Data Article

Data on the characterization of (N-alkylsalicylaldiminato)bis(2-phenylpyridinato)iridium(III)

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ABSTRACT

Herein we report the synthesis, characterization data and photophysical properties of iridium(III) complexes having N-alkylated salicylaldimine and 2-phenylpyridine ligands. The structures of novel iridium complexes were assigned by $^1$H and $^{13}$C NMR, $^1$H–$^1$H COSY, NOESY, HMQC, HMBC, HRMS, IR and XRD analysis. For further information, we obtained photophysical properties in solution and crystalline states.

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1. Data

In this article we share the synthesis and characterization of iridium complexes 1a–1d that exhibit phosphorescence in the solution and crystalline states. Figs. 1 and 2 demonstrate $^1$H and $^{13}$C NMR spectra of 1a–1d in CDCl$_3$. Assignment of $^1$H NMR signals was made by $^1$H–$^1$H COSY and NOESY experiments as shown in Figs. 3 and 4. Assignment of $^{13}$C NMR was made by HMQC and HMBC.
experiments as shown in Fig. 5 and Fig. 6. Figs. 7 and 8 show ORTEP drawings and packing of 1a–1c determined by XRD analysis. Table 1 shows crystallographic data for 1a–1c. Figs. 9 and 10 show UV–vis spectra for 1a–1d in 2-MeTHF and solid state. Figs. 11 and 12 show emission spectra in 2-MeTHF and crystalline state. Table 2 shows photophysical data of 1a–1c in 2-MeTHF and crystalline state.

2. Experimental design, materials, and methods

Melting points were measured in a glass capillary using a Yanagimoto melting point apparatus. \( ^{1} \text{H} \) NMR, \( ^{13} \text{C} \) NMR, \( ^{1} \text{H} \rightarrow ^{1} \text{H} \) COSY, NOESY, HMQC and HMBC spectra of samples in a deuterated chloroform were recorded on a Varian Unity—Inova 500 spectrometer. \( ^{1} \text{H} \) NMR and \( ^{13} \text{C} \) NMR spectra were referenced to a peak of an internal TMS (0.0 ppm for \( ^{1} \text{H} \)) and a deuterated chloroform (77.0 ppm for \( ^{13} \text{C} \)), respectively. IR spectra was recorded on a Bruker Equinox 55 spectrometer in KBr disk at room temperature. HRMS was obtained by using a Bruker microOTOF II spectrometer. UV–vis and emission spectra in 2-MeTHF were recorded on a Jasco V650 and a Jasco FP-6500 spectrometer.
Fig. 1. $^1$H NMR spectra (500 MHz) of complexes (a, e) 1a, (b, f) 1b, (c, g) 1c and (d, h) 1d. (a–d) Aromatic and (e–h) aliphatic regions.
Fig. 2. $^{13}$C NMR spectra (125 MHz) of complexes (a, e) 1a, (b, f) 1b, (c, g) 1c and (d, h) 1d. (a–d) Aromatic and (e–h) aliphatic regions.
Fig. 3. $^1$H–$^1$H COSY spectra (500 MHz) of complexes (a, b) 1a, (c, d) 1b, (e, f) 1c and (g, h) 1d in CDCl$_3$ (298 K, number of $t_1$ increments = 1024, number of $t_2$ increments = 1024, number of scans = 16). (a, c, e, g) Aromatic and (b, d, f, h) aliphatic regions.
Fig. 3. (Continued)
Fig. 4. NOESY spectra (500 MHz) of complexes (a) 1a, (b) 1b, (c) 1c and (d) 1d in CDCl₃ (298 K, mixing time = 1.00 s, number of t₁ increments = 1024, number of t₂ increments = 1024, number of scans = 64).
Fig. 5. HMOC spectra (500 MHz) of complexes (a) 1a, (b) 1b, (c) 1c and (d) 1d in CDCl₃ (298 K, number of t₁ increments = 1024, number of t₂ increments = 1024).
Fig. 6. HMBC spectra (500 MHz) of complexes (a) 1a, (b) 1b, (c) 1c and (d) 1d in CDCl$_3$ (298 K, number of $t_1$ increments = 1024, number of $t_2$ increments = 1024).
X-ray diffraction analysis

Fig. 7. ORTEP representations of (a) Δ-1a, (b) Δ-1b and (c) Δ-1c as their racemic crystals. Thermal ellipsoids are shown at the 50% probability level.

