Supporting Information

Synthesis, X-ray crystal structures, and computational studies of 1,1'-bridged 4,4'-diaryl-2,2'-bibenzimidazoles: building blocks for supramolecular structures

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General

Unless otherwise noted, all reactions were performed under nitrogen. Anhydrous THF was supplied from an Mbraun solvent purification system. Solvents were used as purchased (p. a. grade) without further purification. All reagents were also used as purchased without further purification. Analytical thin layer chromatography was performed with Macherey–Nagel POLYGRAM ALOX N/UV_{254}. Alumina chromatography was performed with deactivated (5% water) Sigma–Aldrich activated aluminum oxide (Type 507C, 150 mesh).

Proton nuclear magnetic resonance (^{1}H-NMR) spectra were recorded on a Bruker Avance 300 at 300 MHz, a Bruker ARX 300 at 300 MHz, or a Bruker Avance 400 at 400 MHz. Chemical shifts are reported relative to $SiMe_4$ (0.00 ppm), $CHCl_3$ (7.26 ppm), or DMSO (2.54 ppm). Multiplicity is indicated by one or more of the following: s (singlet); d (doublet); t (triplet); quint (quintet); m (multiplet); br (broad). Carbon nuclear magnetic resonance (^{13}C-NMR) spectra were recorded on a Bruker ARX 300 at 75 MHz, on a Bruker Avance 400 at 100 MHz or on a Bruker Avance 500 at 125 MHz. Chemical shifts are reported relative to $CDCl_3$ (77.2 ppm) or DMSO-d_6 (39.5 ppm).

4,4'-bis(4-methoxyphenyl)-6,6'-dimethyl-2,2'-bibenzimidazole 2a. To a solution of **1a** (2.18 g, 3.23 mmol) in CH_2Cl_2 (30 mL) was slowly added trifluoroacetic acid (3.5 mL, 5.2 g, 46 mmol). The reaction mixture was stirred 90 min in air at room temperature. Saturated $NaHCO_3$ solution was then added until the reaction mixture tested pH ≈ 8 on indicator paper, at which point a white precipitate formed. A 1:1 mixture of ether and hexane was added to the quenched reaction mixture, which was allowed to stir for an additional 10 min before the precipitate was filtered and washed with water and hexane, and dried in vacuo. Compound **2a** (1.46 g, 95%) was produced as a white powder. Rf 0.18 (CH_2Cl_2); mp. decomp. 299-300°C; ^{1}H NMR (300 MHz, DMSO-d_6, δ) 13.04 (br s, 2H), 8.13 (br d, 4H, J = 7.2 Hz), 7.32 (s, 2H), 7.24 (s, 2H), 7.08 (d, 4H, J = 8.7 Hz), 3.85 (s, 6H), 2.50 (s, embedded in solvent peak) ; ^{13}C NMR (100 MHz, DMSO-d_6, 353 K, δ) 158.4, 143.4, 138.6, 136.6, 132.3, 130.5, 129.8, 129.5, 121.8, 113.5, 110.6, 54.9, 21.0; HRMS (ESI) Calcd for $C_{30}H_{26}N_4O_2Na$ (M + Na^+) 497.1953, found 497.1957.

4,4'-bis(4-methoxyphenyl-2,6-dimethyl)-6,6'-dimethyl-2,2'-bibenzimidazole 2b To a solution of **1b** (2.00 g, 2.74 mmol) in CH_2Cl_2 (30 mL) was slowly added trifluoroacetic acid (3.5 mL, 5.2 g, 46 mmol). The reaction mixture was stirred for 3 h in air at room temperature. Saturated $NaHCO_3$ solution was then added until the reaction mixture tested pH ≈ 8 on indicator paper, at which point a white precipitate formed. Hexane was added and the mixture was stirred before the precipitate was filtered, washed with water and hexane, and dried in vacuo. Compound **2b** (1.40 g, 96 %) was produced as a white powder. Rf 0.46 (CH_2Cl_2); mp. >320° C; ^{1}H NMR (300 MHz, DMSO-d_6, δ) 13.13 (br s, 2H), 7.25 (s, 2H), 6.73 (s, 6H (4H + 2H)), 3.79 (s, 6H), 2.43 (s, 6H), 1.92 (s, 12H); ^{13}C NMR (125 MHz, DMSO-d_6, 350 K, δ) 158.0, 144.2, 139.3, 137.3, 131.8, 131.8, 131.3,

129.7, 112.4, 111.5, 54.8, 21.0, 20.3; HRMS (ESI) Calcd for $C_{34}H_{34}N_4O_2Na$ (M + Na^+) 553.2579, found 553.2578.

