Supporting Information to:

Quinoline-annulated Porphyrins

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Materials and Instruments.

All solvents and reagents (Aldrich, Acros, CIL) were used as received. Analytical (aluminum backed, silica gel 60, 250 µm thickness) and preparative (20 × 20 cm, glass backed, silica gel 60, 500 or 1000 µm thickness) TLC plates, and the flash column silica gel (standard grade, 60 Å, 32-63 µm) used were provided by Sorbent Technologies, Atlanta, GA. \( ^1 \)H and \( ^{13} \)C NMR spectra were recorded on a Bruker DRX400 or a Varian 500 MHz instrument. High and low resolution mass spectra were provided by the Mass Spectrometry Facilities at the Department of Chemistry, University of Connecticut. UV-vis spectra were recorded on a Cary 50, and the fluorescence spectra on a Cary Eclipse spectrophotometer, both Varian Inc, IR spectra on a JASCO FT-IR-410 using a diffuse reflectance unit.

*meso*-Tetraphenylporphyrin-2,3-dione (4) was prepared by DDQ oxidation of the corresponding *meso*-tetraphenylporphyrin-2,3-dihydroxychlorin\(^1\) according to the literature.\(^2\)

**Porphyrin-2,3-dione monoxime (5) and porphyrin-2,3-dione dioxime (6)**

*meso*-Tetraphenyl-2,3-dioxochlorin 3 (120 mg, 1.85 × 10\(^{-4}\) mmol) was dissolved in pyridine (25 mL) in a round-bottom flask equipped with a magnetic stir bar and N\(_2\) gas inlet. H\(_2\)N-OH-HCl (1.2 g, ~100 equiv) was added and the mixture was stirred for 24 h at rt. When the starting material was consumed (reaction control by TLC), the reaction mixture was evaporated to dryness by rotary evaporation, taken up in CH\(_2\)Cl\(_2\) and filter through a glass frit (M). The volume of the filtrate was reduced and the mixture separated on a column chromatography

(CH$_2$Cl$_2$-petroleum ether 3:1), allowing the isolation of 5 in 91% (111 mg) and 6 in 5.0% (6.3 mg).

Spectroscopic data for 5: R$_f$ (silica-CH$_2$Cl$_2$) 0.75; $^1$H NMR (400 MHz, CDCl$_3$): δ 15.7 (s, 1H, exchangeable with D$_2$O), 8.79 (t, $^3$J = 4.0 Hz, 2H), 8.67 – 8.59 (m, 4H), 8.17 (d, $^3$J = 8.0 Hz, 4H), 7.99 (t, $^3$J = 8.0 Hz, 4H), 7.81 – 7.66 (m, 12H), −2.19 (s, 1H, exchangeable with D$_2$O), −2.33 (s, 1H, exchangeable with D$_2$O); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 188.2, 151.8, 145.7, 141.5, 141.4, 141.0, 139.9, 138.9, 138.0, 134.6, 134.5, 134.0, 133.6, 132.8, 129.0, 128.8, 128.4, 128.3, 128.0, 127.9, 127.6, 127.2, 127.1, 124.1, 122.2, 116.5, 114.1; UV-vis (CHCl$_3$) $\lambda_{\text{max}}$ (log ε) 405 (5.37), 460 (4.45), 614 (3.87), 667 (3.75) nm; IR (neat, diffuse reflectance): see Figure S5; HR-MS (ESI$^+$, cone voltage = 30 V, 100% CH$_3$CN) m/e calcd for C$_{44}$H$_{30}$N$_5$O$_2$ ([M·H]$^+$) 660.2400, found 660.2361.

