 412nm and the absorption peaks of **4(a-g)** varied from 344 to 372 nm, which indicated that given various electron-withdrawing and electron-donating substituent on the aryl ring had an obvious effect on the electronic structure of these π-conjugation molecules. Compared with **2** and **4(a-g),** the absorption spectrum of trichromophore compounds **5(a-g)** showed strong bathochromic shift effect. As shown in Figure 2 and Table 2, compounds **5(a-g)** showed UV absorption maxima at 478–485nm, indicating that naphthalimide group and pyrazoline ring extended their π-conjugation system.

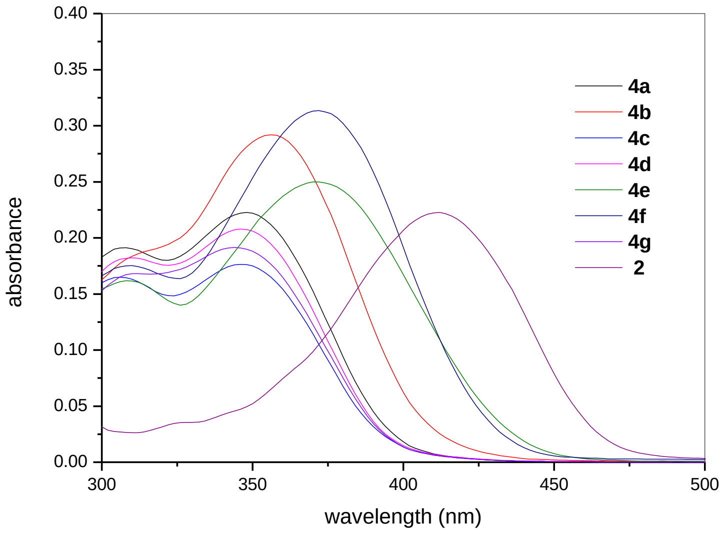


Figure 1. UV-vis spectra of compounds **2** and **4(a-g)** in $CHCl_3$ with the concentration of 1×10^{-5} M

Table 1. The absorption spectra data of compound **2** and **4(a-g)** in $CHCl_3$

Compound	λ_{max}^{abs}(nm)	$\varepsilon(10^4M^{-1}cm^{-1})$	Compound	λ_{max}^{abs}(nm)	$\varepsilon(10^4M^{-1}cm^{-1})$
2	412	2.22	**4d**	346	2.07
4a	348	2.31	**4e**	370	2.49
4b	356	2.92	**4f**	372	3.13
4c	350	1.75	**4g**	344	1.91

From the Figure 2 and Table 2, it can be seen that the absorption spectra of compounds **5(a–g)** were very similar because of their same structure unit of pyrazoline, which confirmed that given substituent Ar of C5 of pyrazoline ring had an negligible effect on the electronic structure of the molecules, because aromatic (Ar) in C5 cannot extend to the π-conjugation system. The molar coefficient of **5d** was slightly larger than other trichromophore compounds, it maybe because the substituent Cl atom on the benzene ring had better auxochrome effect than other atoms.

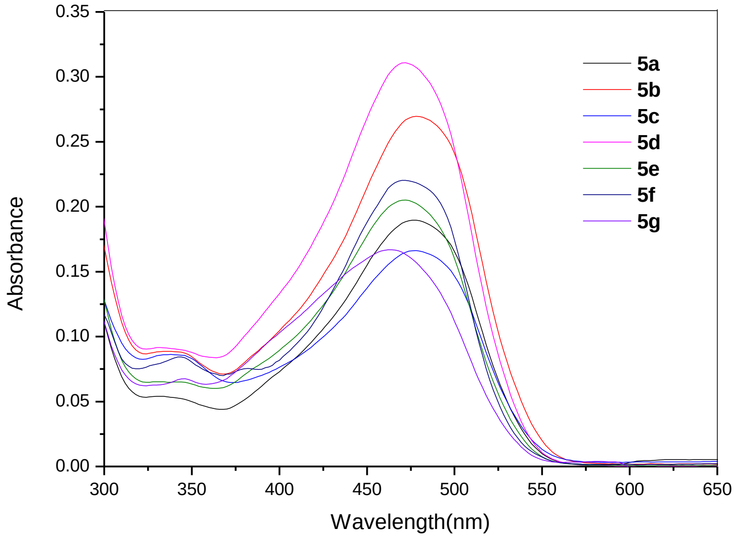


Figure 2. UV-vis spectra of compounds **5(a-g)** in $CHCl_3$ with the concentration of 1×10^{-5} M

