Photophysical properties of 2,3-dihydroquinazolin-4(1H)-one derivatives

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ABSTRACT

2,3-Dihydroquinazolin-4(1H)-one (DHQ) derivatives were synthesized by treatment of isatoic anhydride with amines and subsequent cyclocondensation with aldehydes or ketones. The derivatives were characterized by ^{1}H and ^{13}C NMR, elemental analysis and HRMS. Absorption and emission spectra of DHQ derivatives were recorded in different solvents (hexadecane, benzene, chloroform, methanol and acetonitrile). Both the absorption and the emission maxima are solvent-dependent and red-shifted. Molar extinction coefficients were determined to be 2364–4820 M^{-1} cm^{-1}. The Stokes shifts of the compounds are large and increase with solvent polarity. This feature and the bathochromic effect shown for the absorption and emission processes indicate that the dipole moment of these fluorescent molecules is higher in the excited state than in the ground state. The fluorescence quantum yield and lifetime were obtained in different solvents for DHQ **4**.

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1. Introduction

4-Quinazolinones and their derivatives are an important class of *N*-heterocycles due to their wide range of interesting biological and pharmaceutical properties. These include hypnotic, sedative, analgesic, anti-convulsant, anti-tussive, anti-bacterial, anti-diabetic, anti-inflammatory, anti-tumor activities and others [1]. Among the quinazolin-4(3H)-ones and 2,3-dihydroquinazolin-4(1H)-ones are two of the most studied derivatives, where the first compound can easily be converted into the second by oxidation [2] or through free radical reactions, which can be carried out under UV irradiation [3].

Derivatives of quinazolin-4-one are lesser known dyes and/or pigments, and their luminescent properties are only minimally studied. Derivatives that have been reported on include quinazolin-4(3H)-ones with a styryl **a** [4] or 2-hydroxyphenyl **b** [5] moiety at position 2.
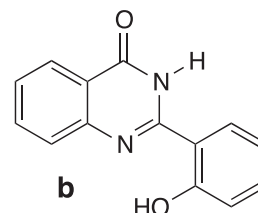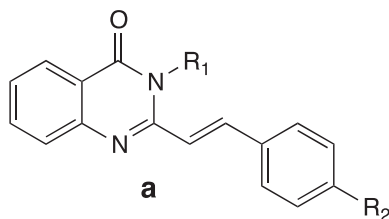
A very important example is the phosphate CPPCQ **c**, which is used as fluorogenic substrate in the field of Enzyme-Labeled Fluorescence (ELF). The CPPCQ **c** is soluble in water and upon enzymatic cleavage of the phosphate group, this weakly blue-fluorescent substrate yields an extremely photostable yellow–green fluorescent precipitate (ELF alcohol **d**), with maximal excitation at 360 nm and maximal emission at 530 nm [6].

Phosphatase

c

d
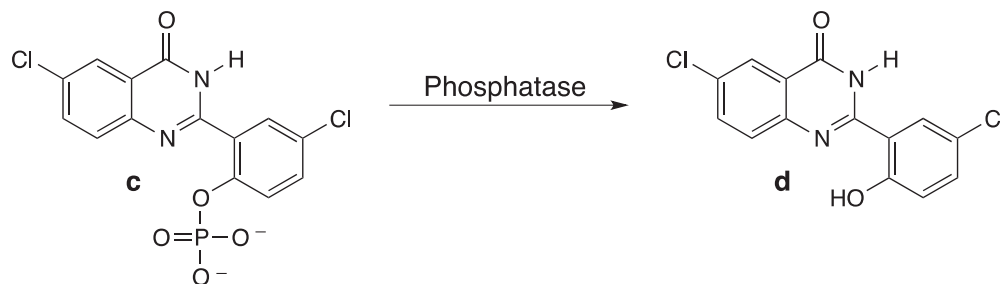
Fluorescent organic molecules have a long history of interest owing to their potential applications in electronics optic, sensors [5] and as materials for organic light emitting diodes (OLEDs) [4]. Certain DHQ derivatives may also have interesting features in their electronic spectra but their photophysical behavior has not been extensively studied. Here we report the spectroscopic and photophysical properties of several 2,3-dihydroquinazolin-4(1H)-one derivatives.

