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# 4-substituted push-pull quinazoline chromophores with extended $\pi$ -conjugated linker

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## Abstract

A series of four push-pull compounds based on a 4-phenylquinazoline scaffold were designed. Three of these compounds exhibit dual state emission with intense fluorescence in solution and solid state. Their emission properties are sensible to environment stimuli with intense emission solvatochromism and halochromism. In case of methoxy derivative, both neutral and protonated form are luminescent and white light emission can be observed by controlled partial protonation.

## 1 | INTRODUCTION

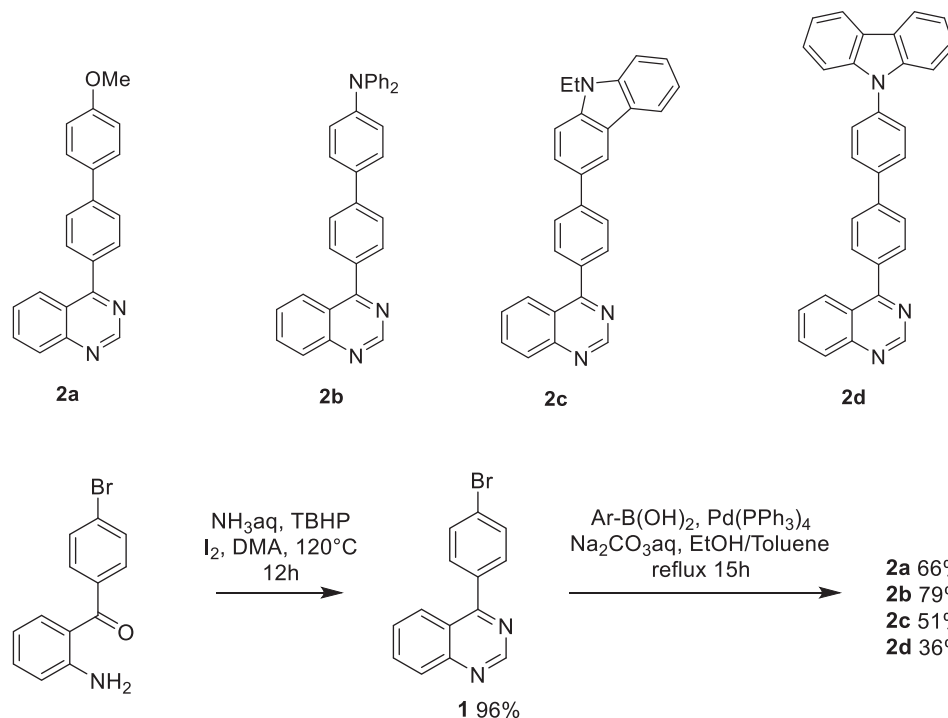
Quinazoline, which corresponds to benzopyrimidine, is a bicyclic azaheterocycle with two nitrogen atoms in position 1 and 3. The quinazoline derivatives have been extensively studied for their biological (anticancer, antibacterial, anti-inflammatory, antimalarial, antihypertensive...) properties [1–5]. Quinazoline derivatives can be also used as ligand of various metal cations [6–8]. More recently, their photophysical properties have begun to be investigated [9–13]. Due to the  $\pi$ -deficient character of quinazoline, quinazolynyl fragments can be used as electron-withdrawing part in push-pull structures [9, 10, 14–16]. Electron-lone pairs of nitrogen atoms can be used for complexation [14–16]. Due to the possibility of extended delocalization into the annelated benzene ring, the quinazolynyl fragments exhibit stronger electron-withdrawing character than their pyrimidinyl analogues [17, 18].

