Myriam Kammoun,[a] Hamida Turki,[a] Rachid El Gharbi,[a]* and Suzanne Fery-Forgues[b]

[a]Laboratoire de Chimie Appliquée: Hétérocycles, Corps Gras et Polymères. Faculté des Sciences de Sfax, 3038 Sfax, Tunisia
[b]Laboratoire des Interactions Moléculaires Réactivité Chimique et Photochimique, UMR CNRS 5623, Université Paul Sabatier, 31062 Toulouse Cedex, France
*E-mail: gharbi.rachid@yahoo.fr

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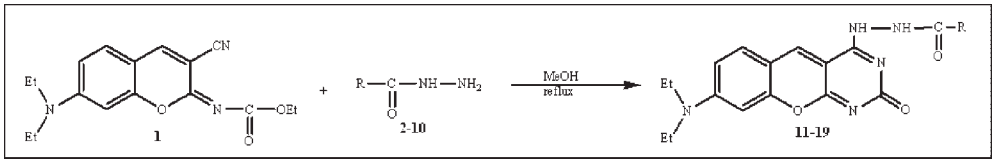

Condensation of 3-cyano-7-diethylamino-*N*-ethoxycarbonyl-iminocoumarin with various hydrazides as N-nucleophiles gave a new series of 2-oxo-2*H*-1-benzopyrano[2,3-*d*]pyrimidines bearing an acylhydrazino fragment in the 4-position. The structures of the products obtained were characterized using IR, ^{1}H NMR, ^{13}C NMR and elemental analysis. The optical properties were reported for three of these compounds.

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INTRODUCTION

Benzopyranopyrimidines have been increasingly investigated because of their important biological properties. They exhibit for example anticancer [1,2], analgesic [3], and antithrombotic [4] activities. In addition, benzopyranopyrimidines could also be of interest because their structure is close to that of rhodols, rhodamines, and fluoresceins [5], which constitute a well-known class of dyes. Different routes have been described in the literature for the synthesis of this class of condensed heterocycles [6–8]. As part of our investigations on the synthesis of benzopyranopyrimidines, we have reported previously [9], a new method based on the transformation of 3-cyano-iminocoumarins under the action of primary aliphatic and aromatic amines, on heating in methanol. We have shown that the reaction proceeded by an original mechanism that involves several consecutive steps including nucleophilic attack of the iminolactone ring, pyran ring opening, E/Z isomerization, and subsequent cyclization. As a continuation of our studies on the reactivity of 3-cyano-iminocoumarins with N-nucleophiles, we wish to report in this article on the condensation of 3-cyano-7-diethylamino-*N*-ethoxycarbonyl-iminocoumarin **1** with different hydrazides. The aim is to obtain a new series of benzopyranopyrimidines incorporating an acylhydrazino fragment in the 4-position, since the presence of this function could improve physiological activities. The optical properties of some benzopyranopyrimidines were also regarded.

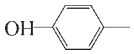
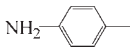
RESULTS AND DISCUSSION

Iminocoumarin **1** was obtained by Knoevenagel condensation followed by *N*-ethoxycarbonylation as previously described in [9]. The condensation of this compound with various hydrazides (**2–11**, see Table 1) was studied, first focusing on a specific system, namely **1** and benzoic hydrazide **2** (Scheme 1), to determine the best conditions for synthesis. The progress of the reaction was followed by TLC analysis and by the changes in FTIR spectra of samples taken at regular intervals from the reaction medium. Thus, the FTIR spectra showed the progressive disappearance of the original peaks at 2214 and 1724 cm^{-1} arising from the C=N and COOEt functions, respectively, as well as the appearance of characteristic peaks of the pyrimidine moiety at 1632 cm^{-1} (C=N), 1664 cm^{-1} (NH—NH—C=O) and 1644 cm^{-1} (C=0). The best result was obtained by refluxing the mixture of **1** and **2** in methanol during 2 h. 4-(2-Benzoylhydrazino)-8-diethylamino-2-oxo-2*H*-1-benzopyrano[*2,3-d*]pyrimidine (**11**) was thus obtained with a yield of 92% (Scheme 1). This product was isolated by simple filtration with high purity as confirmed by ^{1}H NMR, which showed the absence of resonance at 1.37 and 4.32 ppm arising from the original ethoxy fragment, and the presence of two peaks in the region of 10–11 ppm indicating the presence of the NH—NH—CO function.