2. Material and methods

Measurement of the molar extinction coefficients was carried out in several solvents at different concentrations (in triplicate) ranging from 1 to 4×10^{-4} M. An HP 8453 UV/VIS spectrophotometer was used to measure the absorbance of the samples. The molar extinction coefficient was calculated by averaging the slope of the absorption vs concentration lines for six different measurements. Excitation and emission spectra were collected using a Perkin-Elmer LS-55 fluorimeter and recorded using solutions with concentrations between 8×10^{-7} M and 8×10^{-6} M in a 1 cm cuvette.

Quantum yields were determined by an absolute method using an integrating sphere[7]; the experiments were carried out on a FLS920 Fluorescence Spectrometer from Edinburgh Instruments. The quantum yields were also determined by a comparative method where one uses a standard that has similar fluorescence properties as the compound. At least six concentrations in triplicate were measured in order of increasing concentration to obtain the quantum yield of the fluorescence; the samples and standard were measured under the same environmental conditions and instrumental settings (25 °C, Slits 5, λ_{exc} 345 nm). To avoid photodegradation, fresh samples were used for each measurement.

Fluorescence quantum yields of **4** were measured in different solvents and were determined by the comparative method using quinine sulfate as a reference [8,9] and by the integrating sphere technique [7]. For the comparative method, fluorescence quantum yields were calculated according to the following equation:

$$\phi_{\mathrm{F}} = \phi_{\mathrm{F(std)}} \left(\frac{m}{m_{\mathrm{std}}} \right) \left(\frac{n^2}{n_{\mathrm{std}}^2} \right)$$

where ϕ_{F} and $\phi_{\mathrm{F(std)}}$ are the quantum yields of fluorescence of the compound and the standard; m are the slopes of the graphs plotted between the integrated areas of the emission spectra and the absorbances of the samples and the standard; n and n_{std} are refractive indexes of solvent of the probe and the reference, respectively [9b].

The fluorescence lifetimes were recorded on a Fluorescence Spectrometer model FLS920 (Edinburgh Analytical Instruments) using the time correlated photon counting (TCSPC) method. A Nd:YAG laser was used as the excitation source.

2.1. Synthesis and characterization

2.1.1. General procedure for the synthesis of aminobenzamides (GP-1)

A suspension of isatoic anhydride and 1.1 equiv of aryl- or benzyl-amine in EtOAc was stirred at 40 °C or irradiated using microwave heating equipment. After the reaction was completed, as indicated by TLC (eluent hexane:EtOAc 6:4), the brown solution was filtered in a Büchner funnel that had been packed with a layer of celite and activated charcoal; the colorless solution was then evaporated under reduced pressure.

2.2. General procedure for the synthesis of 2,3-dihydro-4(1H)-quinazolinones (GP-2)

1.2 equiv of appropriate aldehyde was added to a solution of aminobenzamide in CH_2Cl_2. To this, 2–5% by weight of p-TsOH was added and refluxed. The reactions were carried out in the absence of light due to possible degradation reactions[3]; the reaction was monitored by TLC (hexane:EtOAc 6:4). A final straw yellow solution was obtained and the crude reaction mixture was purified by flash chromatography eluting with hexane:EtOAc, 9:1–6:4.

2.3. General procedure for the halogenation reaction (GP-3)

2.1 equiv of Et_3N and 2.0–2.27 equiv of Br_2 or 4.2 equiv of NCS was added to a solution of 2,3-dihydro-4(1H)-quinazolinone (0.34 mmol) in $CHCl_3$ (5 mL). The solution was stirred and heated at 50 °C. The colored solution was monitored by TLC (hexane:EtOAc 6:4) until the starting material has completely disappeared. The reaction mixture was evaporated under reduced pressure and the crude of reaction was suspended in water (5 mL), which was extracted with EtOAc ($3 \times$ 5 mL). The organic layer was dried with anhydrous sodium sulfate and evaporated. The residue was purified by flash chromatography eluting with Hex:EtOAc, 9:1–6:4.

## 3. Results and discussion

3.1. Synthesis

2,3-Dihydroquinazolin-4(1H)-one derivatives **4–7** (Scheme 1) were synthesized following the methodology previously reported by our group [10]. The reaction of isatoic anhydride with different amines in ethyl acetate yields the aminobenzamides **1–3**. As shown in Scheme 1, cyclocondensation of aminobenzamides with the appropriate aldehyde in dichloromethane and p-TsOH monohydrate gives the corresponding 2,3-dihydroquinazolin-4(1H)-one.