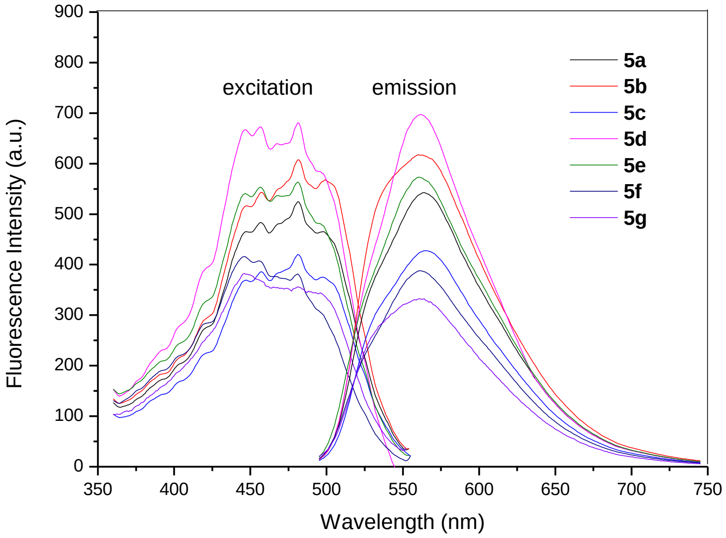


Figure 3. The fluorescence excitation and emission spectra of compounds **5(a-g)** in $CHCl_3$ with the concentration of 1×10^{-5} M

Figure 3 showed the fluorescence spectra of compounds **5(a-g)** in chloroform solution with concentration of 1×10^{-5} M. As shown in Table 2, these compounds had good red fluorenscence and high quantum yields, the quantum yields of **5(a-g)** varied from 0.615 to 0.928. The maximal excitation bands and the maximal emission bands of compound **5(a-g)** varied from 480 to 484 nm and from 559 to 565 nm, respectively, indicating that substituent phenyl group in 5-posision of pyrazoline ring had little influence on the excitation and emission wavelengths, because aromatic (Ar) in C5 cannot extend to the π-conjugation system. However, we found that the substituent Ar affected the fluorescence quantum yields of compounds **5(a-g)**. As listed in Table 2, when Ar was *p*-(Me) phenyl group, the fluorescence quantum yield was higher than that when Ar was *p*-(F) phenyl group.

Table 2. The optical characteristics of compound **5(a-g)** in $CHCl_3$.

Compound	λ_{max}^{abs}(nm)	$\varepsilon(10^4 M^{-1} cm^{-1})$	λ_{max}^{ex}(nm)	λ_{max}^{em}(nm)	$\Delta v(cm^{-1})$	Φ_F
5a	479	1.90	480	561	3008	0.747
5b	479	2.70	482	559	2858	0.928
5c	482	1.66	481	565	3091	0.741
5d	480	3.10	484	560	2804	0.686
5e	478	2.05	480	565	3134	0.630
5f	483	2.19	481	563	3028	0.615
5g	485	1.67	483	562	2910	0.740

In conclusion, a series of novel trichromophore compounds containing coumarin, pyrazoline and naphthalimide groups were designed and synthesized, and their absorption and fluorescence spectral characteristics were investigated in chloroform solution. The results showed their maximal absorption bands and the maximal emission bands varied from 478 to 485 nm and from 559 to 565 nm, respectively. These compounds maybe serve as good red light-emitting materials.