6,11-Dihydro-1,16-bis(4-methoxyphenyl)-3,14-dimethylbisbenzimidazo[1,2-*b*:2',1'-*d*][2,5]benzodiazocine 3a. To a solution of of **2a** (299 mg, 629 μmol) in THF (30 mL) was added Cs_2CO_3 (621 mg, 1.90 mmol), α,α'-dibromo-*o*-xylene (247 mg, 936 mmol), and NaI (97 mg, 647 mmol). The reaction mixture was then stirred under nitrogen at 70 °C for 19 h. Water was added to the reaction mixture and the THF was removed in vacuo, causing a yellow solid to precipitate. Hexane and a small amount of THF (to dissolve yellow byproduct) were added to the water and the two-phase mixture containing a white precipitate was allowed to stir for 30 minutes. The precipitate was then filtered and washed with water and hexane, leaving a white powder. Recrystallization with CH_2Cl_2 / hexane afforded **3a** (260 mg, 72%) as clear crystals. Rf 0.70 (CH_2Cl_2); mp. >320°C; ^{1}H NMR (300 MHz, $CDCl_3$, δ) 8.08 (d, 4H, *J* = 9.0 Hz), 7.44 (m, 4H), 7.30 (br s, 2H), 7.11 (br s, 2H), 7.04 (d, 4H, *J* = 9.0 Hz) 5.13 (s, 4H), 3.86 (s, 6H), 2.53 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$, δ) 159.2, 144.5, 139.4, 136.1, 134.4, 133.7, 133.0, 130.4, 129.7, 129.0, 123.5, 113.9, 107.8, 55.3, 48.4, 21.9; HRMS (ESI) Calcd for $C_{38}H_{32}N_4O_2Na$ (M+Na^+) 599.2423, found 599.2428.

6,11-Dihydro-1,16-bis(4-methoxyphenyl-2,6-dimethyl)-3,14-dimethylbisbenzimidazo-[1,2-*b*:2',1'-*d*][2,5]benzodiazocine 3b. To a solution of **2b** (200 mg, 0.377 mmol) in THF (40 mL) were added Cs_2CO_3 (365 mg, 1.12 mmol), α,α'-dibromo-*o*-xylene (149 mg, 0.565 mmol) and NaI (58 mg, 0.37 mmol). The reaction was stirred under nitrogen at 60 ºC for 7 d. Water was then added and the mixture was stirred. The resulting precipitate was filtered and washed with water and hexane. Alumina column chromatography (hexane/CH_2Cl_2 = 9:1 → 5:5) afforded **3b** (185 mg, 78%). Rf 0.72 (CH_2Cl_2); mp. >320°C; ^{1}H NMR (300 MHz, $CDCl_3$) δ) 7.47 (m, 4H), 7.15 (br s, 2H), 6.93 (br s, 2H), 6.67 (s, 4H), 5.10 (s, 4H), 3.80 (s, 6H), 2.51 (s, 6H), 2.00 (s, 12H); ^{13}C NMR (75 MHz, $CDCl_3$ δ) 158.8, 144.7, 140.7, 138.1, 135.5, 134.3, 134.1, 133.8, 130.6, 130.1, 129.3, 126.4, 113.0, 108.1, 55.3, 48.6, 22.0, 21.4; HRMS (ESI) Calcd for $C_{42}H_{40}N_4O_2Na$ (M + Na^+) 655.3049, found 655.3050.