2,3-Dihydroquinazolin-4(1H)-one **9** was obtained by cyclocondensation of **1** with triethyl orthoformate in toluene, followed by reduction of **8** with 2 equiv of $NaBH_4$ in a 1:1 mixture of THF/MeOH (Scheme 2).
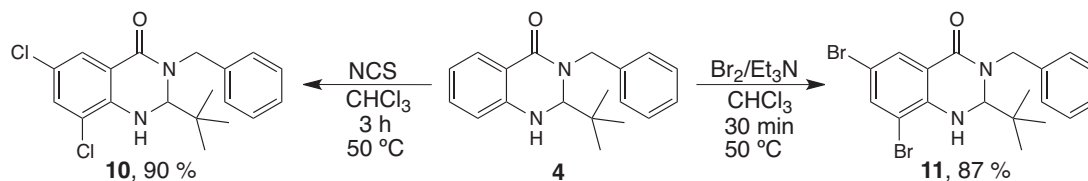
The halogenated 2,3-dihydroquinazolin-4(1H)-ones (**10** and **11**) were obtained by direct halogenation of **4** with NCS and Br_2/Et_3N, respectively (Scheme 3) [11].
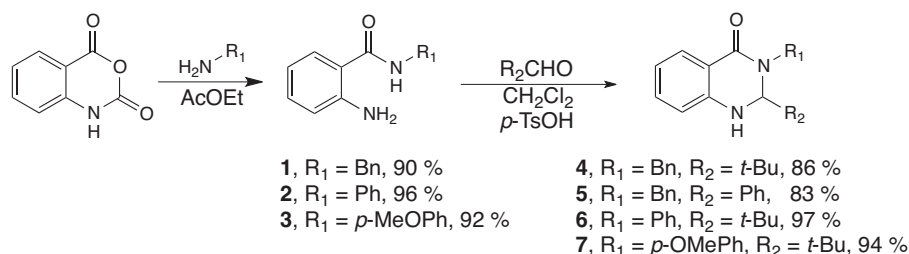
Scheme 1. Synthesis of 2,3-dihydroquinazolin-4(1*H*)-one derivatives **4**–**7**.
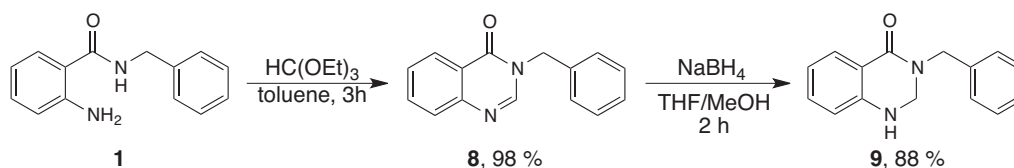
Scheme 2. Synthesis of 2,3-dihydroquinazolin-4(1*H*)-one **9**.

Scheme 3. Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones **10** and **11**.

3.2. Photophysical properties

We have measured the photophysical properties of 2,3-dihydroquinazolin-4(1*H*)-one derivatives **4**–**7** and **9**–**11** in chloroform. The results are listed in Table 1. All compounds display similar absorption and emission profiles (Figs. 1 and 2); we notice a small difference in the absorption band for **6** and emission band for **11**: they are a bit broader than the corresponding bands of other derivatives. The excitation spectra of the **4**–**7** and **9**–**11** derivatives are mirror images of their emission spectra. From their crossing points, the corresponding energies of the excited singlet states (E_{0-0}) were determined to lie between 25,800 and 27,500 cm^{-1}.

Comparing compounds **4**, **5** and **9** which have different substituents at position 2, we can see that the substitution of *t*-Bu at position 2 of quinazolinone **4** by phenyl **5** or hydrogen **9** results

in a blue shift of the absorption and emission band maxima. We also observe that the molar absorptivities are similar, whereas the Stokes shift is similar for **4** and **5** but smaller for **9**.

