Electronic Supporting Information for

Panchromatic Luminescence from Julolidine Dyes Exhibiting Excited

State Intramolecular Proton Transfer

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EXPERIMENTAL DETAILS

General Procedures and Materials. All reactions, except those indicated, were performed under a dry atmosphere of argon. [('Bu₃tpy)PtCl]BF₄,¹ [(ppy)₂Ir-(μ -Cl)]₂,² [Pd(PPh₃)₂Cl₂]³ Juloidine⁴ were prepared according to literature. The (trimethylsilyl)acetylene, copper iodide, 2-phenylpyridine (ppy) and 4-iodoaniline were purchased from Sigma Aldrich and used as purchased. THF was distilled from sodium and benzophenone under un argon atmosphere. 1,4-dichloroethane was distilled from P₂O₅ under an argon atmosphere. ¹H NMR (300, 400 MHz) and ¹³C NMR (75, 100 MHz) were recorded at room temperature in CDCl₃ in a Bruker Advance spectrometer. Chemical shifts for ¹H and ¹³C NMR were referenced to residual signals from CDCl₃. The 128 MHz ¹¹B NMR spectra were recorded at room temperature with B in borosilicate glass as internal references. Chromatographic purifications were performed using 40-63 μ m silica gel or aluminium oxide 90 standardized. The Infrared spectra were recorded on a Perkin-ATR Elmer Spectrum One.

Photophysics. Absorption spectra of dilute solutions ($c = 10^{-5}$ M) of different solvents (acetonitrile, ACN; methanol, MeOH; dichloromethane, DCM; toluene, TOL) were obtained with a Perkin-Elmer Lambda 950 UV/Vis/NIR spectrophotometer. All compounds were stable in solution for at least 24 hours (see Figure SI19 and SI20).

Steady-state photoluminescence spectra were measured in air-equilibrated solutions at room temperature, using an Edinburgh FLS920 fluorometer, equipped with Peltier-cooled R928 (200–850 nm) and supercooled R5509-72 (700–1800 nm) Hamamatsu phototubes, with different excitation wavelengths for the different compounds. Corrected spectra were employed throughout this work by applying to the raw data a correction curve for the wavelength-dependent phototubes response provided by the manufacturer. Photoluminescence quantum yields (ϕ) at room temperature (rt) were evaluated by comparing wavelength integrated intensities (*I*) of the corrected emission spectra with reference to [Ru(bpy)₃]Cl₂ ($\phi_r = 0.028$ in air-equilibrated water)⁵ and Quinine Sulphate ($\phi_r = 0.53$ in air-equilibrated 0.1 N H₂SO₄)⁵ standards, by using the following equation:

$$\phi = \frac{\left(I \cdot n^2 / A\right)}{\left(I_r \cdot n_r^2 / A_r\right)} \cdot \phi_r$$

where *A* and A_r are the absorbance values at the employed excitation wavelength, and *n* and *n_r* are the refractive indexes of the solvents for the investigated and the reference compound, respectively.⁵

Luminescence measurements of 1:1 CH₃OH:CH₂Cl₂ glass at 77 K were conducted by employing quartz capillary tubes immersed in liquid nitrogen, and hosted within homemade quartz cold finger dewar. The concentration was adjusted to obtain absorbance values $A \le 0.1$ at the excitation wavelengths for rt measurements and $0.1 \le A \le 0.2$ for low temperature measurements. Band maxima and relative luminescence intensities are obtained with uncertainties of 2 nm and 20 %, respectively.

Luminescence lifetimes were obtained using an IBH 5000F single-photon counting apparatus equipped with a TBX Picosecond Photon Detection Module and NanoLED pulsed excitation sources ($\lambda_{exc} = 373$ nm) or picosecond pulsed laser source ($\lambda_{exc} = 407$ nm). Phosphorescence lifetimes at 77 K were obtained using SpectraLED ($\lambda_{exc} = 370$ nm). Analysis of the luminescence decay profiles against time was accomplished using the Decay Analysis Software DAS6 provided by the manufacturer. The lifetime values were obtained with an estimated uncertainty of 10 %.