Spectroscopic data for 6: R$_f$ (silica-CH$_2$Cl$_2$/1% MeOH) 0.11; $^1$H NMR (400 MHz, CDCl$_3$): δ 10.8-10.6 (br s, 1H), 8.71 (d, $^3$J = 4.0 Hz, 1H), 8.54 (s, 1H), 8.45 (d, $^3$J = 4.0 Hz, 1H), 8.14 – 8.12 (m, 2H), 8.03 – 7.98 (m, 2H), 7.78 – 7.70 (m, 6H), −2.09 (s, 1H, exchangeable with D$_2$O); $^{13}$C NMR (100 MHz, CDCl$_3$): δ 154.7, 141.6, 141.5, 140.4, 137.2, 134.9, 134.8, 134.4, 133.9, 133.5, 128.5, 128.4, 128.2, 128.1, 127.6, 127.0, 123.4, 113.2; UV-vis (CHCl$_3$) $\lambda_{\text{max}}$ (log ε) 420 (5.11), 530 (4.05), 559 (sh), 610 (3.74), 661 (3.83) nm; IR (neat, diffuse reflectance): See Figure S6; HR-MS (ESI$^+$, cone voltage = 30 V, 100% CH$_3$CN) m/e calcd for C$_{44}$H$_{31}$N$_6$O$_2$ ([M·H]$^+$) 675.2508, found 675.2515.
Figure S1. $^1$H NMR (400 MHz, CDCl$_3$) of 5
Figure S2. $^{13}$C NMR (100 MHz, CDCl$_3$) of 5
Figure S3. ^1^H NMR Spectrum (400 MHz, CDCl3) of 6
Figure S4. $^{13}$C NMR (100 MHz, CDCl$_3$) of 6
Figure S5. FT-IR (neat, diffuse reflectance) of 5

Figure S6. FT-IR Spectrum (neat, diffuse reflectance) of 6
Figure S7. UV-vis spectrum (CH$_2$Cl$_2$) of 6
Mono-quinoline-fused oxo-porphyrin 7.

Monooxime 5 (20.1 mg, 3.05 × 10⁻⁵ mol) was dissolved in toluene (10.0 mL) in a round-bottom flask equipped with a magnetic stir bar. To the stirring solution was added p-TSA (12 mg, 6.31 × 10⁻⁵ mol) and the mixture was heated to reflux for 30 min. When the starting material was consumed (reaction control by UV-vis and TLC), Et₃N (3 drops) were added and the mixture was evaporated to dryness by rotary evaporation. The residue was taken up in CH₂Cl₂ and filtered through a plug of silica gel to removed the excess p-TSA (or salt). The filtrate was washed with water (2 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated to dryness by rotary evaporation. The residue was separated by column chromatography (CH₂Cl₂/1%MeOH). Isolated yields for 7: 76% (14.8 mg).

Spectroscopic data for 7: R_f (silica-CH₂Cl₂) 0.23; ¹H NMR (500 MHz, CD₂Cl₂): δ 9.04 (d, ³J = 4.5 Hz, 1H), 8.96 (d, ³J = 7.5 Hz, 1H), 8.45 (d, ³J = 8.0 Hz, 1H), 8.39 (t, ³J = 5.0 Hz, 2H), 8.25 (d, ³J = 4.5 Hz, 1H), 8.18 (t, ³J = 4.0 Hz, 2H), 8.08 (d, ³J = 7.0 Hz, 2H) 7.99 (d, ³J = 7.0 Hz, 2H), 7.85-7.69 (m, 13H), –0.67 (br s, 2H, exchangeable with D₂O); ¹³C NMR (100 MHz, CD₂Cl₂): δ 195.6, 151.4, 145.4, 143.8, 141.3, 141.1, 139.4, 137.1, 136.1, 135.0, 134.6, 134.1, 133.3, 133.1, 133.0, 132.9, 130.5, 130.4, 130.3, 129.0, 128.7, 128.6, 128.3, 128.2, 128.0, 127.7, 125.6, 123.8, 112.9, 109.1; UV-vis (CHCl₃) λ_max (log ε) 405 (5.17), 480 (4.51), 518 (4.39), 674 (4.16), 750 (4.30) nm; HR-MS (ESI⁺, cone voltage = 30 V, 100% CH₃CN) m/e calcld for C₄₄H₂₈N₅O ([M-H]⁺) 642.2294, found 642.2316.
Figure S8. $^1$H NMR Spectrum (500 MHz, CD$_2$Cl$_2$) of 7
Figure S9. $^{13}$C NMR Spectrum (100 MHz, CD$_2$Cl$_2$) of 7
Figure S10. Partial $^1$H,$^1$H COSY Spectrum (500 MHz, CD$_2$Cl$_2$) of 7