On the other hand, substitution of benzyl at position 3 (**4**) by phenyl **6** or *p*-methoxyphenyl **7** results in a small red-shift of the absorption and emission band maxima, and a small increase of the Stokes shift, while molar absorptivity increases in the order: **4** < **6** < **7**. Compounds **6** and **7** have almost the same absorption and emission maxima, so they have almost the same Stokes shift. The presence of an —OMe group on the phenyl ring does not alter the absorption or emission maximum, but it does cause an increase in the molar absorption coefficient. The absorption and emission maxima of **10** and **11** are red-shifted in relation to compound **4** as a result of the presence of halogens at the 6 and 8 positions. The molar extinction coefficient is also affected by the presence of the
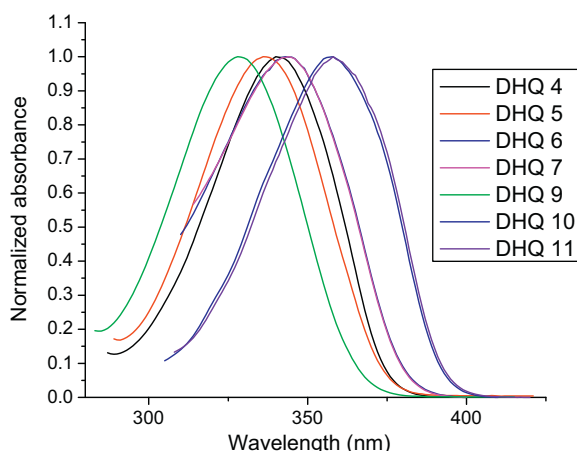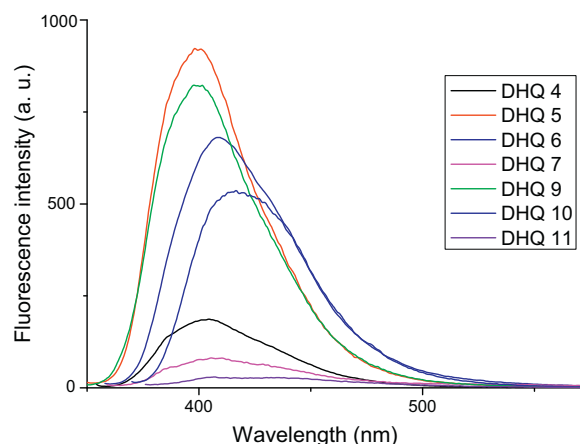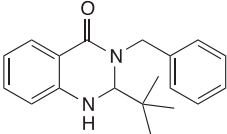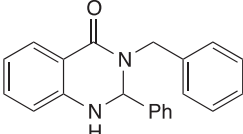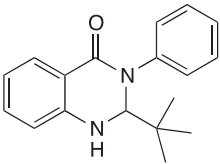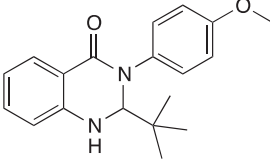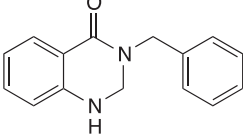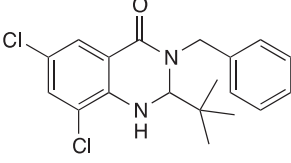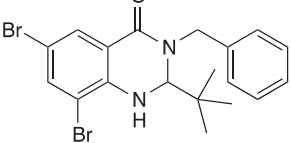

Fig. 1. Normalized absorption spectra of **4**–**7**, **9**–**11** in CHCl₃.

Fig. 2. Emission spectra for **4**–**7**, **9**–**11** in CHCl₃.

Table 1
Electronic absorption and photoluminescence spectral properties of compounds **4–7**, **9–11** in chloroform.

Compound	λ_{abs} (nm) (FWHM)[a]	ε (M^{-1} cm^{-1})	λ_{em} (nm) (FWHM)[a]	$\Delta\gamma_{stokes}$ (cm^{-1})	E_{0-0} (cm^{-1})	ϕ_F[b] (±10%)
4	341 (211,300)	3000	404 (174,840)	4573	27,000	0.055
5	336 (213,900)	2900	398 (190,840)	4636	27,100	0.405
6	344 (175,900)	4130	409 (26,600)	4620	26,800	0.135
7	344 (196,950)	4820	410 (165,300)	4680	26,700	0.006
9	329 (213,750)	3070	398 (182,600)	5269	27,500	0.089
10	357 (203,600)	3720	415 (169,820)	3915	25,900	0.100
11	359 (210,370)	3920	415 (116,880)	3759	25,800	0.002

[a] Full-width at half-maximum as measured in wavenumbers.
[b] ϕ_F Comparative method; SQ ϕ_F 0.55 [H$_2$SO$_4$ 0.5 M], [12] λ_{exc} = 345 nm, 25 °C, with a single point.

halogens; it increases in the order **4** < **10** < **11** while the Stokes shift decreases for DHQ **4** > **10** > **11**.

Fig. 2 shows the fluorescence emission for **4–7**, **9–11**; the absorption maxima were used as excitation wavelength to carry out the emission measurements. As seen in Fig. 2, the strongest photoluminescence is observed for derivative **5**, due to the presence of the phenyl group at the position 2. Moreover, derivatives **5**, **6**, **9** and **10** have stronger luminescence than **4**, while **7** and **11** have weaker fluorescence than **4**. Due to an internal heavy atom effect, the weakest luminescence was observed for compound **11**.