EXPERIMENTAL

Melting point was determined with XT4A micromelting point apparatus and was uncorrected. The ^1H NMR was recorded on a Bruker 300 spectrometer with TMS as internal reference and $CDCl_3$ as solvent. IR were recorded on a Perkin–Elmer PE–983 infrared spectrometer as KBr pellets with absorption in cm^{-1}. MS were obtained with Finnigan Trace MS instrument using EI method. UV spectra were measured on a SP-1900 spectrophotometer. Fluorescence spectra were determined on a Hitachi F-4500. The fluorescent quantum yield of these compounds was measured by comparing with rhodamine B as the standard compound in ethanol solution (λ_{ex} 495 nm; A (Absorption), <0.01; $\Phi_F = 0.89$)[19]

Starting materials. 2-Ethyl-6-hydrazinobenzo[*de*]isoquinoline-1,3-dione **2**,[20] 3-acetyl-2*H*-chromen-2-one (**3**),[21] coumarin-chalcone hybrids **4(a-f)**[22] and **4g**[23] were prepared according to literature. All other chemicals used in this study were commercially available.

General procedure for the synthesis of trichromophore compounds 5: A mixture of **2** (1 mmol) and chalcone **4** (1 mmol) in AcOH (15 mL) was refluxed for 8 h. The progress of the reaction was monitored by TLC. Then the reactant was poured into water. The precipitate was separated by filtration, washed with water, dried and consequently recrystallized from THF: EtOH (1:1, v/v) to afford **5** as red solid.

1-(2-Ethylbenzo[*de*]isoquinoline-1,3-dione-6-yl)-3-(-2-oxo-2*H*-1-benzopyran-3-yl)-5-(phenyl)-2-pyrazoline (5a) : mp 272–273 °C; IR (ν_{max}, KBr, cm^{-1}): 1733, 1681, 1639, 1574, 1415, 1382, 1097, 764; ^{1}H NMR (CDCl$_3$), δ (ppm): 9.59-9.62 (m, 1H, Ar-H), 8.48 (dd, 2H, *J* = 6.9, 6.9 Hz, Ar-H), 7.72-7.78 (m, 1H, Ar-H), 7.60 (dd, 2H, *J* = 2.7, 2.7 Hz, Ar-H), 7.31 (m, 8H, Ar–H), 5.72 (dd, 1H, *J* = 5.1, 5.1 Hz, pyrazoline-H), 4.17-4.25 (m, 2H), 4.12 (dd, 1H, *J* = 12.0, 12.0 Hz, pyrazoline-H), 3.50 (dd, 1H, *J* = 8.1, 8.1 Hz, pyrazoline-H), 1.28-1.37 (t, *J* = 3H, 6.9 Hz, CH$_3$-H); ^{13}C NMR (CDCl$_3$), δ (ppm): 13.35, 35.26, 44.12, 66.63, 111.49, 115.01, 116.63, 119.02, 119.96, 122.63, 123.69, 124.94, 125.18, 125.83, 128.27, 129.34, 130.50, 131.31, 132.13, 132.49, 133.78, 139.46, 139.71, 145.11, 148.08, 153.83, 159.11, 163.67, 164.45. EI mass spectrometry: *m/z* 513.00. M^+ calculated 513.17. Anal. Calcd for $C_{32}H_{23}N_3O_4$: C 74.84, H 4.51, N 8.18. Found: C 74.80, H 4.49, N 8.16.