Figure S11. Partial $^1$H,$^1$H NOESY Spectrum (500 MHz, CD$_2$Cl$_2$) of 7
Figure S12. FT-IR Spectrum (neat, diffuse reflectance) of 7
**Mono-quinoline-fused oxo-porphyrin oxime 8.**

Path A. Mono-quinoline-fused oxo-porphyrin oxime 7 (51.8 mg, 8.07 × 10⁻⁵ mmol) was dissolved in pyridine (20 mL) in a round-bottom flask equipped with a magnetic stir bar and N₂ gas inlet. Solid H₂N-OH·HCl (80 mg) was added and the mixture was heated to reflux for 2 h. When the starting material was consumed (reaction control by TLC), the reaction mixture was allowed to cool to ambient temperature, the solvent was removed by rotary evaporation, partially dissolved in CH₂Cl₂ and the mixture was filtered through a glass frit (M). The volume of the filtrate was reduced and separated by column chromatography (CH₂Cl₂-petroleum ether 3:1). Isolated yield for 8: 86% (46 mg). Alternate synthesis: Reflux of N-oxide 11 in pyridine with excess H₂N-OH·HCl for 2 h converts 11 essentially in near-quantitative yields to 8.

Spectroscopic data for 8: Rᵉ (silica-CH₂Cl₂) 0.35; ¹H NMR (400 MHz, CDCl₃): δ 15.9 (s, 1H, exchangeable with D₂O), 9.42 (d, ³J = 8.0 Hz, 1H), 9.27 (d, ³J = 4.0 Hz, 1H), 8.50 – 8.46 (m, 3H), 8.38 (d, ³J = 4.0 Hz, 1H), 8.32 (d, ³J = 4.0 Hz, 1H), 8.26, (d, ³J = 4.0 Hz, 1H), 8.14 – 8.12 (m, 2H), 8.05 – 8.02 (m, 2H), 7.97 (t, ³J = 8.0 Hz, 1H), 7.94 – 7.91 (m, 2H), 7.86 (t, ³J = 8.0Hz, 1H) 7.77-7.67 (m, 10H), 0.23 (s, 1H, exchangeable with D₂O), 0.18 (s, 1H, exchangeable with D₂O); solubility/aggregation issues in longer runs prevented the recording of high-quality ¹³C NMR spectra. UV-vis (CHCl₃) λₘₐₓ (log ε) 406 (sh), 417 (5.19), 461 (sh), 489 (sh), 574 (sh), 635 (sh), 659 (4.37), 700 (4.43) nm; MS (ESI⁺, cone voltage = 30 V, 100% CH₃CN) m/e calcd for C₄₄H₂₉N₆O ([M.H]⁺) 657.2403, found 657.2454.
Figure S13. $^1$H NMR Spectrum (400 MHz, CDCl$_3$) of 8
Figure S14. FT-IR Spectrum (neat, diffuse reflectance) of 8
Bis-quinoline-fused Porphyrin 9 and Oxadiazole 10.

**Route A:** Bis-oxime 6 (13.3 mg, 1.97 × 10^{-5} mol) was dissolved in toluene (10.0 mL) in a round-bottom flask equipped with a magnetic stir bar, p-TSA (7.5 mg, 4.0 × 10^{-5} mol, ~2.0 equiv) was added, and the mixture was heated to reflux for 30 min. When the starting material was consumed (reaction control by TLC), Et₃N (2 drops) was added and the mixture was evaporated to dryness by rotary evaporation. The residue was taken up in CH₂Cl₂ and filtered through a plug of silica gel. The filtrate was washed with water (2 × 10 mL), dried over anhydrous Na₂SO₄, and evaporated to dryness by rotary evaporation. The residue was separated on a preparative TLC plate (CH₂Cl₂-petroleum ether 1:1). Isolated yields for 9: 12% (1.5 mg), 10: 19% (2.5 mg), and 7: 66% (8.3 mg). **Route B:** Bis-fused porphyrin 9 is prepared in 64% isolated yields from mono-fused oxime 8 using the procedure described in route B.