When the reaction was carried out in acetic acid at room temperature the condensation of **1** with **2** did not

Table 1

Benzopyranopyrimidines obtained by condensation of iminocoumarin (**1**) with various hydrazides.

Hydrazide (RC(O)NH NH_2)	R	Benzopyranopyrimidine	Time (h)	Yield (%)
2		11	2	92
3		12	3	63
4		13	7	52
5		14	1	87
6		15	2	75
7		16	8	48
8		17	16	70
9	CH_3	18	1	45
10		19	3	85

Reaction time and yield according to the nature of substituent R.

take place and no benzopyranopyrimidine **11** was detected. It was found that **1** reacted intramolecularly in these conditions to produce the oxidized derivative **11a** (Scheme 1). This type of compound was reported by Blythin *et al*. [10] when carrying out the Knoevenagel reaction with salicylaldehydes and *N*-cyanoacety-lurethane.

On sight of these results, compound **1** was then condensed with hydrazides **3–10** at reflux in methanol and the progress of the reaction was monitored by TLC. The reagent specific structure induced changes in reactivity and the reaction time had to be modified (Table 1).

In each case, the reaction afforded the corresponding 4-acylhydrazinobenzopyranopyrimidine **12–19** as shown in Scheme 2 that summarizes the synthesis. The FTIR and NMR data relative to these compounds were in good agreement with their structure. It is interesting to note that the presence of both amine and hydrazide functions in hydrazide **6** could lead to the formation of two isomers (**15** and **15a**) resulting from the interaction of the imidic carbon with the two N-nucleophilic centers in the first step of the mechanism (Scheme 3). However, chromatographic and spectroscopic analyses showed that the iminocoumarin was selectively converted into **15** whereas **15a** was not detected, probably because the amine function is less nucleophilic than the hydrazide function. The structure of **15** was clearly confirmed by the ^{1}H NMR spectrum, which showed the presence of Ar—NH_2 (s, 2H, 5.71 ppm), NH—NH—CO (s, 1H, 10.16 ppm and s, 1H, 10.60 ppm) and the absence of peak around 4–5 ppm related to the NH_2 group of the hydrazidic fragment.

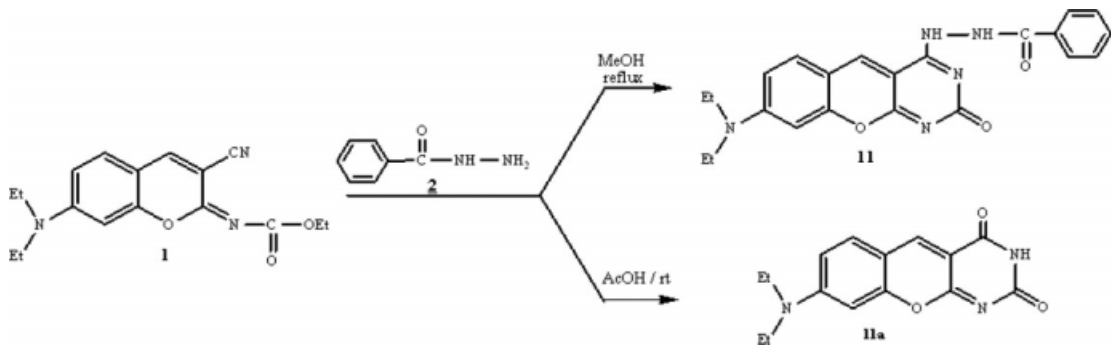
OPTICAL PROPERTIES

The electron conjugated system of benzopyranopyrimidines **11–19** is identical and should be poorly affected by the nature of the R substituent. It can therefore be expected that all these compounds share common spectroscopic behaviour. The optical properties were measured on three of them (namely compounds **12**, **13**, and **15**). The results are gathered in Table 2.