Moreover, involvement of nitrogen electron lone pairs in complexation, protonation, or hydrogen bonding will tune the electron-withdrawing strength of quinazoline and therefore will have influence on intramolecular charge transfer (ICT) into the push-pull chromophores. The quinazoline ring exhibits a basic character ( $pK_a = 3.3$ ) and can be easily protonated, on the other hand, the second nitrogen atom can be hardly protonated [19]. The corresponding quinazolium protonated form exhibits a stronger electron-withdrawing character and ICT is increased leading to red-shifted absorption [20]. Often, protonation of push-pull azaheterocyclic chromophores leads to extinction of emission, but in some cases red-shifted emission is observed and white light emission can be observed through partial protonation by combination of blue emission of the neutral form and orange/red emission of protonated form [21–23] and have found application as white organic light emitting diodes [20]. The emission properties of push-pull quinazoline chromophores are therefore highly sensible to their environment [20, 24, 25].

Thibault Bonnaud and Mathieu Scaviner share first authorship.

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**CHART 1** Structure of quinazoline chromophores **2a-d**.

**SCHEME 1** Synthesis of compounds **2a-d**.

In diazine-based chromophores,  $\pi$ -conjugation extension, for example with biphenylene linkers has a positive effect on emission properties, in particular with moderately strong electron-donating groups such as methoxy [18, 21].

In this contribution, we designed a series of 4-phenylquinazoline chromophores **2a-d** (Chart 1). We combined in this work the stronger electron-withdrawing character and the higher pKa value of quinazoline ring compared to pyrimidine with the positive effect of biphenylene linker on photoluminescence quantum yield. We thoroughly studied the emission properties of studied chromophores in solvent of various polarity as well as the effect of protonation.

## 2 | RESULTS AND DISCUSSION

### 2.1 | Synthesis

Compounds **2** were obtained in two steps from commercially available 2-amino,4'-bromobenzophenone (Scheme 1). The first step consists in a cyclocondensation with  $\text{NH}_3$ , in the presence of *tert*-butylhydroperoxide and iodine in *N,N*-dimethylacetamide (DMA) according to reported procedure [26]. The second step involves a palladium-catalyzed Suzuki–Miyaura cross-coupling reaction with the corresponding commercially available boronic acid. Compounds **2a-d** were obtained with moderate to good

yield and their structure was confirmed by  $^1\text{H}$  and  $^{13}\text{C}$  NMR and high-resolution mass spectrometry.

### 2.2 | Photophysical properties

The photophysical properties of chromophores **2a-d** were examined by UV–vis and photoluminescence (PL) spectroscopy in  $\text{CH}_2\text{Cl}_2$  at room temperature. The data are summarized in Table 1 and normalized spectra are provided in Figure 1. All compounds exhibit two absorption bands in the UV-part of the spectrum. The less energetic one is attributed to transition with a charge transfer character. This band is red-shifted in the following order **2a** < **2d** < **2c** < **2b** in accordance with the strength of the electron-donating substituent. Compounds **2a-d** exhibit blue to orange fluorescence with rather high photoluminescence quantum yield (PLQY) ( $\Phi_F > 0.35$ ) with the emission band that is red-shifted in the same order as the absorption. These compounds display particularly large Stokes shifts ( $> 8000\text{ cm}^{-1}$ ) indicating high polarizability of the molecules and important difference between ground and excited state geometry with planarization of (bi)phenylene linkers. Whereas biphenylene derivative **2b** and **2d** exhibit lower PLQY compared to phenylene analogue [16, 19], in case of methoxy derivative **2a**, the biphenylene linker induces a dramatic increase of PLQY up to 0.38 (4-(4-methoxyphenyl)quinazoline exhibits a PLQY below 0.01 in  $\text{CH}_2\text{Cl}_2$ ). This is in

TABLE 1 UV-vis and photoluminescence data of compound **2a–d** in CH<sub>2</sub>Cl<sub>2</sub>.

