Crystal Structure of Iridium(III) Bis(2-(*p*-biphenyl)quinolyl-*N,C*^{2'})acetylacetonate, (bpq)₂Ir(acac)

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Red single crystals of an iridium(III) complex, [(bpq)₂Ir(acac)] (bpq = 2-(*p*-biphenyl)quinoline, acac = acetylacetonate), were grown from sublimation, and determined using a CCD diffractometer. The *R* value is *R*₁ = 0.0227, and *wR*₂ = 0.0505. The iridium complex shows an approximately octahedral coordination geometry and has a stable sterical configuration. It is expected to be a good candidate for OLEDs.

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In recent decades, the cyclometalated iridium complexes have been extensively investigated because of their long-lived excited states and high luminescent quantum yields applicable to photoreductants¹ and electrophosphorescent materials.² A significant research effort has focused on characterizing the structure of these iridium complexes in order to explore the effect of the structure on the photophysics, thermostability, and electrochemistry. Several examples of crystal structures for iridium complexes containing 2-phenylpyridine-based ligands have been reported,^{3,4} in contrast, there has been few reports on the structure of iridium complexes with 2-arylquinoline-based ligands.

In this work, iridium(III) bis(2-(*p*-biphenyl)quinolyl-*N,C*^{2'})acetylacetonate (bpq)₂Ir(acac) was obtained in two steps using literature methods.⁴ First, a cyclometalated Ir(III) μ -chlorobridged dimer was synthesized by reacting IrCl₃·*n*H₂O and 2-(*p*-biphenyl)quinoline. Then, the dimer was reacted with acetylacetonate in 2-ethoxyethanol. Pure (bpq)₂Ir(acac) was obtained by gradient sublimation. Single crystals of the complex were picked up from red sublimate. The chemical structure of (bpq)₂Ir(acac) is shown in Fig. 1.

The measurement conditions and crystal data are listed in Table 1. Intensity data for (bpq)₂Ir(acac) was collected at room

temperature (*T* = 296 K) on a BRUKER SMART APEX II CCD diffractometer with graphite-monochromated Mo-*K*_α radiation (λ = 0.71073 Å). Data were corrected for LP factors and absorption was applied by using the SADABS program. The structure was solved by direct methods and refined by the full-

Table 1 Crystal data and structure refinement for (bpq)₂Ir(acac)

CCDC No.: 271681
Empirical formula: C ₄₇ H ₃₅ IrN ₂ O ₂
Formula weight: 851.97
Crystal system: triclinic
Space group: <i>P</i> $\bar{1}$; <i>Z</i> = 2
<i>a</i> = 11.3856(10) Å
<i>b</i> = 12.9967(12) Å
<i>c</i> = 13.0495(12) Å
α = 80.131(10)°
β = 86.100(10)°
γ = 72.844(10)°
<i>V</i> = 1817.5(3) Å ³
<i>D</i> _x = 1.557 g/cm ³
<i>T</i> = 296 K
Crystal color: Red
<i>F</i> (0 0 0) = 848
Crystal size: 0.32 × 0.24 × 0.18 mm
Radiation: Mo- <i>K</i> _α
θ scan with θ from 1.87 to 25°
<i>R</i> ₁ = 0.0227 (on <i>F</i> ²)
<i>wR</i> ₂ = 0.0505 (on <i>F</i> ²)
(Δ / σ) _{max} = 0.04
($\Delta\rho$) _{max} = 1.057 eÅ ⁻³
($\Delta\rho$) _{max} = -0.747 eÅ ⁻³
No. of reflections measured = 9232
No. of reflections used = 6295
No. of parameters = 471
Goodness-of-fit on <i>F</i> ² = 1.032
Measurement: BRUKER SMART APEX II CCD diffractometer
Program system: SMART V 5.625 and SAINT V 6.23C
Structure determination: direct methods (SHELXS-97)
Refinement: full matrix least-squares (SHELXL-97)

Fig. 1 Chemical structure of (bpq)₂Ir(acac).

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Bond Lengths (Å)		Bond Angles (deg)	
Ir(1)-C(36)	1.972(3)	N(1)-Ir(1)-N(2)	176.49(8)
Ir(1)-C(15)	1.985(3)	Ir(1)-N(1)-C(1)	127.00(2)
Ir(1)-N(1)	2.061(2)	C(15)-Ir(1)-N(1)	80.29(11)
Ir(1)-N(2)	2.077(2)	Ir(1)-N(1)-C(9)	113.50(19)
Ir(1)-O(1)	2.1555(19)	Ir(1)-C(15)-C(10)	113.20(2)
Ir(1)-O(2)	2.160(2)	C(1)-N(1)-C(9)	119.30(3)
Torsion Angles (deg)			
C(14)-C(13)-C(16)-C(21)		47.7(5)	
N(1)-C(9)-C(10)-C(15)		-0.6(4)	
C(9)-N(1)-C(1)-C(2)		170.3(3)	
C(8)-C(9)-C(10)-C(11)		0.4(5)	
C(35)-C(34)-C(37)-C(42)		-37.6(4)	
N(2)-C(30)-C(31)-C(36)		-6.0(4)	
C(30)-N(2)-C(22)-C(23)		171.2(3)	

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