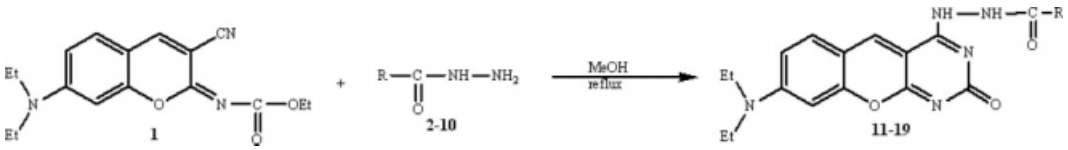
Compounds **12**, **13**, and **15** were poorly soluble in organic solvents. All were studied in dimethylsulfoxide, but only two of them could be dissolved in dichloromethane. The solutions were filtered on paper filter prior to spectroscopic study. However, for most of the samples, the absorption spectrum was particularly wide, with a markedly high baseline. This indicates that in spite of filtration, some particles were present in solution and scattered light.

For each sample, the excitation spectrum was much narrower than the absorption spectrum. For compound **13**, both spectra had almost the same maximum. For **15**

Scheme 1



Scheme 2. General scheme of the synthesis. Substitutent R as described in Table 1.



and **12**, the excitation spectra showed a considerable blue shift reaching 50 nm compared with the absorption spectra. Besides, the excitation spectra did not vary with the emission wavelength, and conversely, the emission spectra were independent of the excitation wavelength. This indicates the presence of only one fluorophore in each solution. The discrepancy between the absorption and excitation spectra could thus be attributed to the presence of nonfluorescent impurities. But, the TLC analyses of the three compounds gave a single spot, so the presence of impurities could be discarded. Consequently, it is most likely that these poorly soluble compounds form nonfluorescent aggregates in solution, these aggregates being particularly visible at long wavelengths on the absorption spectra.

The emission spectra did not show fine resolution. Their maximum was rather close for the three compounds, around 520 nm in DMSO and slightly above 500 nm in dichloromethane. A small solvent effect can thus be noted. The lifetimes were found to be monoexponential, above the nanosecond. The quantum yields were not calculated since the absorption spectra corresponded to two species at least, only one of which is fluorescent. Therefore, the quantum yields measured would have been lower than their actual value.

If comparing the properties of these benzopyranopyrimidines with those of parent compound **1** in the same solvents [11,12], it appears, as could be expected, that cyclization has led to a moderate shift of the absorption and emission wavelengths towards longer wavelengths. In contrast, the fluorescence lifetime is quite similar for the two types of compounds.

CONCLUSION

This article reported the facile synthesis of new 2-oxo-2*H*-1-benzopyrano[*2,3-d*]pyrimidines from the condensation of a 3-cyanoiminocoumarin with various hydrazides. The products were easily purified and obtained with a good yield. Their limited solubility in most of organic solvents could be a drawback for subsequent applications. However, it is possible that these compounds display interesting biological properties, which would deserve further investigations.

EXPERIMENTAL

7-Diethylamino-2-hydroxybenzaldehyde, malonitrile and hydrazides were commercially available from Aldrich. All melting points were determined on an Electrothermal 9100 apparatus. Infrared spectra were registered on a Jasco FT-IR 420 spectrophotometer apparatus using KBr pellets. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker WP 200 spectrometer at 300 MHz in DMSO-d_6 with TMS as internal standard (chemical shifts in ppm).