Compd <sup>a</sup>	UV-vis $\lambda_{max}$ , nm ( $\epsilon$ , mM <sup>-1</sup> ·cm <sup>-1</sup> )	PL $\lambda_{max}$ , nm	$\Phi_F^b$	Stokes shift cm <sup>-1</sup>
<b>2a</b>	328 (21.0)	452	0.38	8363
<b>2b</b>	303 (27.0), 366 (24.6)	593	0.37	10,458
<b>2c</b>	350 (19.8)	512	0.63	9040
<b>2d</b>	328 (27.0), 344 (25.8)	511	0.35	10,918

<sup>a</sup>All spectra were recorded at room temperature at  $c = 1.0 \times 10^{-5}$ – $2.0 \times 10^{-5}$  M.

<sup>b</sup>Fluorescence quantum yield ( $\pm 10\%$ ) determined relative to 9,10-bis-phenylethynyl-anthracene in cyclohexane ( $\Phi_F = 1.00$ ) [27].

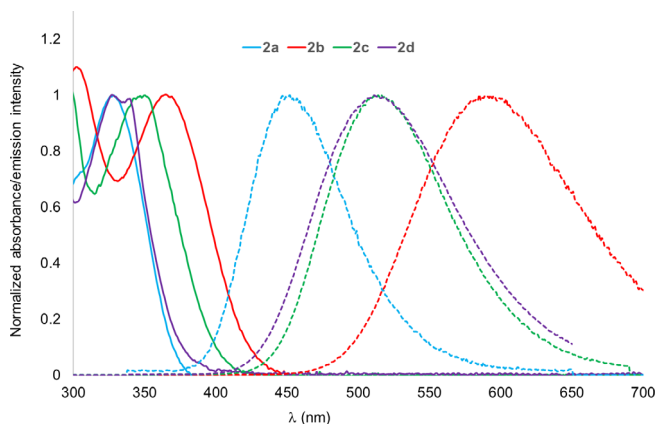


FIGURE 1 Normalized absorption (solid lines) and emission (dashed lines) spectra of compounds **2a–d** in CH<sub>2</sub>Cl<sub>2</sub> ( $c = 1.0 \times 10^{-5}$ – $2.0 \times 10^{-5}$  M).

accordance with observation of other series of diazine chromophores [20].

In order to study ICT charge transfer into compounds **2a–d**, the photophysical properties were measured in a series of aprotic solvents of increasing polarity. Whereas no significant shift of charge transfer absorption band is observed, a positive emission solvatochromism is shown: when the polarity is increased according to Reichardt polarity scale [28], a bathochromic shift of emission is observed (Table 2, Figures 2 and S1–S6). This is explained by the stabilization of the highly polarized excited state in polar solvents [29–31]. The positive emission solvatochromism is associated with broader emission band and decreased emission intensity in the more polar solvents. Stokes shifts were plotted versus the Reichardt polarity parameter  $E_T(30)$  (Figure S3) and as expected the slope of the regression line increased in the following order: **2a** < **2c** < **2d** < **2b** in accordance with the strength of the corresponding electron-donating groups. Compound **2b** exhibits a particularly large solvatochromic range covering the whole visible spectrum with blue emission in nonpolar heptane up to red/near infrared in MeCN (Figure 2).

Solid state emission of compounds **2a–c** was also demonstrated. Compounds **2a**, **2b**, and **2c** diluted in KBr

matrix exhibit respectively sky blue, green, and blue-green emission with high intensity (Figure 3). These compounds can be therefore considered as efficient dual-state emitters [32, 33]. Compound **2d** is much less emissive in solid state.

The effect of acidic media on the photophysical properties of compounds **2** was also tested with acidified CH<sub>2</sub>Cl<sub>2</sub> solution containing trifluoroacetic acid (TFA) ( $10^{-2}$  M). Photophysical properties of protonated form are summarized in Table 3. Whereas an extinction of emission is observed for compounds **2b–d**, the addition of acid leads to bathochromically shifted absorption and emission with an increased PLQY for **2a**. Figure 4 displays the modification of absorption and emission spectra upon progressive addition of TFA. In terms of emission a progressive disappearance of the emission band of the neutral form centered at 452 nm is observed whereas a new red-shifted band corresponding to the protonated form centered at 599 nm progressively appears. A progressive emission color change from blue to orange going through white is observed. CIE 1931 chromaticity coordinates for 12.5 eq of TFA (0.33; 0.32) were really close to pure white light (0.33; 0.33).