Absorption and emission spectral titrations were performed by gradual addition of a standard solution of trifluoroacetic acid to the compounds L_1 and L_3 in ACN (c = 5.9×10^{-6} M and 6.3×10^{-6} M, respectively), up to 1000 equivalents. Emission spectra were recorded upon excitation at the wavelength of the isosbestic point, observed in the corresponding absorption analysis ($\lambda_{exc} = 420$ and 425 nm for L_1 and L_3 , respectively).



Compound L₁: 9-formyl-1,1,7,7-tetramethyl-2,3,6,7-tetrahydro-*1H*,*5H*-pyrido [3,2,1-ij] quinoline (300 mg, 1.10 mmol) and 4-iodoaniline (360 mg, 1.64 mmol) were placed in a Schlenk tube and solubilized in anhydrous ethanol (15 mL). Then a crystal of p-TsOH was added under an argon flow. The resulting solution was stirred at 95 °C over night. After evaporation of the solvent the crude was treated with CH₂Cl₂ and washed several times with water. The organic layer was dried over anhydrous cotton wool, concentrated under vacuum and purified by column chromatography (SiO₂, CH₂Cl₂/petroleum ether: 5/5, v/v). The product was obtained as a yellow powder (300 mg, 75 %).¹H NMR (CDCl₃, 300 MHz) δ : 1.27 (s, 6H), 1.49 (s, 6H), 1.71-1.79 (m, 4H), 3.18 (t, ³*J* = 5.6 Hz, 2H), 3.27 (t, ³*J* = 5.9 Hz, 2H), 6.96 (s, 1H), 6.98 (d, ³*J* = 8.7 Hz, 2H), 7.65 (d, ³*J* = 8.6 Hz, 2H), 8.34 (s, 1H), 13.87 (s, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ : 28.4, 30.9, 31.8, 32.2, 36.3, 39.9, 47.1, 47.5, 89.0, 109.1, 114.6, 122.7, 123.0, 128.4, 138.2, 146.7, 149.0, 160.6, 161.6; IR-ATR (cm⁻¹) v: 488, 818, 944, 1029, 1216, 1309, 1514, 1576, 1619, 2854, 2926; MS-EI neat matter: *m/z* (intensity, %) calc. for [M]: 474.1 found: 474.0 (100); Anal. calc. for C₂₃H₂₇IN₂O: C, 58.23; H, 5.74; N, 5.91. Found: C, 57.98; H, 5.39; N, 5.59.



Compound L₂: To the solution of L₁ (300 mg, 0.63 mmol), [Pd(PPh₃)₂Cl₂] (44 mg, 0.06 mmol) and CuI (12 mg, 0.06 mmol) in THF (10 mL), (Trimethylsilyl)acetylene (0.27 mL, 1.90 mmol) and DIPA (2 mL) were added. The reaction media was stirred at room temperature over night. After evaporation of the solvents, the crude was solubilized in dichloromethane and washed with water. After drying the organic phase under hydrophilic cotton and concentration under vacuum the crude was purified by flash chromatography (SiO₂) eluting with petroleum ether/CH₂Cl₂, 7/3, v/v). The product was obtained as a brown powder quantitatively. ¹H NMR (CDCl₃, 400 MHz) δ : 0.26 (s, 9H), 1.27, (s, 6H), 1.50 (s, 6H), 1.72-1.79 (m, 4H), 3.19 (t, *J* = 5.5 Hz, 2H), 3.27 (t, *J* = 5.5 Hz, 2H), 6.97 (s, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.46 (d, *J* = 8.0 Hz, 2H), 8.38 (s, 1H, -CHN), 13.94 (s, 1H, -OH); ¹³C NMR (CDCl₃, 100 MHz) δ : 0.0 [-Si(CH₃)₃], 28.3, 30.8, 31.7, 32.2, 36.3, 39.8, 47.0, 47.4, 94.1, 105.3, 109.2, 120.7, 122.7, 128.4, 132.9, 146.7, 149.1, 160.8, 161.3; 444.3; EI neat matter: *m/z* (intensity, %) calc. for [M]: 444.7 found: 444.1 (100); 372.1 (100); Anal. calc. for C₂₈H₃₆N₂OSi: C, 75.63; H, 8.16; N, 6.30. Found: C, 75.43; H, 7.84; N, 6.01.