Scheme 3

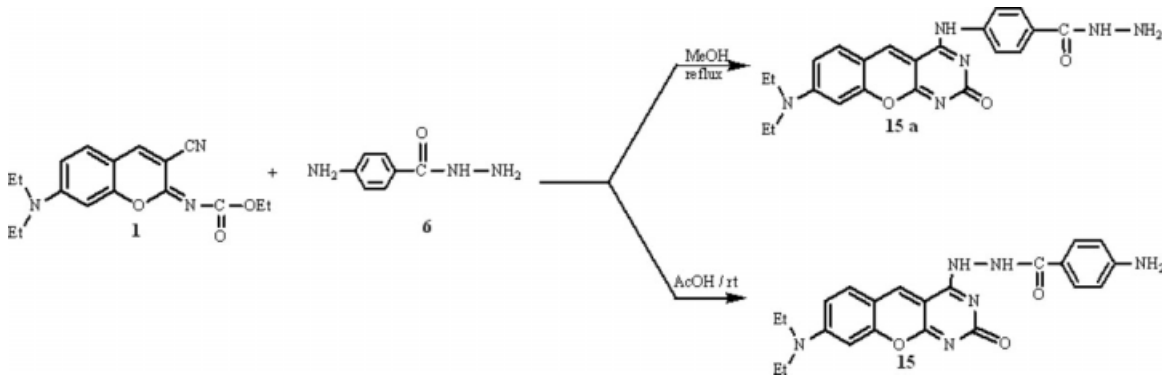


Table 2

Maximum absorption wavelength (λ_{abs}), maximum excitation wavelength (λ_{ex}), maximum emission wavelength (λ_{em}), and fluorescence lifetime (τ) for compounds 12, 13, and 15 in dimethylsulfoxide and dichloromethane.

Compound	λ_{abs} (nm)	λ_{ex} (nm)	λ_{em} (nm)	τ (ns)
12 (DMSO)	504	458	520	2.6 ± 0.3
13 (DMSO)	486	476	518	2.9 ± 0.2
13 (CH_2Cl_2)	484 (508 sh)	480	502	3.2 ± 0.2
15 (DMSO)	508	480	520	1.5 ± 0.2
15 (CH_2Cl_2)	502	451	507	1.9 ± 0.2

UV/vis absorption spectra were recorded on a Hewlett-Packard 8452A diode array spectrophotometer. Steady state fluorescence work was performed on a Photon Technology International (PTI) Quanta Master 1 spectrofluorometer. All excitation and emission spectra were corrected. Fluorescence decay was measured with the stroboscopic technique using a Strobe Master fluorescence lifetime spectrophotometer from PTI. The excitation source was a flash lamp filled with a mixture of nitrogen and helium (30/70). Data were collected over 200 channels with a time-base of 0.1 ns per channel. Analysis of fluorescence decay was performed using the multiexponential method software from PTI. All spectrophotometric measurements were conducted in a thermostated cell at 25°C.

3-Cyano-*N*-ethoxycarbonyl-7-diethylamino iminocoumarin 1. 3-Cyano-*N*-ethoxycarbonyl-7-diethylamino iminocoumarin **1** was prepared as previously described in [9].

General procedure for the synthesis of benzopyranopyrimidines 11–19. A solution of 3-cyano-*N*-ethoxycarbonyl-7-diethylamino iminocoumarin 1 (1.56 g, 5 mmol) and (5 mmol) of hydrazide in 30 mL of absolute methanol was refluxed during the time indicated in Table 1. After complete reaction, the benzopyranopyrimidine obtained was separated by filtration and washed with ethanol. For spectroscopic use, compound 12, 13, and 15 were purified on TLC plates using chloroform as the eluent.

4-(2-Benzoylhydrazino)-8-diethylamino-2-oxo-2H-benzopyrano[2,3-d]pyrimidine (11). Mp: 244°C. IR (cm^{-1}): 1632 (C═N), 1644 (C═O), 1664 (N─CO─N), 3331 (NH). ^{1}H NMR: δ = 1.14 (t, *J* = 6.9 Hz, 6H, CH_3), 3.51 (q, *J* = 6.9 Hz, 4H, CH_2N), 6.74 (s, 1H, H_9), 6.88 (d, *J* = 9.0, 1H, H_7), 7.45–7.55 (3 H, COR), 7.75 (d, *J* = 9.0 Hz, 1H, H_6), 7.87–7.89 (2 H, COR), 8.47 (s, 1H, H_5), 10.49 (s, 1H, NH), 10.64 (s, 1H, NH). *Anal*. Calcd. for $C_{22}H_{21}N_5O_3$: C, 65.50; H, 5.25; N, 17.36. Found: C, 65.46; H, 5.33; N, 17.35.