Spectroscopic data for 9: Rₓ (CH₂Cl₂/1% MeOH) 0.30; ¹H NMR (400 MHz, CD₂Cl₂): δ 8.89 (br s, 1H), 8.71-8.69 (m, 8Hz, 1H), 8.28 (br s, 1H), 7.90 (d, two overlapping doublets, ³J = 4.0 Hz, 2H), 7.83 (d, ³J = 8.0 Hz, 2H), 7.71-7.67 (m, 4H), 7.40 (d, ³J = 4.0 Hz, 2H), 7.30 (br s, 1H); solubility/aggregation issues in longer runs prevented the recording of high-quality ¹³C NMR spectra. UV-vis (CHCl₃) λ_max (log ε) 392 (4.60), 432 (sh), 513 (3.77), 571 (sh), 619 (3.95), 675 (4.03), 775 (3.79) nm; MS (ESI⁺, cone voltage = 30 V, 100% CH₃CN) m/e calcd for C₄₄H₂₇N₆ ([M·H]⁺) 639.2292, found 639.2294.

Spectroscopic data for 10: Rₓ (silica-CH₂Cl₂) 0.86; ¹H NMR (400 MHz, CDCl₃): δ 8.95 (d, ³J = 4.0 Hz, 1H), 8.86 (d, ³J = 4.0 Hz, 1H), 8.71 (s, 1H), 8.21 (d, ³J = 8.0 Hz, 2H), 8.16 (d, ³J = 8.0 Hz, 2H), 7.89 – 7.74 (m, 6H), –2.72 (s, 1H, exchangeable with D₂O); ¹³C NMR (100 MHz, CDCl₃): δ 163.8, 155.4, 141.7, 140.8, 139.1, 138.0, 137.5, 134.7, 134.6, 133.4, 129.1, 129.0, 128.3, 128.0, 127.8, 127.1, 123.2, 117.3; UV-vis (CHCl₃) λ_max (log ε) 418 (5.48), 524 (4.27), 586
(4.13), 588 (4.08), 641 (3.60) nm; MS (ESI+, cone voltage = 30 V, 100% CH₃CN) m/e calcd for C₄₄H₂₈N₆O ([M-H]+) 657.2403, found 657.2413.

Figure S15. FT-IR Spectrum (neat, diffuse reflectance) of 9
Figure S16. $^1$H NMR Spectrum (400 MHz, CD$_2$Cl$_2$) of 9
Since free base 9 exhibited solubility problems that made particularly the 13C NMR very hard to record, we also prepared the Ni(II) complex 9Ni. It proved more soluble than the free base:

(9Ni). Free base 12H₂ (11.4 mg, 1.79 × 10⁻⁵ mol) was dissolved in pyridine (5 mL) and added to a hot solution of pyridine (10 mL) and Ni(CH₃CO₂)₂·4H₂O (27 mg, 1.08 × 10⁻⁴ mol, 6.0 equiv) in a round-bottom flask equipped with a magnetic stir bar. The mixture was heated to reflux for 30 min. When the starting material was consumed (reaction control by UV-vis and TLC), the resulting mixture was allowed to cool to room temperature, the solvent was removed in vacuo and taken up in CH₂Cl₂. The resulting mixture was separated on a preparative TLC plate (CH₂Cl₂/5% MeOH). Isolated yield for 9Ni is 55% (7.1 mg): Rᵣ (silica-CH₂Cl₂/2% MeOH) 0.19; ¹H NMR (500 MHz, CDCl₃): δ 8.65 (d, 7.5 Hz, 1H), 8.62 (d, 7.5 Hz, 1H), 8.58 (d, 3J = 4.5 Hz, 1H), 8.03 (d, 3J = 4.5 Hz, 1H), 7.82 – 7.77 (m, 2H), 7.50 (d, 2H), 7.64 (s, 1H) 7.62 – 7.58 (m, 3H). ¹³C NMR (100 MHz, CDCl₃): δ 151.1, 147.5, 145.0, 143.9, 139.1, 138.8, 135.5, 132.7, 132.6, 131.9, 130.9, 129.9, 129.6, 128.7, 127.9, 127.7, 127.6, 126.5, 124.8, 109.6; UV-vis (CHCl₃) λ_max (log e) 402 (4.52), 473 (4.42), 691 (3.91), 727 (4.00), 765 (4.05) nm; MS (ESI⁺, cone voltage = 30 V, 100% CH₃CN) m/e calcd for C₄₄H₂₂₅N₆Ni ([M.H]⁺) 695.1489, found 695.1488.
Figure S17. $^1$H NMR Spectrum (500 MHz, CDCl$_3$) of 9Ni
Figure S18. $^{13}$C NMR Spectrum (100 MHz, CDCl$_3$) of 9Ni
Figure S19. $^1$H NMR Spectrum (400 MHz, CDCl$_3$) of 10
Figure S20. $^{13}$C NMR Spectrum (100 MHz, CDCl$_3$) of 10
**Figure S21.** UV-vis spectrum (CH$_2$Cl$_2$) of 10

**Figure S22.** FT-IR Spectrum (neat, diffuse reflectance) of 10
Mono-quinoline-fused Porphyrin N-oxide 11.

Monooxime 5 (12 mg, $1.74 \times 10^{-5}$ mol) was dissolved in CH$_2$Cl$_2$ (10.0 mL) in a round-bottom flask equipped with a magnetic stirring bar. DDQ (8 mg, $3.5 \times 10^{-5}$ mol, 2 equiv) was added and the mixture was stirred for 30 min. When the starting material was consumed (reaction control by TLC), the reaction mixture was filtered through a short plug of silica gel to removed excess DDQ. The filtrate was washed with water ($2 \times 10$ mL), dried over anhydrous Na$_2$SO$_4$, and evaporated to dryness by rotary evaporation. Product 11 was purified on a preparative tlc plate (CH$_2$Cl$_2$/2% MeOH) and isolated in 94% yields (11 mg).

Spectroscopic data for 11: R$_f$ (silica-CH$_2$Cl$_2$/5% MeOH) 0.72; $^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 9.21 (d, $^3J = 5.0$ Hz, 1H), 9.06 (d, $^3J = 8.0$ Hz, 1H), 9.00 (d, $^3J = 7.5$ Hz, 1H), 8.58 (d, $^3J = 5.0$ Hz, 1H), 8.50 (d, $^3J = 5.0$ Hz, 1H), 8.35 – 8.31 (m, 3H), 8.11 (d, $^3J = 8.0$ Hz, 2H), 8.04 (d, $^3J = 8.0$ Hz, 2H), 7.96 (t, $^3J = 7.5$ Hz, 1H), 7.81 (d, $^3J = 8.0$ Hz, 2H), 7.78 – 7.70 (m, 10H), −0.05 (br s, 2H, exchangeable with D$_2$O); $^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 184.6, 155.6, 153.7, 146.4, 144.9, 143.7, 141.2 141.0, 140.8, 138.7, 138.1, 136.3, 135.7, 134.3, 134.1, 133.7, 133.4, 132.4, 132.3, 131.1, 130.8, 129.5, 128.4, 128.2, 127.7, 127.5, 127.3, 127.0, 126.9, 125.3, 123.3, 121.9, 114.2, 101.5; UV-vis (CHCl$_3$) $\lambda_{max}$ (log $\varepsilon$) 416 (5.35), 427 (sh), 496 (4.67), 534 (4.67), 668 (4.21), 737 (4.49) nm; MS (ESI$^+$, cone voltage = 30 V, 100% CH$_3$CN) m/e calcd for C$_{44}$H$_{28}$N$_5$O$_2$ ([M·H$^+$]), 658.2243 found 658.2213.
Figure S23. $^1$H NMR Spectrum (500 MHz, CDCl$_3$) of 11
Figure S24. $^{13}$C NMR Spectrum (125 MHz, CDCl$_3$) of 11
Figure S25. Partial $^1$H, $^1$H COSY Spectrum (500 MHz, CDCl$_3$) of 11