Finally, fluorescence quantum yields (ϕ_F) of DHQ derivatives vary dramatically throughout these chemical modifications, from 0.43 (the highest value corresponds to **5**) to 0.002 for **11** (the lowest value). Comparing the ϕ_F values for **4**, **5** and **9**, we hypothesize that the smaller ϕ_F value for **4** is due to the elimination photochemical reaction. This seems to imply that photophysical and photochemical process are competing. Since neither DHQ **5** or **9**

has an appropriate substituent to form a stable free radical, they can only release the absorbed energy through the photophysical processes. The ϕ_F value is higher for these derivatives. In addition, derivative **5** has an aromatic ring at position 2, and this also increases ϕ_F.

Similar to DHQ **4**, compounds **6** and **7** are sensitive to light and therefore have a low fluorescence quantum yield.

For compound **10** we found that the presence of Cl does not diminish the fluorescence process but rather, we found that the ϕ_F value is higher compared to DHQ **4**, probably due to the fact that DHQ **10** is not as sensitive to light as compound **4**.

On the other hand, the presence of bromine in DHQ **11** practically inhibits the fluorescence process; the fact that DHQ is less sensitive to light suggests that state leading to homolytic rupture between the *tert*-butyl carbon and the carbon 2 is the excited singlet state.
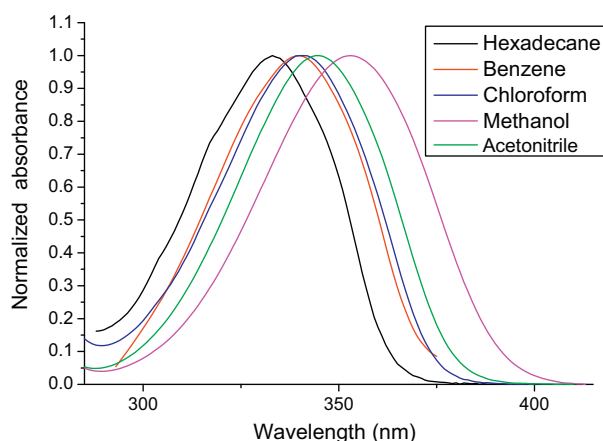
Fig. 3. UV absorption spectra of **4** in different solvents.

3.3. Solvent dependence

To probe the excited state of DHQs further, we examined the effect of solvent on the absorption and photoluminescent properties of 3-benzyl-2-*tert*-butyl-2,3-dihydro-4(1*H*)-quinazolinone **4**. These results are shown in Table 2. Hexadecane, benzene, chloroform, methanol, and acetonitrile were used to cover a range of solvent polarity–polarizability (SPP), acidity (SA), and basicity (SB). The various solvent parameters are listed in Table 2.

Normalized UV spectra of **4** in different solvents are shown in Fig. 3; where absorption band maxima (λ_{abs}) are located between 333 and 344 nm. Here, the absorption band maxima shift to higher wavelengths (lower energies) as the dielectric constant and H-bonding ability of the solvent increases. The absorption band of **4** in methanol is wider than in the other solvents, while the absorption band in hexadecane is the narrowest.

The values of the molar extinction coefficient did not change significantly between 2900 and 3000 $M^{-1}\,cm^{-1}$ in different solvents. The bathochromic effect, due to the polarity of the solvent and the molar extinction coefficient, suggest that the signal corresponds to a π–π^* electron transition. [13]

Fig. 4 shows fluorescence spectra of **4** in different solvents, the spectral maxima appeared between 381 and 439 nm. In the case of chloroform and acetonitrile, the emission spectra are almost identical. Comparing the emission spectra of **4** in different solvents we can see that they display a significant red shift upon increasing solvent polarity. This trend indicates an increase in molecular dipole on going from the ground state (S_0) to the lowest singlet excited state (S_1). This is a common photophysical feature of organic compounds with an electron donor such alkylamino group and an acceptor moiety such as C=O attached to an aromatic ring [14].