Compound L₃: The solution of L₂ (172 mg, 0.39 mmol) and KF (2.25 g, 38.67 mmol) in a mixture of THF (20 mL), MeOH (2 mL) and water (0.5 mL) was refluxed during 12 hours. After the reaction media has cold down to room temperature, the solvents were evaporated and the crude was solubilized with dichloromethane, neutralized with aqueous HCl (2M) and washed several times with water. The organic phase was dried under hydrophilic cotton and evaporated under vacuum. The crude was purified by silica flash chromatography, eluting with petroleum ether/CH₂Cl₂: 6/4 to 5/5, v/v) to give the L₃ as a yellow powder (110 mg, 76 %). ¹H NMR (CDCl₃, 400 MHz) &: 1.27 (s, 6H), 1.50 (s, 6H), 3.09 (s, 1H), 3.19 (t, *J* = 5.8 Hz, 4H), 3.27 (t, *J* = 5.8 Hz, 4H), 6.97 (s, 1H), 7.18 (d, *J* = 4.2 Hz, 2H), 7.49 (d, *J* = 4.2 Hz, 2H), 8.38 (s, 1H), 13.95 (s, 1H, -OH); ¹³C NMR (CDCl₃, 100 MHz) &: 28.4, 30.8, 31.8, 32.2, 36.3, 39.9, 47.0, 47.5, 83.8, 109.2, 114.6, 118.5, 120.9, 122.7, 128.5, 133.2, 146.8, 149.6, 160.8, 161.6; DEPT (CDCl₃, 100 MHz) CH₃, CH positive mode δ : 28.4, 30.8, 120.8, 128.5, 133.2 CH₂ negative mode δ : 36.3, 39.9, 47.0, 47.5; IR-ATR (cm⁻¹) v: 781, 1156, 1193, 1236, 1305, 1501, 1585, 1609, 1739, 2096 (-C≡C-, vw), 2923, 3239 (Csp-H, m); EI neat matter: *m/z* (intensity, %) calc. for [M]: 372.2 found: 372.1 (100); Anal. calc. for C₂₅H₂₈N₂O: C, 80.61; H, 7.58; N, 7.52. Found: C, 80.24; H, 7.21; N, 7.04.



Compound BL₁: BF₃Et₂O (0.28 mL, 1.90 mmol) was added dropwise to a solution of L₁ (150 mg, 0.32 mmol) in 1,4-dichloroethane (6 mL) under an argon flow. After this mixture had been stirred for 20-30 min at room temperature, DIPEA (0.16 mL, 0.966 mmol) was added and the reaction mixture was stirred for 3 hours at room temperature. After evaporation of the solvent, the crude was purified by silica column chromatography eluting with CH₂Cl₂ and recristallized from a CH₂Cl₂/ethanol mixture giving the compound as yellow solids (155 mg, 89 %).¹H NMR (CDCl₃, 300 MHz) δ : 1.25 (s, 6H), 1.52 (s, 6H), 1.71-1.78 (m, 4H), 3.31 (t, ³*J* = 5.7 Hz, 2H), 3.41 (t, ³*J* = 6.0 Hz, 2H), 6.97 (s, 1H), 7.24 (d, ³*J* = 8.6 Hz, 2H), 7.72 (d, ³*J* = 8.6 Hz, 2H), 7.88 (s, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ : 28.1, 29.8, 31.7, 32.0, 35.2, 38.9, 47.5, 47.9, 91.8, 107.4, 114.8, 124.9, 125.7, 127.5, 138.3, 143.2, 151.6, 157.2, 158.4; DEPT (CDCl₃, 100 MHz) CH₃, CH positive mode δ : 28.1, 29.8, 124.9, 127.5, 138.3, 157.2, negative mode (CH₂) δ : 35.2, 38.9, 47.5, 47.9; ¹¹B NMR (CD₃Cl, 128 MHz) δ : 0.91 (t, *J*_{B-F} = 17.4 Hz); IR-ATR (cm⁻¹) v: 488 (Csp²-I), 780, 818, 944, 1029, 1216, 1309 vs, 1514 vs, 1576, 1620, 2924; MS-EI neat matter: *m/z* (intensity, %) calc. for [M]: 522.1 found: 522.1 (80), 473.0 (100) [M-BF₂]; Anal. calc. for C₂₃H₂₆BF₂IN₂O: C, 52.90; H, 5.02; N, 5.36. Found: C, 51.60; H, 5.39; N, 5.04.