1-(2-Ethylbenzo[*de*]isoquinoline-1,3-dione-6-yl)-3-(-2-oxo-2*H*-1-benzopyran-3-yl)-5-(p-tolyl)-2-pyrazoline (5b) : mp 270–271 °C; IR (ν_{max}, KBr, cm^{-1}): 1735, 1682, 1641, 1575, 1382, 1098, 752, 472; ^{1}H NMR (CDCl$_3$), δ (ppm): 9.60 (dd, 1H, *J* = 1.17 Hz, Ar-H), 8.64 (dd, 1H, *J* = 1.14 Ar-H), 8.30-8.32 (m, 1H, Ar-H), 7.71-7.77 (m, 1H, Ar-H), 7.55-7.61 (m, 2H, Ar-H), 7.30-7.38 (m, 2H, Ar–H), 7.09-7.19 (m, 4H, Ar–H), 6.85-6.88 (m, 1H, Ar–H), 5.69 (dd, 1H, *J* = 8.1, 8.1 Hz, pyrazoline-H), 4.21 (dd, 2H, *J* = 7.2, 7.2 Hz, CH$_2$-H), 4.09 (dd, 1H, *J* = 11.4, 11.4 Hz, pyrazoline-H), 3.48 (dd, 1H, *J* = 8.4, 8.4 Hz, pyrazoline-H), 2.29 (s, 3H), 1.30 (t, 3H, *J* = 6.9 Hz, CH$_3$-H); ^{13}C NMR (CDCl$_3$), δ (ppm): 13.32, 21.04, 35.21, 44.08, 66.43, 111.48, 114.84, 116.59, 119.00, 119.98, 122.58, 123.62, 124.87, 125.09, 128.49, 129.94, 130.46, 131.24, 132.11, 132.41, 133.82, 136.69, 138.04, 139.33, 145.08, 148.03, 153.77, 159.02, 163.63, 164.42. EI mass spectrometry: *m/z* 527.01. M^+ Calculated 527.18. Anal. Calcd for $C_{33}H_{25}N_3O_4$: C 75.13, H 4.78, N 7.96. Found: C 75.09, H 4.76, N 7.80.

1-(2-Ethylbenzo[*de*]isoquinoline-1,3-dione-6-yl)-3-(-2-oxo-2*H*-1-benzopyran-3-yl)-5-(*p*-methoxy-phenyl)-2-pyrazoline (5c) : mp 230–231 °C; IR (ν_{max}, KBr, cm^{-1}): 1731, 1679, 1640, 1574, 1383, 1244, 1098, 766, 477; ^{1}H NMR (CDCl$_3$), δ (ppm): 9.56-9.58 (m, 1H, Ar-H), 8.62-8.65 (m, 1H, Ar-H), 8.31-8.34 (m, 2H, Ar-H), 7.72-7.77 (m, 1H, Ar-H), 7.55-7.61 (m, 2H, Ar-H), 7.22-7.39 (m, 6H, Ar–H), 6.80-6.89 (m,2H, Ar–H), 5.68 (dd, 1H, *J* = 8.1, 8.1Hz, pyrazoline-H), 4.21 (dd, 2H, *J* = 7.2, 7.2Hz, -CH$_2$), 4.08 (dd, 1H, J = 10.4, 10.4Hz , pyrazoline-H). 3.75 (s, 3H, OCH_3), 3.48 (dd, 1H, J = 8.1, 8.1Hz, pyrazoline-H), 1.29 (t, 3H, J = 6.9 Hz, $-CH_3$); ^{13}C NMR ($CDCl_3$), δ (ppm): 13.35, 35.26, 44.14 , 55.26, 66.25, 111.48, 114.84, 116.59, 119.00, 120.03, 122.60, 123.76, 124.92, 125.15, 127.10, 128.54, 130.46, 131.29, 131.64, 132.12, 132.45, 133.74, 139.39, 145.20, 148.10, 153.81, 159.12, 159.39,163.69, 164.47. EI mass spectrometry: *m/z* 542.91. M^+ calculated 543.18. Anal. Calcd for $C_{33}H_{25}N_3O_5$: C 72.92, H 4.64, N 7.73. Found: C 72.89, H 4.62, N 7.70.