Fig. 8. Packing in (a) 1a, (b) 1b and (c) 1c crystals.

spectrometer respectively. Emission lifetime in 2-MeTHF was measured on an Optical Building Blocks Corp. EasyLife-X.

Crystals of 1a–1c suitable for X-ray diffraction studies were prepared by recrystallization from hexane/ethyl acetate mixture, and analyzed using a Rigaku XtaLAB P200 diffractometer with Mo-Kα radiation. The structures of 1a–1c were solved by direct methods and refined using the full-matrix least-squares method. In subsequent refinements, the function $\Sigma_0(F_o^2 - F_c^2)^2$ was minimized, where $F_o$ and $F_c$ are the observed and calculated structure factor amplitudes, respectively. The positions of non-hydrogen atoms were determined from difference Fourier electron-density maps and refined anisotropically. All calculations were performed with the Crystal Structure crystallographic software package, and illustrations were drawn using ORTEP [1].
### Table 1: Crystallographic data for 1a–1c.

|   | 1a                                    | 1b                                    | 1c                                    |
|---|---------------------------------------|---------------------------------------|---------------------------------------|
| formula | C₃₄H₃₂N₃OIr                        | C₃₇H₃₈NO₃Ir                          | C₄₃H₅₀N₃OIr                          |
| Mᵣ   | 690.87                               | 732.95                                | 817.11                                |
| T/K  | 113                                  | 113                                   | 113                                   |
| crystal color, habit | yellow, chip | yellow, chip                             | yellow, chip                         |
| crystal size/mm | 1.00 × 0.50 × 0.1 | 0.20 × 0.20 × 0.05                   | 0.20 × 0.05 × 0.01                   |
| crystal system | monoclinic                        | monoclinic                            | triclinic                             |
| space group | P₂₁/c (#14)                        | P₂₁/n (#14)                           | P-1 (#2)                              |
| a/Å   | 16.290(2)                            | 16.334 (2)                            | 9.6352 (16)                           |
| b/Å   | 7.98165(11)                          | 9.8695(13)                            | 15.051(2)                             |
| c/Å   | 17.405(2)                            | 19.189(3)                             | 26.536(4)                             |
| α/°   | 90                                   | 90                                    | 75.107(5)                             |
| β/°   | 97.790(3)                            | 94.793(4)                             | 89.320(7)                             |
| γ/°   | 90                                   | 90                                    | 87.117(7)                             |
| V/Å³  | 2757.6(6)                            | 3082.7(7)                             | 3714.3(10)                            |
| Z     | 4                                    | 4                                     | 4                                     |
| ρcalcd/g cm⁻³ | 1.664                               | 1.579                                | 1.461                                 |
| μ (MoKα)/cm⁻¹ | 48.877                             | 43.772                               | 36.411                                |
| F(000) | 1368.00                             | 1464.00                              | 1656.00                               |
| 2θmax/° | 55.0                              | 55.0                                  | 55.0                                  |
| No. of reflns measd | 28631                             | 35714                                | 71214                                 |
| No. of obsd reflns | 6304                         | 7043                                  | 16964                                 |
| No. variables | 352                             | 379                                   | 865                                   |
| R₁ (I > 2σ(I))   | 0.0378                             | 0.0291                                | 0.0485                                |
| wR₂ (all reflns) | 0.912                             | 0.0696                                | 0.1166                                |
| Goodness of fit | 0.975                           | 1.008                                 | 0.9971                                |