4-[2-(3-Fluorobenzoyl)hydrazino]-8-diethylamino-2-oxo-2H-benzopyrano[2,3-d]pyrimidine (12). Mp: 249°C. IR (cm^{-1}): 1634 (C═N, C═O), 1664 (N─CO─N), 3331 and 3335 (NH). ^{1}H NMR: δ = 1.14 (t, *J* = 6.9 Hz, 6H, CH_3), 3.51 (q, *J* = 6.9 Hz, 4H, CH_2N), 6.75 (s, 1H, H_9), 6.89 (dd, *J* = 7.2, 1.8 Hz, 1H, H_7), 7.39 (m, 1H, COR), 7.53 (m, 1H, COR), 7.76 (m, 2H, COR), 7.74 (d, *J* = 7.2, 1H, H_6), 8.49 (s, 1H, H_5), 10.50 (s,1H, NH), 10.60 (s, 1H, NH). ^{13}C NMR: δ = 12.71 (CH_3), 44.90 (CH_2─NH), 96.73 (C_9), 104.92, 110.00 (C_{12}, C_{13}), 111.80 (C_7), 114.74, 118.26, 124.35, 130.63 (CH─R), 132.02 (C_6), 136.76 (R), 138.04 (C_5), 140.76 (C_8), 153.31 (C_4), 156.14 (C_{10}), 160.52 (C_{11}), 162.13 (C─F), 163.75 (C═O), 167.95 (NH─C═O). *Anal*. Calcd. for $C_{22}H_{20}FN_5O_3$: C, 62.70; H, 4.78; N, 16.62. Found: C, 63.10; H, 4.80; N, 16.50.

4-[2-(2-Thienylcarbonyl)hydrazino]-8-diethylamino-2-oxo-2H-benzopyrano[2,3-d]pyrimidine (13). Mp: 247°C. IR (cm^{-1}): 1619 (C═N), 1639 (C═O), 1660 (N─CO─N), 3289 (NH). ^{1}H NMR: δ = 1.14 (t, *J* = 7.0 Hz, 6H, CH_3), 3.51 (q, *J* = 7.0 Hz, 4H, CH_2N), 6.76 (d, *J* = 2.1 Hz, 1H, H_9), 6.61 (s, 1H, COR), 6.89 (dd, *J* = 9.0, 2.1 Hz, 1H, H_7), 7.18 (s, 1H, COR), 7.71 (d, *J* = 9.0 Hz, 1H, H_6), 7.80 (s, 1H, COR) , 8.46 (s, 1H, H_5), 10.46 (s,1H, NH), 10.69 (s, 1H, NH). *Anal*. Calcd. for $C_{20}H_{19}N_5O_3S$: C, 58.67; H, 4.68; N, 17.10. Found: C, 59.01; H, 4.64; N, 17.11.

4-[2-(4-Hydroxybenzoyl)hydrazino]-8-diethylamino-2-oxo-2H-benzopyrano[2,3-d]pyrimidine (14). Mp: 270°C. IR (cm^{-1}): 1608 (C═N), 1636 (C═O), 1664 (N─CO─N), 3326 (NH), 3423 (OH). ^{1}H NMR: δ = 1.13 (t, *J* = 6.9 Hz, 6H, CH_3), 3.50 (q, *J* = 6.9 Hz, 4H, CH_2N), 6.73 (s, 1H, H_9), 6.82 (d, *J* = 8.7 Hz, 2H, COR), 7.77 (d, *J* = 8.7 Hz, 2H, COR), 6.85 (d, *J* = 9.3 Hz, 1H, H_7), 7.72 (d, *J* = 9.3 Hz, 1H, H_6), 8.42 (s, 1H, H_5), 10.28 (s,1H, OH), 10.46 (s,1H, NH), 10.69 (s, 1H, NH). *Anal*. Calcd. for $C_{22}H_{21}N_5O_4$: C, 63.00; H, 5.05; N, 16.70. Found: C, 62.90; H, 5.23; N, 17.71.