Compound BL₃: BF₃Et₂O (0.14 mL, 0.966 mmol) was added dropwise to a solution of L3 (120 mg, 0.322 mmol) in 1,4-dichloroethane (4 mL) under an argon flow. After this mixture has been stirred for 20-30 min at room temperature, DIPEA (0.16 mL, 0.966 mmol) was added and the reaction mixture was stirred for additional 40 min at room temperature. After evaporation of the solvent, the crude was purified by column chromatography (SiO₂, CH₂Cl₂/petroleum ether: 8/2, v/v) and recristallized from a CH₂Cl₂/ethanol mixture giving the compound as orange solids (127 mg, 94 %). ¹H NMR (CDCl₃, 400 MHz) δ : 1.26 (s, 6H), 1.52 (s, 6H), 1.71-1.78 (m, 4H), 3.12 (s, 1H, -C*H* acetylene), 3.29-3.32 (m, 2H), 3.41 (t, ³*J* = 6.1 Hz, 2H), 6.99 (s, 1H), 7.49 (AB system, ³*J* = 32.3 Hz, ⁴*J* = 8.5 Hz, 4H), 7.92 (s, 1H); ¹³C NMR (CDCl₃, 100 MHz) δ : 28.1, 29.8, 31.7, 32.0, 35.2, 38.8, 47.5, 47.9, 83.1, 107.4, 114.8, 12.7, 122.9, 125.7, 127.5, 133.1, 143.6, 151.7, 157.2, 158.5; ¹¹B NMR (CD₃Cl, 128 MHz) δ : 0.95 (t, *J*_{B-F} = 16.9 Hz); IR-ATR (cm⁻¹) v: 507, 544, 781, 838, 979, 1021, 1156, 1192, 1236, 1305; 1454, 1500, 1585, 1609, 1739, 2095 (-C=C-, vw), 2849, 2923, 2955 (-C-OH, w), 3239 (Csp-H, m); MS (EI neat matter): *m/z* (intensity, %) calc. for [M]: 420.2 found: 420.1 (100); 371.1 (80), [M-F]; Anal. calc. for C₂₅H₂₇BF₂N₂O: C, 71.44; H, 6.47; N, 6.67. Found: C, 71.22; H, 6.27; N, 6.44.



Figure SI1. ¹H NMR spectrum of compound L_1 in CDCl₃



Figure SI2. ¹³C NMR spectrum of compound L₁ in CDCl₃



Figure SI3. ¹H NMR spectrum of compound L_2 in CDCl₃



Figure SI4. ¹³C NMR spectrum of compound L₂ in CDCl₃



Figure SI5. ¹H NMR spectrum of compound L₃ in CDCl₃



Figure SI6. ¹³C NMR spectrum of compound L₃ in CDCl₃



Figure SI7. ¹H NMR spectrum of compound BL₁ in CDCl₃



Figure SI8. ¹³C NMR spectrum of compound BL₁ in CDCl₃



Figure SI9. ¹H NMR spectrum of compound BL₃ in CDCl₃



Figure SI10. ¹³C NMR spectrum of compound BL₃ in CDCl₃



Figure SI11. Absorption spectra of L_n and BL_n (n = 1, 3) in CH₃CN (top) and CH₃OH (bottom)



Figure SI12. Normalized absorption of L_1 (top) and L_3 (bottom) in different solvents at rt



Figure SI13. Normalized absorption of BL₁ (top) and BL₃ (bottom) in different solvents at rt



Figure SI14. Emission spectra in different solvents at rt of L_1 and BL_1 (top), L_3 and BL_3 (bottom). Spectra are scaled for the quantum yield



Figure SI15. Absorption (top) and emission (bottom) spectra of L_1 in CH₃CN solution ($c = 5.9 \times 10^{-6}$ M) at rt upon addition of trifluoroacetic acid (from 0 eq, blue line, to 1000 eq, red line); $\lambda_{exc} = 420$ nm, isosbestic point