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**Photophysical Data**

![Graph showing UV–vis spectra of complexes 1a–1d in 2-MeTHF (2.0 × 10⁻⁴ M) at 298 K.](image)

**Fig. 9.** UV–vis spectra of complexes 1a–1d in 2-MeTHF (2.0 × 10⁻⁴ M) at 298 K.

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a. $R_1 = \Sigma|\langle F_o \rangle - |F_c | | / \Sigma(\langle F_o \rangle$.

b. $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2 / \Sigma w(F_o^2)]^{1/2}$.
Fig. 10. Normalized diffuse reflectance UV–vis spectra of crystals 1a–1d at 298 K.

Fig. 11. Emission spectra of complexes 1a–1d in 2-MeTHF (2.0 × 10⁻⁴ M) at 77 K (λ<sub>ex</sub> = 415 nm).

Fig. 12. Normalized emission spectra of crystals 1a–1d at (a) 298 K and (b) 77 K (λ<sub>ex</sub> = 450 nm).
Table 2
Photophysical data for 1a–1d[a].

| Complex | State   | $\lambda_{abs}$ [nm] | $\lambda_{em}$ [nm] | $\phi$[d] | $\tau$ [fs] | $k_r \times 10^{-5}$ [s$^{-1}$] | $k_{nr} \times 10^{-5}$ [s$^{-1}$] |
|---------|---------|----------------------|---------------------|-----------|------------|-----------------------------|-----------------------------|
| 1a      | 2-MeTHF | 397                  | $463$ (561)         | 0.13      | 5.03       | 0.26 (2.0)                  |                            |
| 1b      | 2-MeTHF | 397                  | $463$ (527, 564)    | 0.13      | 4.98       | 0.26 (2.0)                  |                            |
| 1c      | 2-MeTHF | 397                  | $463$ (572)         | 0.15      | 5.09       | 0.29 (2.0)                  |                            |
| 1d      | 2-MeTHF | 397                  | $463$ (538, 568)    | 0.14      | 4.87       | 0.29 (2.1)                  |                            |
| 1a      | crystal | 399                  | 576 (550, 584)      | 0.02      | 0.06       | 0.61 (16)                   |                            |
| 1b      | crystal | 399                  | 573 (541, 578)      | 0.03      | 0.12       | 0.47 (21)                   |                            |
| 1c      | crystal | 399                  | 579 (553, 580)      | 0.02      | 0.11       | 0.30 (33)                   |                            |
| 1d      | crystal | 399                  | 574 (541, 572)      | 0.03      | 0.10       | 0.51 (19)                   |                            |

[a] Data were obtained at 298 K and 77 K. Values in parentheses are those measured at 77 K.
[b] $2.0 \times 10^{-4}$ M.
[c] $\lambda_{ex} = 415$ nm.
[d] Determined by the absolute method using an integrating sphere.
[e] $\lambda_{em} = 415$ nm.
[f] Determined based on the quantum yield and lifetime.
[g] No data due to non-emission properties at 298 K.

3. Synthesis

Complexes 1a–1d were prepared by reaction of $\mu$-chlorobis(2-phenylpyridine)iridium dimer [2] with the corresponding salicylaldimine and Na$_2$CO$_3$ in boiling 2-ethoxyethanol.