The effect of protonation can be also observed in solid state for compound **2a**. KBr pellets were fabricated using various amount of camphorsulfonic acid (CSA) and their emission spectra were displayed in Figure 5. A red shift of emission band is observed with increasing the amount of CSA, but no white light emission was observed.

### 3 | CONCLUSION

To conclude, this work demonstrates that push-pull 4-substituted quinazoline derivatives with extended  $\pi$ -linker exhibit particularly efficient fluorescence properties. Their sensibility to external stimuli such as solvent polarity and pH makes them interesting for sensing applications. The possibility of white light emission by controlled protonation of methoxy derivative **2a** could be interesting for the development of white light emitting diodes.

TABLE 2 Emission solvatochromism of compounds **2a–d** in various aprotic solvents.

Compd	<i>n</i> -heptane (30.9) <sup>a</sup>	Toluene (33.9) <sup>a</sup>	1,4-dioxane (36.0) <sup>a</sup>	THF (37.4) <sup>a</sup>	CHCl <sub>3</sub> (39.1) <sup>a</sup>	CH <sub>2</sub> Cl <sub>2</sub> (40.7) <sup>a</sup>	Acetone (42.2) <sup>a</sup>	MeCN (45.6) <sup>a</sup>
<b>2a</b>	433	434	436	443	443	452	465	478
<b>2b</b>	446	483	506	553	560	593	618	648
<b>2c</b>	432	451	466	493	500	516	541	559
<b>2d</b>	424	438	450	482	493	511	547	585

<sup>a</sup>E<sub>r</sub>(30) Dimroth–Reichardt polarity parameter in kcal mol<sup>-1</sup>.

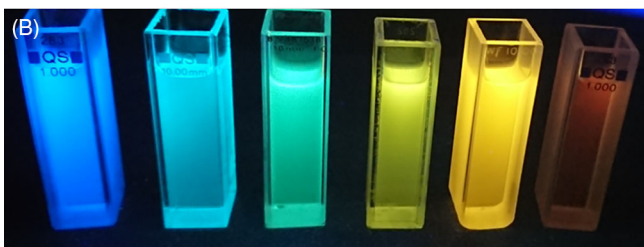
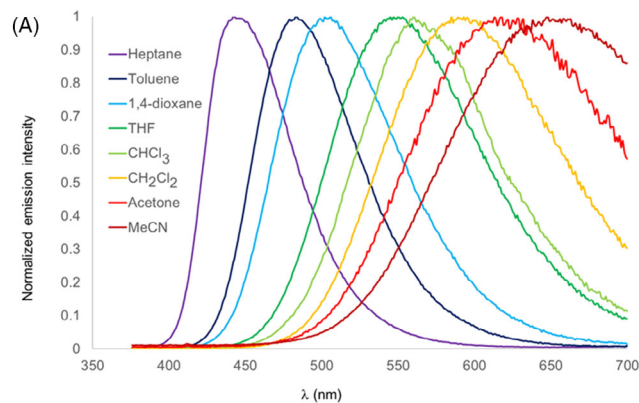


FIGURE 2 Part A: normalized emission spectra of quinazoline **2b** in a series of solvents with different polarity at 298 K part B: photographs of solution of **2b** in *n*-heptane, toluene, 1,4-dioxane, CHCl<sub>3</sub>, CH<sub>2</sub>Cl<sub>2</sub>, and MeCN (from left to right) at 298 K under irradiation ( $\lambda_{exc} = 365$  nm)  $c = 10$   $\mu$ M.