Figure S26. Partial $^1$H, $^1$H NOESY Spectrum (500 MHz, CDCl$_3$) of 11
Figure S27. FT-IR Spectrum (neat, diffuse reflectance) of 11
**Bis-quinoline-fused N-oxide 12.**

Prepared in 96% isolated yields (20.8 mg) from monooxime 8 (22.3 mg, 3.31 × 10⁻⁴ mmol) dissolved in CH₂Cl₂ (10 mL) and DDQ (16.9 mg, 7.44 × 10⁻⁵ mol, ~2.3 equiv) as described for N-oxide 11.

Spectroscopic data for 12: Rᵋ (silica-CH₂Cl₂/10% MeOH) 0.78; ¹H NMR (400 MHz, CDCl₃): δ 9.20 (d, ³J = 8.0 Hz, 1H), 8.98 – 8.93 (m, 2H), 8.89 (d, ³J = 8.0 Hz, 1H), 8.77 (br s, 2H), 8.15 (br s, 1H), 8.10 (d, ³J = 4.0 Hz, 1H), 7.95 – 7.82 (m, 10H), 7.75 – 7.65 (m, 6H); ¹³C NMR (100 MHz, CDCl₃): δ 145.1, 140.3, 139.6, 133.6, 133.5, 132.2, 131.2, 130.7, 129.6, 128.8, 128.7, 128.5, 127.9, 127.5, 127.4, 126.9, 126.5, 126.0, 125.5, 125.2, 121.2, 102.6; UV-vis (CHCl₃) λ_max (log ε) 412 (4.80), 439 (sh), 485 (sh), 521 (4.12), 568 (3.66), 638 (3.77), 703 (4.14), 779 (4.44) nm; MS (ESI⁺, cone voltage = 30 V, 100% CH₃CN) m/e calcd for C₄₄H₂₇N₆O ([M-H]⁺) 655.2241, found 655.2211.
Figure S28. FT-IR Spectrum (neat, diffuse reflectance) of 12
Figure S29. $^1$H NMR Spectrum (400 MHz, CDCl$_3$) of 12
Figure S30. $^{13}$C NMR Spectrum (100 MHz, CDCl$_3$) of 12
Details to the X-ray Crystal Structure of Bis-quinoline-fused N-oxide 12.

Two crystallographically independent molecules are both flip disordered with the main difference being the position of the oxygen atom, being bonded to one or the other pyridyl N atom. In one of the two molecule sites the disorder also affects about half of the remainder of the molecule, and restraints and constraints were used in the refinement. The occupancy ratios are 0.268(5) to 0.732(5) for the two disordered oxygen atoms O1A and O1B, and 0.522(3) to 0.478(3) for the two disordered fragments. Equivalent bond distances in the two fragments were restraint to be similar, and overlapping atoms were constrained to have identical ADPs. The atoms C4C, C18C, C20C, N4C and C16C were also restrained to be approximately isotropic.

Figure S31. ORTEP plot of X-ray crystal structure of 12. Thermal ellipsoid plot of the asymmetric unit. Bonds unique to the minor units are shown as dashed lines.
**Figure S32.** ORTEP plot of X-ray crystal structure of 12. Thermal ellipsoid plot of molecules A and B. Bonds unique to the minor B unit are shown as dashed lines.

**Figure S33.** ORTEP plot of X-ray crystal structure of 12. Thermal ellipsoid plot of molecules C and D. Bonds unique to the minor B unit are shown as dashed lines.
Figure S34. ORTEP plot of X-ray crystal structure of 12. Thermal ellipsoid plot of molecules A with the minor moiety B omitted.