3.1. Complex 1a ($n = 5$)

M.p. = 272–273 °C, IR (KBr): 3050, 2950, 2922, 2852, 1616, 1582, 1530, 1475, 1452, 1417, 1356, 1156, 910, 755 cm$^{-1}$; $^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 0.57–0.73 (m, 5 H, H$^{16,18}$), 0.83–0.97 (m, 3 H, H$^{15,17}$).
1.08–1.17 (m, 1 H, H15), 3.10 (ddd, J = 5.5, 10.5, 10.5 Hz, 1 H, H14), 3.33 (ddd, J = 5.5, 10.5, 10.5 Hz, 1 H, H14),
6.13 (dd, J = 11.7, 7.5 Hz, 1 H, H5a), 6.36 (dd, J = 11.7, 7.5 Hz, 2 H, H8b), 6.39 (ddd, J = 1.4, 6.9, 7.8 Hz, 1 H, H11b),
6.66 (dd, J = 1.4, 8.8 Hz, 1 H, H8b), 6.66 (ddd, J = 1.4, 7.5, 7.5 Hz, 1 H, H7), 6.72 (ddd, J = 1.4, 7.5, 7.5 Hz, 1 H, H7b),
6.80 (ddd, J = 11.7, 7.5, 7.8 Hz, 1 H, H6b), 6.82 (ddd, J = 11.7, 7.5, 7.8 Hz, 1 H, H5b), 6.96 (ddd, J = 1.3, 5.7, 7.3 Hz,
1 H, H2), 7.06 (dd, J = 1.8, 7.8 Hz, 1 H, H12), 7.10 (ddd, J = 1.2, 5.7, 7.3 Hz, 1 H, H2), 7.17 (ddd, J = 1.8, 6.9,
8.8 Hz, 1 H, H8b), 7.53 (dd, J = 1.4, 7.8 Hz, 1 H, H2), 7.57 (dd, J = 1.4, 7.8 Hz, 1 H, H2), 7.65 (ddd, J = 1.5, 7.3,
8.0 Hz, 1 H, H3b), 7.73 (ddd, J = 1.5, 7.3, 8.0 Hz, 1 H, H3b), 7.82 (ddd, J = 0.7, 1.2, 8.0 Hz, 1 H, H4b), 7.87 (ddd, J = 0.7,
1.2, 8.0 Hz, 1 H, H4b), 8.01 (s, 1 H, H13), 8.43 (ddd, J = 0.7, 1.5, 5.7 Hz, 1 H, H14), 8.86 (ddd, J = 0.7, 1.5,
5.7 Hz, 1H, H14); 13C NMR (125 MHz, CDCl3) δ 13.9 (C18), 22.1 (C17), 28.8 (C16), 30.5 (C15), 64.3 (C14), 112.9 (C11),
118.1 (C4), 118.8 (C3), 120.1 (C9), 121.1 (C6), 121.2 (C12a), 121.4 (C2), 121.6 (C5), 123.7 (C9), 124.0 (C8),
129.25 (C7 or 7'), 129.28 (C7 or 7'), 131.8 (C6), 133.38 (C10), 133.41 (C8), 134.7 (C12), 136.5 (C3),
136.7 (C3a), 144.49 (C5 or 5'), 144.50 (C5 or 5'), 148.77 (C1 or 1'), 148.83 (C1 or 1'), 150.2 (C8a), 153.2 (C8a),
161.6 (C13), 166.0 (C6a), 168.5 (C4a), 169.0 (C4a); HRMS (APCI): m/z calcd for [191IrC34H32N3O]: 689.2146; found:
689.2147 [M+].

3.2. Complex 1b (n = 8)

M.p. = 239–240 °C, IR (KBr): 3029, 2919, 2851, 1617, 1582, 1475, 1453, 1359, 1157, 1059, 1030, 758