Absorption spectra of **4** obtained in different solvents have the same profile as the excitation spectra, but the absorption is wider than the excitation band and they show profiles that are mirror images or their emission spectra, in agrement with
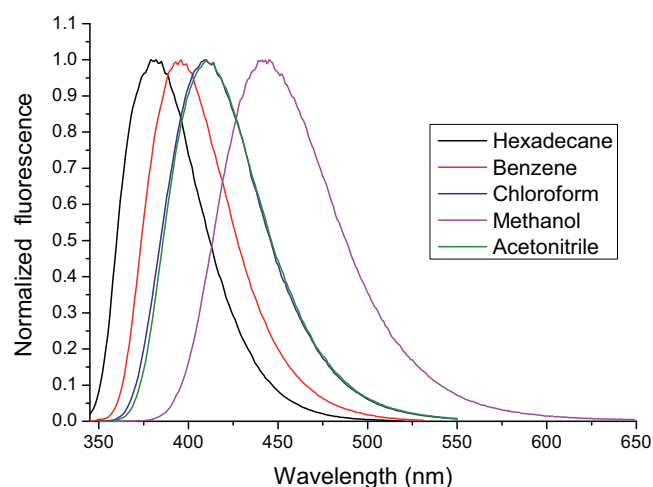

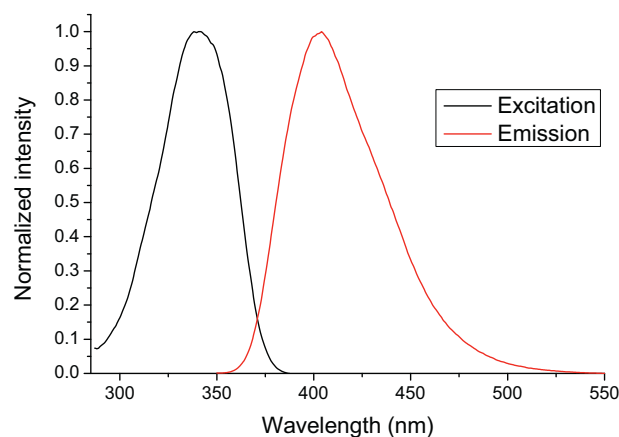**Fig. 4.** Fluorescence spectra of DHQ **4** in different solvents, 25 °C, λ_{exc} = 345 nm.

Fig. 5. Normalized excitation and emission spectra of **4** in chloroform, 25 °C, λ_{exc} = 335 nm, λ_{em} = 404 nm.

the Franck–Condon principle and Kasha's rule for both absorption and emission (for example, **4** in chloroform). Compound **4** shows large Stokes shifts in the range of 3783–5549 cm^{-1} (Table 2). Which increases with emprical solvent polarity and solvent basicity (Fig. 5). The greater stabilization of the excited state relative to the ground state with increasing solvent basicity supports the assignment of the excited state as a charge transfer state with an amine donor.

Also, from the crossing points of the normalized excitation and emission spectra the corresponding energies of excited singlet were estimated to lie between 73 and 77 kcal/mol. [15]

The fluorescence quantum yield using the comparative method of **4** in CHCl$_3$ and CH$_3$OH were found to be 0.055 and 0.034, respectively. Absolute fluorescence quantum yields in C$_6$H$_6$ and CH$_3$CN were determined using a calibrated integrating sphere system and

Table 2
Solvent dependent photophysical properties of **4** in different solvents.

Solvent	λ_{abs} (nm) (FWHM)[a]	ε ($M^{-1}\,cm^{-1}$)	λ_{em} (nm) (FWHM)[a]	$\Delta\gamma_{stokes}$ (cm^{-1})	E_{0-0} (cm^{-1})	ϕ_F ($\pm10\%$)	τ_s (ns)
n-C$_{16}$H$_{34}$	333 (227,380)	–	381 (197,730)	3783	27900	–	4.97
C$_6$H$_6$	339 (213,790)	2971	393 (193,900)	4053	25,900	0.080[b]	5.11
CHCl$_3$	341 (221,940)	3012	404 (169,220)	4573	27,000	0.055[c]	5.86
CH$_3$OH	353 (203,850)	2919	439 (137,400)	5549	25,500	0.034[c]	5.24
CH$_3$CN	344 (221,640)	2906	409 (171,470)	4619	26,700	0.090[b]	2.43

[a] Full-width at half-maximum as measured in wavenumbers.

[b] ϕ_F Integrating sphere technique, 25 °C.