4-[2-(4-Aminobenzoyl)hydrazino]-8-diethylamino-2-oxo-2H-benzopyrano[2,3-d]pyrimidine (15). Mp: 245°C. IR (cm^{-1}): 1605 (C═N), 1637 (C═O), 1663 (N─CO─N), 3210 and 3334 (NH), 3439 (NH_2). ^{1}H NMR: δ = 1.12 (t, *J* = 6.9 Hz, 6H, CH_3), 3.47 (q, *J* = 6.9 Hz, 4H, CH_2N), 5.71 (s, 2H, NH_2), 6.57 (d, *J* = 8.4 Hz, 2H, COR), 6.65 (d, *J* = 8.4 Hz, 2H, COR), 6.69 (s, 1H, H_9), 6.82 (d, *J* = 9.3 Hz, 1H, H_7), 7.67 (d, *J* = 9.3 Hz, 1H, H_6), 8.36 (s, 1H, H_5), 10.16 (s,1H, NH), 10.60 (s, 1H, NH). ^{13}C NMR: δ = 12.69 (CH_3), 44.84 (CH_2─NH), 96.68 (C_9), 105.67, 109.90 (C_{12}, C_{13}), 111.58 (C_7), 112.77 (CH─R), 120.49 (C─R) 129.78 (C_6), 131.67 (CH─R),136.96 (C_5), 137.38 (C─R), 152.32 (C_8), 152.93 (C_4), 155.81 (C_{10}), 156.30 (C_{11}) 163.57 (C═O), 168.09 (NH─C═O). *Anal*. Calcd. for $C_{22}H_{22}N_6O_3$: C, 63.15; H, 5.30; N, 20.08. Found: C, 62.90; H, 5.33; N, 20.00.

4-[2-(2-Furoyl)hydrazino]-8-diethylamino-2-oxo-2H-benzopyrano[2,3-d]pyrimidine (16). Mp: 246°C. IR (cm^{-1}): 1607 (C═N), 1636 (C═O), 1663 (N─CO─N), 3290 (NH). ^{1}H NMR: δ = 1.13 (t, *J* = 7.0 Hz, 6H, CH_3), 3.51 (q, *J* = 7.0 Hz, 4H, CH_2N), 6.66 (m, 1H, COR), 6.76 (d, *J* = 2.1 Hz, 1H, H_9), 6.89 (dd, *J* = 9.0, 2.1 Hz, 1H, H_7), 7.24 (d, 1H, COR), 7.76 (d, *J* = 9.0 Hz, 1H, H_6), 7.89 (m, 1H, COR), 8.46 (s, 1H, H_5), 10.15 (s,1H, NH), 10.55 (s, 1H, NH). *Anal*. Calcd. for $C_{20}H_{19}N_5O_4$: C, 61.06; H, 4.87; N, 17.80. Found: C, 61.20; H, 4.93; N, 17.84.

4-(2-Isonicotinoylhydrazino)-8-diethylamino-2-oxo-2H-benzopyrano[2,3-d]pyrimidine (17). Mp: >270°C. IR (cm^{-1}): 1607 (C═N), 1640 (C═O), 1661 (N─CO─N),3234 (NH). ^{1}H NMR: δ = 1.15 (t, *J* = 6.9 Hz, 6H, CH_3), 3.52 (q, *J* = 6.9

Hz, 4H, CH_2N), 6.66 (m, 1H, COR), 6.68 (d, J = 2.1 Hz, 1H, H_9), 6.89 (dd, J = 7.0, 1.5 Hz, 1H, H_7), 7.73 (d, 1H, COR), 7.77 (d, J = 7.0 Hz, 1H, H_6), 7.82 (dd, 2H, COR), 8.53 (s, 1H, H_5), 10.67 (s, 2H, 2NH). *Anal*. Calcd. for $C_{21}H_{20}N_6O_3$: C, 62.37; H, 4.98; N, 20.78. Found: C, 61.97; H, 4.80; N, 20.44.