cm−1; 1H NMR (500 MHz, CDCl3) δ 0.57–0.75 (m, 2 H, H16b), 0.78–0.93 (m, 6 H, H15a,17,21), 1.01 (ddd,
J = 7.5, 7.5, 7.5 Hz, 2 H, H18), 1.08–1.14 (m, 3 H, H15b,19), 1.22 (ddd, J = 7.5, 7.5, 7.5 Hz, 2 H, H20b),
3.09 (ddd, J = 5.8, 10.1, 10.1 Hz, 1 H, H14a), 3.33 (ddd, J = 5.8, 10.1, 10.1 Hz, 1 H, H14b), 6.13 (dd, J = 1.1, 7.5 Hz,
1 H, H14b), 6.36 (dd, J = 1.1, 7.5 Hz, 1 H, H14a), 6.36 (ddd, J = 1.4, 6.9, 7.8 Hz, 1 H, H11b), 6.66 (ddd, J = 1.4,
8.8 Hz, 1 H, H9b), 6.66 (ddd, J = 1.4, 7.5, 7.5 Hz, 1 H, H9a), 6.72 (dd, J = 1.4, 7.5, 7.5 Hz, 1 H, H9b), 6.80 (dd, J = 1.1,
7.5, 7.8 Hz, 1 H, H9a), 6.82 (dd, J = 1.1, 7.5, 7.8 Hz, 1 H, H9b), 6.96 (ddd, J = 1.3, 5.7, 7.3 Hz, 1 H, H9b), 7.06 (dd,
J = 1.8, 7.8 Hz, 1 H, H12), 7.10 (dd, J = 1.2, 5.7, 7.3 Hz, 1 H, H9b), 7.17 (dd, J = 1.8, 6.9, 8.8 Hz, 1 H, H10b),
7.53 (dd, J = 1.8, 7.8 Hz, 1 H, H9b), 7.56 (dd, J = 1.4, 7.8 Hz, 1 H, H15a,17,21), 7.65 (ddd, J = 1.5, 7.3, 8.0 Hz, 1 H, H3b),
7.73 (ddd, J = 1.5, 7.3, 8.0 Hz, 1 H, H3b), 7.82 (ddd, J = 0.7, 1.2, 8.0 Hz, 1 H, H4b), 7.87 (ddd, J = 0.7, 1.2, 8.0 Hz, 1 H, H4b),
8.01 (s, 1 H, H13), 8.43 (ddd, J = 0.7, 1.5, 5.7 Hz, 1 H, H14a), 8.86 (ddd, J = 0.7, 1.5, 5.7 Hz, 1 H, H14b); 13C NMR
(125 MHz, CDCl3) δ 14.1 (C21), 22.6 (C20), 26.7 (C16), 29.0 (C17 or 18), 29.1 (C17 or 18), 30.9 (C15), 31.7
(C19), 64.3 (C14), 112.8 (C11), 118.0 (C4 or 4'), 118.1 (C4 or 4'), 120.08 (C6), 121.14 (C12a), 121.17 (C12b), 121.4 (C4a),
121.6 (C2), 123.7 (C12), 123.9 (C5), 124.1 (C8 or 7), 129.2 (C7 or 7'), 129.3 (C7 or 7'), 131.8 (C8 or 7), 133.3 (C10)
133.5 (C8), 134.7 (C12), 136.4 (C3), 136.7 (C3), 144.46 (C5 or 5'), 144.49 (C5 or 5'), 148.75 (C1 or 1'), 148.81 (C1 or 1'),
150.3 (C8b), 153.2 (C8b), 161.6 (C13), 165.8 (C9a), 168.5 (C4a), 169.0 (C4a); HRMS (APCI): m/z calcd for
[191IrC34H32N3O]: 731.2615; found: 787.2611 [M+].
3.3. Complex 1c (n = 14)