[c] ϕ_F Comparative method; SQ ϕ_F 0.55 [H$_2$SO$_4$ 0.5 M], [12] λ_{exc} = 345 nm, 25 °C.
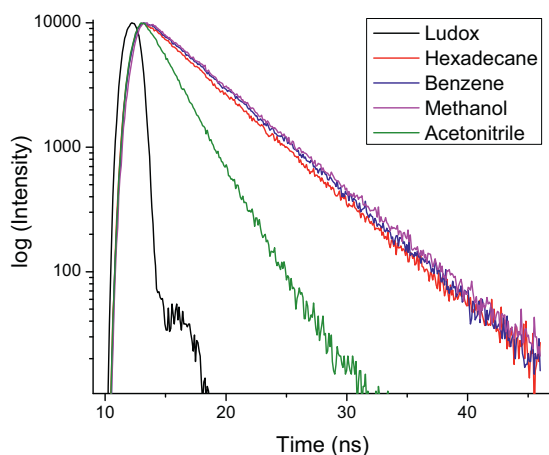
Fig. 6. Fluorescence decay curves and the instrument response function (Ludox). For excitation, a pulsed laser with a repetition rate of 10 MHz and pulse length of 200 ps (full width half maximum) was used.

were found to be 0.08 (8%) and 0.09 (9%). The fluorescence lifetime (τ_s) of **4** was determined using time-correlated single photon counting (TCSPC), recording at the emission maxima in the corresponding solvent. The lifetime of **4** in hexadecane, benzene, chloroform and methanol was about 5 ns, while in acetonitrile 2.43 ns, the kinetics followed single exponential decay independent of solvent (Fig. 6).

Closer examination of the solvent effect can provide insight into the nature of the lowest energy excited state. We can fit the spectral shifts to linear functions of a global solvent parameter, P, using solvent parameters provided by Catalán et al. [16] (Table 3) and supplying proportionality constants, (avg. $R^2 = 0.97$). The parameter P, as it applies to **4**, is equal to $0.53 \cdot SPP + 0.09 \cdot SA + 0.38 \cdot SB$, where SPP is the solvent polarity-polarizability, SA is the solvent acidity,
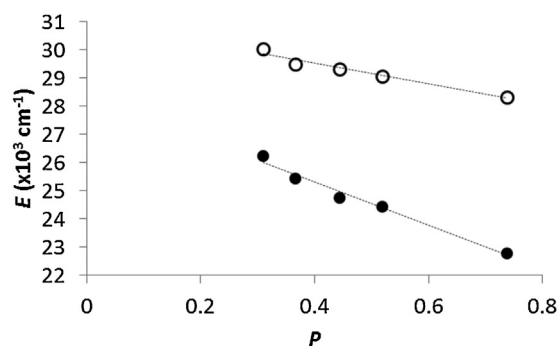

Fig. 7. Plot of the absorption and emission maxima as a function of empirical solvent parameters, where $P = 0.53 \cdot SPP + 0.09 \cdot SA + 0.38 SB$.

Table 3
Various empirical parameters for solvents used in this study.[a]

Solvent	SPP	SA	SB
n-$C_{16}H_{34}$[b]	0.57	0.09	0
C_6H_6	0.67	0.12	0
$CHCl_3$	0.79	0.07	0.05
CH_3OH	0.86	0.55	0.61
CH_3CN	0.9	0.29	0.04

[a] Parameters from Ref. [16b]. SPP is the solvent polarity–polarizability, SA is the solvent acidity and SB is the solvent basicity.
[b] Parameters reported for dodecane.

and SB is the solvent basicity (Fig. 7). Fits to single solvent parameters were generally poor ($R^2 = 0.6$ to 0.8). While the dependence on solvent polarity supports stabilization of the excited state, the dependence on solvent basicity suggests a large shift along the atom coordinate due to elongation of the N—H bond in the excited state. This is consistent with our assignment of lowest excited state as a CT state.

Table 4
Solvent dependent Photophysical properties of **5–7**, **9–11** in different solvents.