1-(2-Ethylbenzo[*de*]isoquinoline-1,3-dione-6-yl)-3-(-2-oxo-2*H*-1-benzopyran-3-yl)-5-(4-chlorophenyl)pyrazoline (5d) : mp 279–280 °C; IR (ν_{max}, KBr, cm^{-1}): 1733, 1681, 1641, 1574, 1384, 1299, 1097, 748,476; ^1H NMR ($CDCl_3$), δ (ppm): 9.51 (dd, 1H, J = 1.2, 1.2 Hz, Ar-H), 8.65 (dd, 1H, J = 12, 12 Hz, Ar-H), 8.32-8.35 (m, 2H, Ar-H), 7.73-7.78 (m, 1H, Ar-H), 7.56-7.62 (m, 2H, Ar-H), 7.27- 7.39 (m, 5H, Ar–H), 6.83 (d, J = 8.4, 8.4 Hz, 1H, Ar–H), 5.70 (dd, 1H, J = 8.4, 8.4 Hz, pyrazoline-H), 4.21 (dd, 2H, J = 7.2, 7.2 Hz, $-CH_2$), 4.11 (dd, 1H, J = 8.7, 8.7Hz, pyrazoline-H), 3.49 (dd, 1H, J = 8.4, 8.4 Hz, pyrazoline-H), 1.31 (t, 3H, J = 6.9Hz, CH_3-H); ^{13}C NMR ($CDCl_3$), δ (ppm): 13.35, 35.29, 44.11, 53.41, 66.03, 111.57, 115.46, 116.66, 118.98, 119.79, 122.72, 123.80, 124.98, 125.32, 127.30 128.60, 129.03, 129.30, 129.55, 130.45, 131.36, 132.01, 132.59, 133.49, 134.12, 138.21, 139.62, 144.96, 148.08, 153.87, 159.14, 163.58, 164.37. EI mass spectrometry: *m/z* 547.02. M^+ Calculated 547.13. Anal. Calcd for $C_{32}H_{22}ClN_3O_4$: C 70.14, H 4.05, N 7.67. Found: C 70.11, H 4.03, N 7.65.

1-(2-Ethylbenzo[*de*]isoquinoline-1,3-dione-6-yl)-3-(-2-oxo-2*H*-1-benzopyran-3-yl)-5-(4-bromophenyl)pyrazoline (5e) : mp 292–293 °C; IR (ν_{max}, KBr, cm^{-1}): 1733, 1678, 1640, 1573, 1298, 1099, 748, 474; ^1H NMR ($CDCl_3$), δ (ppm): 9.51-9.54 (m, 1H, Ar-H), 8.65 (dd, 1H, J = 1.2, 1.2 Hz, Ar-H), 8.56-8.67 (m, 1H, Ar-H), 7.73-7.78 (m, 1H, Ar-H), 7.56-7.62 (m, 2H, Ar-H), 7.01-7.45 (m, 8H, Ar–H), 6.81-6.84 (m, 1H, Ar–H), 5.69 (dd, 1H, J = 8.7, 8.7Hz, pyrazoline- H), 4.26 (dd, 2H, J = 6.9, 6.9Hz, $-CH_2$), 4.16 (dd, 2H, J = 6.9, 6.9Hz, pyrazoline-H), 3.48 (dd, 1H, J = 8.4, 8.4 Hz, pyrazoline-H), 1.31 (t, 3H, J = 6.9 Hz, CH_3-H); ^{13}C NMR ($CDCl_3$), δ (ppm): 13.35, 35.30, 44.06, 66.07, 111.55, 115.45, 116.66, 118.97, 119.76, 122.18, 122.71, 123.79, 124.99, 125.34, 127.62, 128.61, 129.52, 130.45, 131.38, 131.97, 132.03, 132.50, 132.61, 133.49,138.72, 139.65, 144.93, 148.08, 153.86, 159.16, 163.59, 164.38. EI mass spectrometry: *m/z* 591.06, M^+ calculated 591.08. Anal. Calcd for $C_{32}H_{22}BrN_3O_4$: C 64.87, H 3.74, N 7.09. Found: C 64.83, H 3.70, N 7.05.