M.p. = 187.5–188.5 °C, IR (KBr): 3041, 2922, 2851, 2356, 1617, 1475, 1454, 1059. 756, 734, 675 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 0.58–0.76 (m, 2 H, H¹⁶), 0.78–0.93 (m, 6 H, H¹⁵a,¹⁷,²⁷), 1.01 (ddd, J = 7.5, 7.5, 7.5, 7.5 Hz, 2 H, H¹⁸), 1.09–1.16 (m, 3 H, H¹⁵b,¹⁹), 1.16–1.33 (m, 14 H, H²⁰–²⁶), 3.10 (ddd, J = 5.8, 10.1, 10.1 Hz, 1 H, H¹⁴), 3.34 (ddd, J = 5.8, 10.1, 10.1 Hz, 1 H, H¹⁴), 6.14 (dd, J = 1.1, 7.5 Hz, 1 H, H⁸), 6.37 (dd, J = 1.1, 7.5 Hz, 1 H, H⁸), 6.38 (ddd, J = 1.4, 6.9, 7.8 Hz, 1 H, H¹¹), 6.67 (dd, J = 1.4, 8.8 Hz, 1 H, H⁶), 6.67 (ddd, J = 1.4, 7.5, 7.5 Hz, 1 H, H⁹), 6.73 (ddd, J = 1.4, 7.5, 7.5 Hz, 1 H, H⁹), 6.81 (ddd, J = 1.1, 7.5, 7.8 Hz, 1 H, H⁸), 6.81 (ddd, J = 1.1, 7.5, 7.8 Hz, 1 H, H⁸), 6.98 (ddd, J = 1.3, 5.7, 7.3 Hz, 1 H, H¹²), 7.07 (dd, J = 1.8, 7.8 Hz, 1 H, H¹²), 7.11 (ddd, J = 1.2, 5.7, 7.3 Hz, 1 H, H¹²), 7.18 (ddd, J = 1.8, 6.9, 8.8 Hz, 1 H, H¹⁰), 7.55 (dd, J = 1.4, 7.8 Hz, 1 H, H¹⁰), 7.57 (dd, J = 1.4, 7.8 Hz, 1 H, H¹⁰), 7.66 (ddd, J = 1.5, 7.3, 8.0 Hz, 1 H, H¹³), 7.74 (ddd, J = 1.5, 7.3, 8.0 Hz, 1 H, H¹³), 7.83 (ddd, J = 0.7, 1.2, 8.0 Hz, 1 H, H⁴), 7.88 (ddd, J = 0.7, 1.2, 8.0 Hz, 1 H, H⁴), 8.02 (s, 1 H, H¹²), 8.43 (ddd, J = 0.7, 1.5, 5.7 Hz, 1 H, H¹¹), 8.87 (ddd, J = 0.7, 1.5, 5.7 Hz, 1 H, H¹¹); ¹³C NMR (125 MHz, CDCl₃) δ 141 (C¹⁷), 22.7 (C²⁶), 26.7 (C¹⁹), 29.1 (C¹⁷), 29.35, 29.42, 29.5, 29.6, 29.66, 29.71, 30.9 (C¹⁵), 31.9 (C₂⁸), 64.3 (C¹⁴), 112.9 (C¹¹), 118.0 (C⁴), 118.8 (C⁴), 120.2 (C⁶), 121.12 (C¹²), 121.14 (C¹²), 121.4 (C²), 121.6 (C³), 123.7 (C⁵), 123.95 (C⁵), 123.99 (C⁵), 129.2 (C⁷ or ⁷), 129.3 (C⁷ or ⁷), 131.8 (C⁶), 133.39 (C¹⁰), 133.41 (C⁸), 134.7 (C¹²), 136.5 (C³), 136.6 (C³), 144.46 (C⁵ or ⁵), 144.50 (C⁵ or ⁵), 148.8 (C¹ or ¹), 148.9 (C¹ or ¹), 150.2 (C⁸a), 153.3 (C⁸a), 161.6 (C¹³), 165.7 (C⁸a), 168.5 (C⁸a), 169.0 (C⁸a); HRMS (APCI): m/z calcd for ¹⁹¹IrC₄₃H₅₀N₉O: 815.3554; found: 815.3545 [M⁺].