Figure S35. ORTEP plot of X-ray crystal structure of 12. Thermal ellipsoid plot of molecules C with the minor moiety D omitted.
Figure S36. ORTEP plot of X-ray crystal structure of 12. Thermal ellipsoid packing plot. Molecules A and B: red; molecules C and D: blue.
Table 1. Experimental details, X-ray diffraction analysis of 12

<table>
<thead>
<tr>
<th>Crystal data</th>
<th>12 (internal code: 10mz505_0m)</th>
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<td>C₄₄H₂₆N₆O</td>
</tr>
<tr>
<td>$M_r$</td>
<td>654.71</td>
</tr>
<tr>
<td>Crystal system, space group</td>
<td>Monoclinic, $P2_1/c$</td>
</tr>
<tr>
<td>Temperature (K)</td>
<td>100</td>
</tr>
<tr>
<td>$a$, $b$, $c$ (Å)</td>
<td>25.595 (6), 9.572 (2), 25.369 (6)</td>
</tr>
<tr>
<td>$\beta$ (°)</td>
<td>97.710 (4)</td>
</tr>
<tr>
<td>$V$ (Å³)</td>
<td>6159 (2)</td>
</tr>
<tr>
<td>$Z$</td>
<td>8</td>
</tr>
<tr>
<td>$F(000)$</td>
<td>2720</td>
</tr>
<tr>
<td>$D_\text{x}$ (Mg m⁻³)</td>
<td>1.412</td>
</tr>
<tr>
<td>Radiation type</td>
<td>Mo $\text{K}\alpha$</td>
</tr>
<tr>
<td>$\mu$ (mm⁻¹)</td>
<td>0.09</td>
</tr>
<tr>
<td>Crystal shape</td>
<td>Plate</td>
</tr>
<tr>
<td>Colour</td>
<td>Black</td>
</tr>
<tr>
<td>Crystal size (mm)</td>
<td>$0.25 \times 0.21 \times 0.09$</td>
</tr>
<tr>
<td>Data collection</td>
<td></td>
</tr>
<tr>
<td>Diffractometer</td>
<td>Bruker AXS SMART APEX CCD diffractometer</td>
</tr>
<tr>
<td>Radiation source</td>
<td>fine-focus sealed tube</td>
</tr>
<tr>
<td>Absorption correction</td>
<td>Multi-scan, Apex2 v2009.7-0 (Bruker, 2009)</td>
</tr>
<tr>
<td>$T_{\text{min}}$, $T_{\text{max}}$</td>
<td>0.655, 0.746</td>
</tr>
<tr>
<td>No. of measured, independent and observed [$I &gt; 2\sigma(I)$] reflections</td>
<td>36350, 15006, 7663</td>
</tr>
<tr>
<td>$R_{\text{int}}$</td>
<td>0.073</td>
</tr>
<tr>
<td>$\theta$ values (°)</td>
<td>$\theta_{\text{max}} = 28.3$, $\theta_{\text{min}} = 0.8$</td>
</tr>
<tr>
<td>Range of $h$, $k$, $l$</td>
<td>$h = -34 \rightarrow 27$, $k = -12 \rightarrow 11$, $l = -33 \rightarrow 28$</td>
</tr>
<tr>
<td>Refinement</td>
<td></td>
</tr>
<tr>
<td>$R[F^2 &gt; 2\sigma(F^2)]$, $wR(F^2)$, $S$</td>
<td>0.069, 0.179, 1.02</td>
</tr>
<tr>
<td>No. of reflections</td>
<td>15006</td>
</tr>
<tr>
<td>No. of parameters</td>
<td>1017</td>
</tr>
<tr>
<td>No. of restraints</td>
<td>91</td>
</tr>
<tr>
<td>H-atom treatment</td>
<td>H-atom parameters constrained</td>
</tr>
<tr>
<td>$\Delta_{\text{max}}$, $\Delta_{\text{min}}$ (e Å⁻³)</td>
<td>0.32, -0.27</td>
</tr>
</tbody>
</table>