## 4 | EXPERIMENTAL SECTION

### 4.1 | Materials and methods

All solvents were reagent grade for synthesis. Starting materials were purchased from Sigma-Aldrich or TCI and were used without further purification. Thin layer chromatography (TLC) was conducted on pre-coated aluminum sheets with 0.20 mm Merck Alugram SIL G/UV254 with fluorescent indicator UV254 and 0.25 mm Merck silica gel (60-F254). Column chromatography was carried out using Mecherey-Nagel silica gel 60 (particle size 63–200  $\mu$ m). Melting points were measured on a

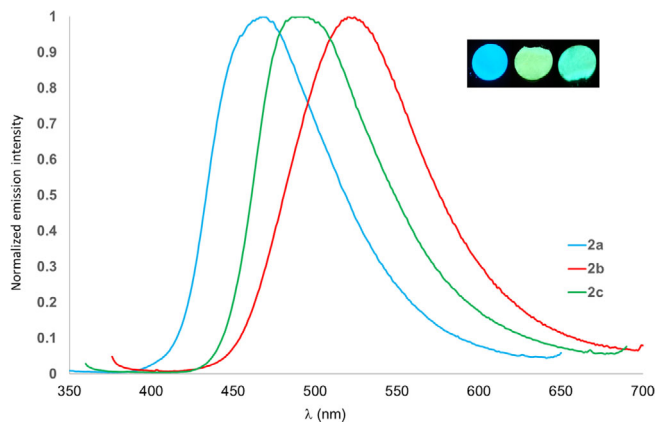


FIGURE 3 Normalized emission spectra of KBr pellets (2 wt%) of compound **2a–c**. Excitation was performed at absorption maximum in CH<sub>2</sub>Cl<sub>2</sub> solution. Inset: photographs of KBr pellets (from left to right **2a**, **2b**, and **2c**) under irradiation ( $\lambda_{exc} = 365$  nm).

Kofler hot stage. NMR spectra were recorded in CDCl<sub>3</sub> on a Bruker AC-300 spectrometer. The chemical shifts  $\delta$  are reported in ppm and are referenced to the appropriate solvent signals of CDCl<sub>3</sub> (<sup>1</sup>H,  $\delta = 7.27$  ppm; <sup>13</sup>C,  $\delta = 77.0$  ppm). The coupling constants  $J$  are given in Hz. In the <sup>1</sup>H NMR spectra, the following abbreviations are used to describe the peak patterns: s (singlet), d (doublet), t (triplet), q (quadruplet), m (multiplet). Acidic impurities in CDCl<sub>3</sub> were removed by treatment with solid K<sub>2</sub>CO<sub>3</sub>. High-resolution mass analyses were carried out at the “Centre Régional de Mesures Physiques de l’Ouest” (CRMPO, Université de Rennes) using a Bruker MicroTOF-Q II instrument. UV–visible and fluorescence spectroscopy studies were conducted on a Spex Fluoromax-3 Jobin-Yvon Horiba spectrophotometer. Compounds were excited at their absorption maxima (band of lowest energy) to record the emission spectra. All solutions were measured with optical densities below 0.1. Emission spectra are corrected. Stokes shifts were calculated considering the lowest energetic absorption band.

TABLE 3 UV-vis and photoluminescence data in acidic solution (TFA  $10^{-2}$  M in  $\text{CH}_2\text{Cl}_2$ ).

Compd <sup>a</sup>	UV-vis $\lambda_{\text{max}}$ , nm ( $\epsilon$ , $\text{mM}^{-1}\cdot\text{cm}^{-1}$ )	PL $\lambda_{\text{max}}$ , nm	$\Phi_{\text{F}}$ <sup>b</sup>	Stokes shift $\text{cm}^{-1}$
2a	265 (18.6), 401 (14.8)	599	0.51	8243
2b	306 (16.6), 331 (17.0), 491 (6.3)	- <sup>c</sup>	- <sup>c</sup>	- <sup>c</sup>
2c	287 (30.1), 340 (9.7), 463 (12.0)	- <sup>c</sup>	- <sup>c</sup>	- <sup>c</sup>
2d	292 (30.2), 360 (17.8), 422sh (7.1)	- <sup>c</sup>	- <sup>c</sup>	- <sup>c</sup>

<sup>a</sup>All spectra were recorded at room temperature at  $c = 1.0 \times 10^{-5}$ – $2.0 \times 10^{-5}$  M and were corrected.