4-(2-Acetylhydrazino)-8-diethylamino-2-oxo-2H-benzopyrano[2,3-d]pyrimidine (18). Mp: >270°C. IR (cm^{-1}): 1609 (C═N), 1636 (C═O), 1662 (N─CO─N),3232 (NH).^{1}H NMR: δ = 1.14 (t, J = 6.9 Hz, 6H, CH_3), 2.17 (s, 3H, CH_3), 3.42 (q, J = 6.9 Hz, 4H, CH_2N), 6.63 (d, J = 2.1 Hz, 1H, H_9), 6.78 (d, J = 7.0, 2.1 Hz, 1H, H_7), 7.50 (d, J = 7.0 Hz, 1H, H_6), 8.12 (s, 1H, H_5), 10.73 (s, 1H, NH), 10.76 (s, 1H, NH). *Anal*. Calcd. for $C_{17}H_{19}N_5O_3$: C, 59.81; H, 5.61; N, 20.52. Found: C, 60.07; H, 5.63; N, 20.80.

4-[2-(2-Hydroxybenzoyl)hydrazino]-8-diethylamino-2-oxo-2H-benzopyrano[2,3-d]pyrimidine (19). Mp: >270°C. IR (cm^{-1}): 1606 (C═N), 1640 (C═O), 1661 (N─CO─N),3234 (NH), 3433 (OH).^{1}H NMR: δ = 1.13 (t, J = 6.9 Hz, 6H, CH_3), 3.51 (q, J = 6.9 Hz, 4H, CH_2N), 6.77 (s, J = 1.5 Hz, 1H, H_9), 6.90 (dd, J = 7.0, 1.5 Hz, 1H, H_7), 7.39 (dd, 2H, COR), 7.75 (d, J = 7.0 Hz, 1H, H_6), 7.90 (d, 2H, COR), 8.53 (s, 1H, H_5), 10.60 (s, 1H, 1NH), 10.75 (s, 1H, OH), 10.90 (s, 1H, NH). *Anal*. Calcd. for $C_{22}H_{21}N_5O_4$: C, 63.00; H, 5.05; N, 16.70. Found: C, 62.61; H, 5.42, N, 16.57.

Synthesis of 8-diethylamino 2*H*-benzopyrano[2,3-*d*]pyrimidine-2,4(3*H*)-dione (11a). A mixture of (1.56 g, 5 mmol) of 1 and (0.68 g, 5 mmol) of benzoic hydrazide 2 were stirred at 20°C in 20 mL of glacial acetic acid for 20 h. The product was isolated by filtration and recrystallized in dimethylsulfoxide. Mp 250°C. Yield: 55%. IR (cm^{-1}): 1641 (C═O), 3246 (NH). ^{1}H NMR (300 MHz, DMSO-d_6): δ = 1.14 (t, J = 6.9 Hz, 6H, CH_3), 3.53 (q, J = 6.9Hz, 4H, CH_2N), 6.80 (d, J = 1.5 Hz, 1H, H_9), 6.89 (dd, J = 6.9, 1.5 Hz, 1H, H_7), 7.74 (d, J = 6.9 Hz, 1H, H_6), 8.59 (s, 1H, H_5), 11.06 (s,1H, NH). ^{13}C NMR: δ = 12.86 (CH_3), 45.29 (CH_2─NH), 96.97 (C_9), 104.98, 110.31 (C_{12}, C_{13}), 112.49 (C_7), 133.41 (C_6), 144.38 (C_5), 154.89 (C_8), 157.34 (C_4), 157.57 (C_{10}), 162.52 (C═O), 168.57 (C═O). *Anal*. Calcd. for $C_{15}H_{15}N_3O_3$: C, 63.15; H, 5.30; N, 14.73. Found: C, 62.96; H, 5.21; N, 14.69.

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