Figure SI16. Absorption (top) and emission (bottom) spectra of L_3 in CH₃CN solution ($c = 6.3 \times 10^{-6}$ M) at rt upon addition of trifluoroacetic acid (from 0 eq, blue line, to 1000 eq, red line); $\lambda_{exc} = 425$ nm, isosbestic point



Figure SI17. Normalized fluorescence (full line) and phosphorescence (dotted line) spectra of L_1 and BL_1 (top), L_3 and BL_3 (bottom) in 1:1 CH₃OH:CH₂Cl₂ glass at 77 K



Figure SI18. Normalized irradiance spectra of L_1 and L_3 in 1:1 CH₃OH:CH₂Cl₂ glass at 77 K (top) and as pure powder film at rt (bottom)



Figure SI19. Absorption spectra of L_1 (top) and L_3 (bottom) in CH₃CN at rt



Figure SI20. Absorption spectra of BL_1 (top) and BL_3 (bottom) in CH₃CN at rt

	$\lambda_{ m max}$, nm ($\varepsilon_{ m max}$ × 10 ⁻³ , M ⁻¹ cm ⁻¹) ^{<i>a</i>}	$\lambda_{ m max}$, nm ($arepsilon_{ m max} imes 10^{-3}$, M $^{-1}$ cm $^{-1}$) b
L ₁	216 (27.3), 279 (10.1), 400 (49.4)	219 (29.5), 274 (9.9), 399 (46.2)
L ₃	221 (22.9), 273 (9.7), 407 (40.3)	219 (25.8), 271 (10.1), 406 (37.1)
BL ₁	221 (23.5), 277 (8.4), 421 (64.0)	221 (25.0), 276 (8.5), 419 (62.0)
BL ₃	221 (25.1), 275 (12.8), 426 (64.5)	220 (30.1), 273 (13.2), 423 (62.6)

Table SI1. Absorption features of L_n and BL_n (n = 1, 3)

^{*a*} In CH₃CN solution at rt. ^{*b*} In CH₃OH solution at rt.

		TOL			DCM			МеОН	
	$\lambda_{\rm max}$, nm	φ	τ, ns	$\lambda_{\rm max}$, nm	φ	τ, ns	$\lambda_{\rm max}$, nm	φ	τ, ns
L ₁		0.7×10 ⁻³			0.5×10 ⁻³			0.3×10 ⁻³	
E form	440		< 0.02	468		< 0.02	462		0.03
K form	588		0.12	596		0.13	586		0.07
L_3		0.9×10 ⁻³			0.7×10 ⁻³			0.4×10 ⁻³	
E form	464		< 0.02	478		< 0.02	466		< 0.02
K form	590		0.13	602		0.14	592		0.08
BL ₁	467	0.52	1.10	473	0.69	1.67	471	0.69	1.80
BL ₃	471	0.48	1.04	477	0.71	1.65	475	0.72	1.76

Table SI2. Luminescence properties of L_n and BL_n (n = 1, 3) in different solvents at rt

For quantum yield determination in air-equilibrated solutions, $\lambda_{exc} = 400$ nm. For lifetime measurements, $\lambda_{exc} = 407$ nm, for L₁ and L₃, $\lambda_{exc} = 373$ nm for BL₁ and BL₃.

	λ_{\max} , nm b	<i>τ</i> , ns ^{<i>c</i>}
L ₁	460 sh, 540 sh, 590	
E form (fluo)		1.1
E form (phos)		11.6×10 ⁶
K form (fluo)		4.6
L_3	464, 500, 540, 590	
E form (phos)		1.3
E form (phos)		54.6×10 ⁶
K form (fluo)		6.2
BL ₁		
fluo	458	1.87
phos	584	17.1×10 ⁶
BL ₃		
fluo	463	1.84
phos	572	38.1×10 ⁶

Table SI3. Luminescence properties of L_n and BL_n (n = 1, 3) at 77 K^{*a*}

^{*a*} In 1:1 CH₂Cl₂:CH₃OH solution. ^{*b*} $\lambda_{\text{exc}} = 420$ nm. ^{*c*} $\lambda_{\text{exc}} = 373$ nm and 370 nm for fluorescence and

phosphorescence lifetime measurements, respectively.

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