<sup>b</sup>Fluorescence quantum yield ( $\pm 10\%$ ) determined relative to 9,10-bis-phenylethynyl-anthracene in cyclohexane ( $\Phi_{\text{F}} = 1.00$ ) [27].

<sup>c</sup>No emission detected.

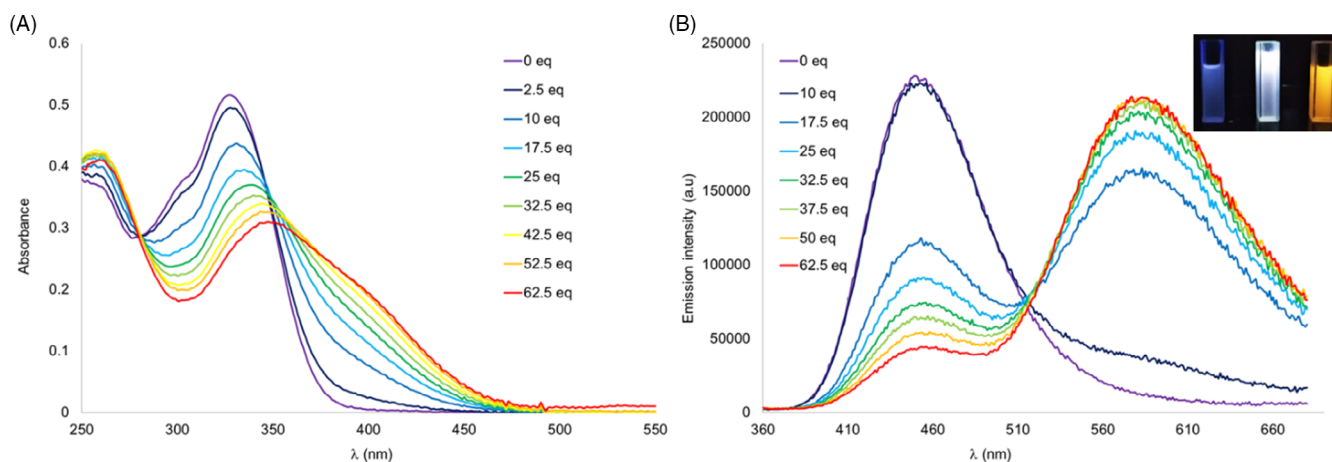


FIGURE 4 Change in absorption (part A) and emission ( $\lambda_{\text{exc}} = 350$  nm) spectra of the  $\text{CH}_2\text{Cl}_2$  solution of **2a** ( $c = 2.5 \times 10^{-5}$  M) upon addition of trifluoroacetic acid (TFA). Inset: change in the color of the  $\text{CH}_2\text{Cl}_2$  solution of **2a** after addition of 0 (left) 10 (middle) and 400 equivalents (right) of TFA, photograph was taken in the dark upon irradiation with a hand-held UV-lamp (365 nm).

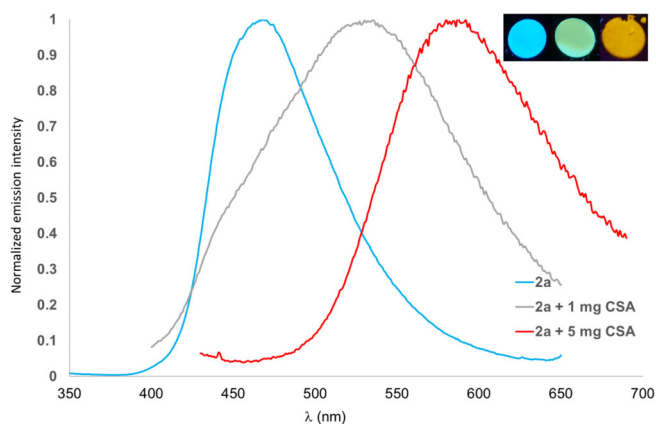


FIGURE 5 Normalized emission spectra of KBr pellets (2 wt%) of compound **2a** with different amount of camphorsulfonic acid (CSA). Excitation was performed at 340, 400, and 430 nm. Inset: photographs of KBr pellets with increasing amount of CSA from left to right under irradiation ( $\lambda_{\text{exc}} = 365$  nm).