3.4. Complex 1d (n = 18)

M.p. = 176–177 °C, IR (KBr): 3048, 2923, 2851, 1617, 1583, 1532, 1475, 1453, 1416, 1357, 1332, 1157 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 0.58–0.75 (m, 2 H, H¹⁶), 0.79–0.93 (m, 6 H, H¹⁵a,¹⁷,²⁷), 1.01 (ddd, J = 7.5, 7.5, 7.5, 7.5 Hz, 2 H, H¹⁸), 1.08–1.15 (m, 3 H, H¹⁵b,¹⁹), 1.15–1.33 (m, 22 H, H²⁰–³⁰), 3.10 (ddd, J = 5.8,
10.1, 10.1 Hz, 1 H, $H^{14}$), 3.34 (ddd, $J = 5.8, 10.1, 10.1$ Hz, 1 H, $H^{14}$), 6.14 (ddd, $J = 1.1, 7.5$ Hz, 1 H, $H^{8}$), 6.37 (ddd, $J = 1.4, 7.5$ Hz, 1 H, $H^{10}$), 6.38 (ddd, $J = 1.4, 7.5$ Hz, 1 H, $H^{8}$), 6.67 (ddd, $J = 1.4, 8.8$ Hz, 1 H, $H^{9}$), 6.67 (ddd, $J = 1.4, 7.5$ Hz, 1 H, $H^{10}$), 6.81 (ddd, $J = 1.1, 7.5$ Hz, 1 H, $H^{8}$), 6.81 (ddd, $J = 1.1, 7.5$ Hz, 1 H, $H^{10}$), 6.98 (ddd, $J = 1.3, 5.7$ Hz, 1 H, $H^{9}$), 7.07 (dd, $J = 1.8, 7.8$ Hz, 1 H, $H^{12}$), 7.11 (dd, $J = 1.2, 5.7, 7.3$ Hz, 1 H, $H^{10}$), 7.18 (dd, $J = 1.8, 6.9, 8.8$ Hz, 1 H, $H^{10}$), 7.55 (dd, $J = 1.4, 7.8$ Hz, 1 H, $H^{12}$), 7.66 (dd, $J = 1.4, 7.8$ Hz, 1 H, $H^{12}$), 7.74 (dd, $J = 1.5, 7.3, 8.0$ Hz, 1 H, $H^{12}$), 8.02 (s, 1 H, $H^{13}$), 8.43 (ddd, $J = 0.7, 1.5, 5.7$ Hz, 1 H, $H^{1}$), 8.87 (ddd, $J = 0.7, 1.5, 5.7$ Hz, 1 H, $H^{1}$); $^{13}$C NMR (125 MHz, CDCl$_3$): δ 14.1 (C$_{31}$), 22.7 (C$_{30}$), 26.7 (C$_{16}$), 29.0 (C$_{17}$), 29.35, 29.42, 29.5, 29.61, 29.65, 29.67, 29.71, 30.9 (C$_{15}$), 31.9 (C$_{29}$), 64.3 (C$_{14}$), 112.8 (C$_{11}$), 118.0 (C$_{4}$), 118.8 (C$_{4}$), 120.2 (C$_{6}$), 121.1 (C$_{6}$), 121.2 (C$_{12a}$), 121.4 (C$_{2}$), 121.6 (C$_{2}$), 123.7 (C$_{5}$), 123.9 (C$_{5}$), 124.1 (C$_{6}$), 129.25 (C$_{7}$ or $^7$), 129.27 (C$_{7}$ or $^7$), 131.8 (C$_{8}$), 133.3 (C$_{10}$), 133.4 (C$_{8}$), 134.7 (C$_{12}$), 136.4 (C$_{3}$), 136.6 (C$_{3}$), 144.46 (C$_{5}$ or $^5$), 144.49 (C$_{5}$ or $^5$), 148.76 (C$_{1}$ or $^1$), 148.83 (C$_{1}$ or $^1$), 150.3 (C$_{8a}$), 153.2 (C$_{8a}$), 161.6 (C$_{13}$), 166.0 (C$_{3a}$), 168.5 (C$_{4a}$), 169.0 (C$_{4a}$); HRMS (APCI): m/z calcd for $^{191}$IrC$_{47}$H$_{58}$N$_{3}$O: 871.4180; found: 871.4174 [M$^+$]. Anal. Calcd for IrC$_{47}$H$_{58}$N$_{3}$O: C, 64.65; H, 6.70; N, 4.81. Found: C, 64.46, H, 6.61, N, 4.76.

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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