Compound	Solvent	λ_{abs} (nm)	FWHM (cm^{-1})	ε (M^{-1} cm^{-1})	λ_{em} (nm)	FWHM[a]	$\Delta\gamma_{stokes}$ (cm^{-1})	E_{0-0} (cm^{-1})
5	n-$C_{16}H_{34}$	325	186,943	–	383	212,230	4660	28,063
	C_6H_6	334	191,905	2540	393	195,810	4495	27,417
	$CHCl_3$	336	213,900	2900	398	190,840	4636	27,100
	CH_3OH	345	175,580	2515	442	150,927	6721	25,490
	CH_3CN	337	212,288	2364	407	177,056	5104	26,884
6	n-$C_{16}H_{34}$	337	178,507	–	388	189,638	3901	27,569
	C_6H_6	343	179,439	3730	400	170,736	4155	26,912
	$CHCl_3$	344	175,900	4130	409	166,970	4620	26,800
	CH_3OH	356	179,962	3630	449	156,129	5818	25,136
	CH_3CN	348	185,308	3665	417	161,840	4755	26,189
7	n-$C_{16}H_{34}$	–[a]	–	–	–[a]	–	–	–
	C_6H_6	342	196,094	3600	400	54,620	4240	26,413
	$CHCl_3$	344	196,950	4820	410	165,300	4680	26,700
	CH_3OH	355	172,827	4014	444	149,982	5646	25,382
	CH_3CN	348	191,124	3300	413	164,637	4405	26,251
9	n-$C_{16}H_{34}$	323	226,002	–	381	203,036	4713	28,388
	C_6H_6	329	221,028	2880	395	189,673	5079	27,680
	$CHCl_3$	329	213,750	3070	398	182,600	5269	27,500
	CH_3OH	340	204,213	3058	443	153,637	6839	25,687
	CH_3CN	332	217,012	2700	416	143,509	6082	25,945
10	n-$C_{16}H_{34}$	350	228,657	–	402	184,673	3695	26,600
	C_6H_6	355	213,440	2760	407	185,214	3599	26,237
	$CHCl_3$	357	203,600	3720	415	169,820	3915	25,900
	CH_3OH	367	199,563	3270	448	154,832	4927	24,857
	CH_3CN	360	207,170	2731	420	164,586	3968	25,834
11	n-$C_{16}H_{34}$	351	226,634	–	399	186,260	3427	26,617
	C_6H_6	356	222,083	2985	–[b]	–	–	–
	$CHCl_3$	359	210,370	3920	415	116,880	3759	25,800
	CH_3OH	368	205,391	3810	447	–	4803	24,978
	CH_3CN	360	187,692	3070	422	160,341	4081	25,967

[a] The derivative was insoluble and no absorption or emission was observed.
[b] No fluorescence was observed.

DHQ derivatives **5**–**7**, **9**–**11** showed a similar photophysical behavior in different solvents as the compound **4**. The spectral features are reported in Table 4.

A solvatochromic effect could be observed for these derivatives; the absorption maximum is between 323 and 368 nm and shifts to the red as the polarity of the solvent increase. In agreement to the values obtained from FWHM, the absorption bands of almost all derivatives are wider in methanol than in other solvents, while the narrowest absorption bands were found to be in hexadecane.

As seen in Table 4, the molar absorption coefficient ranges between 2364 and 4820 $M^{-1}\,cm^{-1}$; the highest measured values for ε were obtained in chloroform.

Concerning the fluorescence emission maxima, the DHQ derivatives also showed a bathochromic effect according to the polarity and the ability to form hydrogen bonds of the solvent. The emission maxima of the compounds **5**–**7**, **9**–**11** is located between 381 and 449; on the other hand, the wider emission bands for almost all compounds were observed in methanol, the exception being DHQ **7**, in which the very broad emission bands of benzene were observed and the DHQ **9** in which the widest emission band was obtained in acetonitrile.

The Stokes shift also increases with solvent polarity, and as a consequence the biggest difference between the maximum of the absorption and the maximum of the fluorescence band is observed in methanol and a minimum difference is observed in hexadecane.

Finally, the energies of the excited singlet state (E_{0-0}) were determined to be 24,857–28,063 cm^{-1}.

4. Conclusions

Absorption and emission maximum for DHQ **4** were found to be red shifted as a function of solvent; the Stokes shifts of these derivatives were also affected by polarity and the ability of the solvent to form hydrogen bonds. Therefore, methanol was the solvent that caused the highest red shift and the biggest difference between absorption and emission maximum (Stokes shift). On the other hand, the molar extinction coefficients were found not to vary as a function of the solvent. Fluorescence quantum yield of **4** in different solvents was found to be between 0.034 and 0.090.

2,3-Dihydroquinazolin-4(1*H*)-one derivatives with different substituents at the 2, 3, 6 and 8 positions were synthesized; these compounds have the same profiles of absorption and emission than **4**. Their spectral shapes consist of a single broad band, with a maximum that is solvent dependent; the results suggest that the transitions involved here are π–π* in nature. The 2,3-dihydroquinazolin-4(1*H*)-one compounds exhibit attractive photophysical properties and for this reason they are good candidates for deeper investigations.

Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.jphotochem.2014.07.005.

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