#### 4.1.1 | Synthesis of 4-(4-bromophenyl)quinazoline (**1**)

2-amino,4'-bromobenzophenone (420 mg, 1.52 mmol),  $\text{I}_2$  (170 mg, 20 mol%), tert-butylhydroperoxyde (70% aq, 4 equiv, 785 mg),  $\text{NH}_3$  (25% aq, 2 equiv, 185 mg) were added to a 10 mL Schlenk tube, followed by addition of dimethylacetamide (15 mL). The mixture was stirred at  $120^\circ\text{C}$ . After 12 h the solution was cooled to room temperature, diluted with ethyl acetate (15 mL), washed with brine. The aqueous layers were extracted with EtOAc ( $4 \times 15$  mL), the combined organic layers were dried over  $\text{MgSO}_4$ , filtered, and evaporated under vacuum. The residue was purified by column chromatography on silica gel (petroleum ether:ethyl acetate = 3:1) to afford the desired product **1** as a colorless solid (418 mg, 96%).  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm) 9.38 (s, 1H), 8.16–8.07 (m, 2H), 7.96–7.91 (m, 1H), 7.75–7.61 (m, 5H). Data are similar to the literature [26].

#### 4.1.2 | General procedure for Suzuki–Miyaura cross-coupling reaction

A stirred mixture of 4-(4-bromophenyl)quinazoline (**1**) (285 mg, 1 mmol), the corresponding arylboronic acid (1.2 mmol), Pd(PPh<sub>3</sub>)<sub>4</sub> (60 mg, 0.05 mmol), 1 M aqueous sodium carbonate (1.2 mmol, 1.2 mL), and ethanol (1.5 mL) in degassed toluene (15 mL) was heated at reflux under nitrogen for 15 h in a Schlenk tube. The reaction mixture was cooled, filtered, and dissolved with a mixture of EtOAc and water 1:1 (50 mL) and the organic layer was separated. The aqueous layer was extracted with EtOAc (2 × 25 mL). The combined organic extracts were dried with MgSO<sub>4</sub> and the solvents were evaporated.

#### 4.1.3 | 4-(4'-methoxy-[1,1'-biphenyl]-4-yl)quinazoline (**2a**)

Purified by column chromatography on silica gel (petroleum ether: EtOAc = 3:1). Cream solid (66%, 206 mg), mp: 177–179°C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ (ppm) 9.40 (s, 1H), 8.23 (d, *J* = 8.4 Hz, 1H), 8.13 (d, *J* = 8.4 Hz, 1H), 7.94–7.91 (m, 1H), 7.87 (d, 2H, *J* = 8.4 Hz), 7.77 (d, 2H, *J* = 8.4 Hz), 7.66–7.63 (m, 3H), 7.04 (d, 2H, *J* = 8.4 Hz), 3.89 (s, 3H), <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ (ppm) 168.1, 159.7, 154.7, 151.2, 142.6, 135.4, 133.7, 132.7, 130.5, 129.0, 128.3, 127.7, 127.1, 126.9, 123.2, 114.4, 55.4. HRMS (ESI/ASAP), *m/z* calculated for C<sub>21</sub>H<sub>17</sub>N<sub>2</sub>O [M + H]<sup>+</sup> 313.1335, found 313.1337.