1-(2-Ethylbenzo[*de*]isoquinoline-1,3-dione-6-yl)-3-(-2-oxo-2*H*-1-benzopyran-3-yl)-5-(4-fluorophenyl)pyrazoline (5f) : mp 268–269 °C; IR (ν_{max}, KBr, cm^{-1}): 1732, 1678, 1641, 1575, 1519, 1386, 1298, 1097, 751, 474; ^1H NMR ($CDCl_3$), δ (ppm): 9.51 (dd, 1H, J = 1.2, 1.2Hz, Ar-H), 8.65 (dd, 1H, J = 1.2, 1.2Hz, Ar-H), 8.32-8.34 (m, 2H, Ar-H), 7.75 (m, 1H, dd, 1H, J = 7.2, 7.2Hz, Ar-H), 7.56-7.61 (m, 2H, Ar-H), 7.22-7.39 (m, 3H, Ar–H), 6.97-7.02 (m, 2H, Ar–H), 6.83-6.86 (m, 1H, Ar–H), 5.71 (dd, 1H, J = 8.1, 8.1Hz, pyrazoline-H), 4.21 (dd, 2H, J =7.2, 7.2Hz, $-CH_2$), 4.11 (dd, 1H, J = 10.4, 10.4Hz, pyrazoline-H), 3.49 (dd, 1H, *J* = 8.4, 8.4Hz, pyrazoline-H), 1.29 (t, 3H, *J* = 6.9Hz, $-CH_3$); ^{13}C NMR ($CDCl_3$), δ (ppm): 13.31, 35.26, 44.16, 66.00, 111.62, 115.37, 116.15, 116.44, 116.62, 118.97, 119.83, 122.68, 123.82, 124.94, 125.26, 127.55, 127.66, 128.56, 130.42, 131.32, 131.99, 132.53, 133.48, 135.42, 135.46, 139.54, 145.02, 148.06, 153.84, 159.13, 160.73, 163.59, 160.73, 163.59, 164.01, 164.35. EI mass spectrometry: *m/z* 531.01. M^+ Calculated 531.16. Anal. Calcd for $C_{32}H_{22}FN_3O_4$: C 72.31, H 4.17, N 7.91. Found: C 72.29, H 4.15, N 7.87.

1-(2-Ethyl-benzo[*de*]isoquinoline-1,3-dione-6-yl)-3-(-2-oxo-2*H*-1-benzopyran-3-yl)-5-(2-thiophene)-2-yrazoline (5g) : mp 270–271 °C; IR (ν_{max}, KBr, cm^{-1}): 1734, 1683, 1640, 1574, 1421, 1380, 1301, 1247, 1097, 910, 766; ^{1}H NMR ($CDCl_3$), δ (ppm): 9.26-9.29 (m, 1H, Ar-H), 8.64 (dd, 1H, *J* = 0.9, 0.9 Hz, Ar-H), 8.37-8.41 (m, 2H, Ar-H), 7.72-7.78 (m, 1H, Ar-H), 7.55-7.62 (m, 2H, Ar-H), 7.33- 7.39 (m, 2H, Ar–H), 7.12-7.15 (m, 2H, Ar-H), 6.97-6.98 (m, 1H, Ar–H), 6.84-6.87 (m, 1H, Ar–H), 5.98-5.99 (m, 1H, pyrazoline-H), 4.21 (dd, 2H, *J*=6.9, 6.9Hz, $-CH_2$), 4.13 (dd, 2H, *J* = 8.1, 8.1Hz, pyrazoline-H), 3.69 (dd, 1H, *J* = 8.1, 8.1 Hz, pyrazoline-H), 1.31 (t, 3H, *J* = 6.9Hz, CH_3-H); ^{13}C NMR ($CDCl_3$), δ (ppm): 13.35, 35.31, 44.50, 62.92, 113.02, 116.22, 116.64, 119.03, 119.88, 122.72, 124.48, 124.96, 125.38, 125.45, 125.69,128.64, 130.16, 131.30, 131.95, 132.54, 132.92, 139.69, 142.47, 145.68, 148.49, 153.91, 159.29, 163.69, 164.37. EI mass spectrometry: *m/z* 519.00. M^+ calculated 519.13. Anal. Calcd for $C_{30}H_{21}N_3O_4S$. C 69.35, H 4.07, N 8.09. Found: C 69.31, H 4.04, N 8.05.

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