#### 4.1.4 | 4-(4'-*N,N*-diphenylamino-[1,1'-biphenyl]-4-yl)quinazoline (**2b**)

Purified by column chromatography on silica gel (petroleum ether: EtOAc = 3:1). Pale yellow solid (79%, 350 mg), mp: 171–173°C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ (ppm) 9.39 (s, 1H), 8.23 (d, *J* = 8.4 Hz, 1H), 8.13 (d, *J* = 8.4 Hz, 1H), 7.93–7.91 (m, 1H), 7.87 (d, 2H, *J* = 8.4 Hz), 7.77 (d, 2H, *J* = 8.4 Hz), 7.67–7.64 (m, 1H), 7.57 (d, 2H, *J* = 8.4 Hz), 7.31–7.25 (m, 4H), 7.19–7.15 (m, 6H), 7.06 (t, 2H, *J* = 7.2 Hz), <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ (ppm) 168.1, 154.7, 151.2, 147.9, 147.6, 142.5, 135.5, 133.7, 133.7, 130.6, 129.4, 129.0, 127.9, 127.7, 127.1, 126.8, 124.7, 123.6, 123.2. HRMS (ESI/ASAP), *m/z* calculated for C<sub>32</sub>H<sub>24</sub>N<sub>3</sub> [M + H]<sup>+</sup> 450.1965, found 450.1965.

#### 4.1.5 | 4-[4-(9-ethyl-9H-carbazol-3-yl)-phenyl]quinazoline (**2c**)

Purified by column chromatography on silica gel (petroleum ether: EtOAc = 3:1). Cream solid (51%, 203 mg),

mp: 195–197°C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ (ppm) 9.42 (s, 1H), 8.43 (d, *J* = 1.8 Hz, 1H), 8.28 (d, *J* = 8.4 Hz, 1H), 8.20–8.13 (m, 2H), 7.97–7.91 (m, 5H), 7.82 (dd, *J*<sub>1</sub> = 8.4 Hz, *J*<sub>2</sub> = 1.8 Hz, 1H), 7.68–7.63 (m, 1H), 7.54–7.44 (m, 3H), 7.30–7.28 (m, 1H), 4.43 (q, *J* = 7.2 Hz, 2H), 1.49 (t, *J* = 7.2 Hz, 3H), <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ (ppm) 168.2, 154.8, 151.2, 144.0, 140.5, 139.8, 135.1, 133.6, 131.2, 130.6, 129.0, 127.7, 127.4, 127.2, 126.0, 125.2, 123.6, 123.3, 123.1, 120.5, 119.1, 108.9, 108.7, 37.7, 13.8. HRMS (ESI/ASAP), *m/z* calculated for C<sub>28</sub>H<sub>22</sub>N<sub>3</sub> [M + H]<sup>+</sup> 400.1808, found 400.1807.

#### 4.1.6 | 4-(4'-9H-carbazol-9-yl-[1,1'-biphenyl]-4-yl)quinazoline (**2d**)

Purified by column chromatography on silica gel (petroleum ether: EtOAc = 3:1). Cream solid (36%, 161 mg), mp: 187–189°C. <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ (ppm) 9.43 (s, 1H), 8.26 (d, *J* = 8.4 Hz, 1H), 8.19–8.15 (m, 3H), 7.99–7.90 (m, 7H), 7.73–7.65 (m, 3H), 7.53–7.42 (m, 4H), 7.66–7.63 (m, 3H), 7.35–7.30 (m, 2H), <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ (ppm) 167.9, 154.7, 151.3, 142.0, 140.8, 139.3, 137.6, 136.4, 133.8, 130.7, 129.1, 128.7, 127.8, 127.5, 127.4, 127.0, 126.0, 123.5, 123.2, 120.4, 120.1, 109.8. HRMS (ESI/ASAP), *m/z* calculated for C<sub>32</sub>H<sub>22</sub>N<sub>3</sub> [M + H]<sup>+</sup> 448.1808, found 448.1808.

#### ACKNOWLEDGMENTS

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#### DATA AVAILABILITY STATEMENT

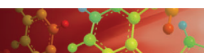
The data that supports the findings of this study are available in the supplementary material of this article.

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