7,8-Dihydro-1,13-bis(4-methoxyphenyl)-3,11-dimethyl-6*H*-benzimidazo[2',1':3,4]-[1,4]diazepino[1,2-*a*]benzimidazole 3c. To a solution of **2a** (300 mg, 0.632 mmol) in THF (30 mL) were added Cs_2CO_3 (620 mg, 1.90 mmol), 1,3-dibromopropane (642 mg, 3.12 mmol) in THF (20 mL) and NaI (94 mg, 0.63 mmol). The reaction was stirred under nitrogen at 65 ºC for 22 h. The reaction mixture was then added to a biphasic mixture of water and hexane, stirred and filtered. The precipitate was collected and dried in vacuo to give **3c** as a white powder (242 mg, 75%). Rf 0.19 (CH_2Cl_2); mp. decomp. 310-311°C; ^{1}H NMR (300 MHz, $CDCl_3$, δ) 8.03 (d, 4H, *J* = 8.7 Hz), 7.27 (br s, 2H), 7.11 (br s, 2H), 6.93 (d, 4H, *J* = 8.7 Hz) 4.37 (t, 4H, *J* = 6.2 Hz), 3.84 (s, 6H), 2.62 (quint, 2H, *J* = 6.2 Hz), 2.56 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$, δ) 159.3, 143.5, 138.9, 136.5, 134.5, 132.7, 130.7, 130.6, 123.7, 113.9, 107.8, 55.5, 42.8, 28.2, 22.3; HRMS (ESI) Calcd for $C_{33}H_{30}N_4O_2Na$ (M + Na^+) 537.2266, found 537.2268.

7,8-Dihydro-1,13-bis(4-methoxyphenyl-2,6-dimethyl)-3,11-dimethyl-6*H*-benzimid-azo[2',1':3,4][1,4]diazepino[1,2-*a*]-benzimidazole 3d. To a solution of **2b** (201 mg, 0.379 mmol) in THF (30 mL) were added Cs_2CO_3 (304 mg, 0.933 mmol), 1,3-dibromopropane (160 μL, 317 mg, 1.57 mmol) and NaI (61 mg, 0.41 mmol). The mixture was stirred under nitrogen for 5 days at 70 ºC. Water was added and THF was selectively removed in vacuo. Hexane was added to the water mixture and a white precipitate was filtered and dried in vacuo. Alumina column chromatography (CH_2Cl_2/hexane = 5:5) afforded **3d** as a white powder. Rf 0.64 (CH_2Cl_2); mp. >320°C; ^{1}H NMR (400 MHz, $CDCl_3$ δ) 7.17 (s, 2H), 6.90 (s, 2H), 6.61 (s, 4H), 4.34 (t, 4H, *J* = 6.4 Hz), 3.76 (s, 6H),

2.61 (quint, 2H, J = 6.2 Hz) 2.53 (s, 6H), 1.98 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$ δ) 158.7, 144.3, 140.6, 138.2, 135.9, 134.0, 133.7, 130.9, 126.5, 112.9, 107.8, 55.3, 41.9, 28.7, 22.1, 21.4; HRMS (ESI) Calcd for $C_{37}H_{38}N_4O_2Na$ (M + Na^+) 593.2892, found 593.2896.

7,8-Dihydro-1,13-bis(4-methoxyphenyl)-3,11-dimethyl-6*H*-benzimidazo[2',1':3,4][1,4]diazino[1,2-*a*]benzimidazole 3e. To a solution of **2a** (299 mg, 0.629 mmol) in 50 mL were added Cs_2CO_3 (616 mg, 1.89 mmol), 1-bromo-2-chloroethane (404 mg, 2.83 mmol) in 20 mL THF and NaI (98 mg, 0.65 mmol). The reaction was stirred under nitrogen at 65 ºC for 7 d. The reaction mixture was cooled to rt and water was added. The mixture was allowed to stir for 20 min, and the resulting precipitate was filtered and dried under vacuum. Recrystallization from DMF afforded **3e** as white crystals (209 mg, 66% yield). Rf 0.28 (CH_2Cl_2); mp. >320°C; ^{1}H NMR (300 MHz, DMSO-d_6, δ) 8.10 (d, 4H, J = 9.0 Hz), 7.51 (br s, 2H), 7.35 (br s, 2H), 7.14 (d, 4H, J = 9.0 Hz), 4.81 (s, 4H), 3.88 (s, 6H), 2.58 (s, 6H); ^{13}C NMR (100 MHz, DMSO-d_6, 350K) δ) 158.6, 140.8, 139.0, 134.9, 133.2, 131.0, 131.1, 129.6, 122.4, 113.6, 108.3, 54.9, 21.0; HRMS (ESI) Calcd for $C_{32}H_{28}N_4O_2Na$ (M + Na^+) 523.2110, found 523.2117.

7,8-Dihydro-1,13-bis(4-methoxyphenyl-2,6-dimethyl)-3,11-dimethyl-6*H*-benzimidazo[2',1':3,4][1,4]diazino[1,2*a*]benz-imidazole 3f. To a solution of **2b** (200 mg, 0.377 mmol) in THF (30 mL) was added Cs_2CO_3 (492 mg, 1.51 mmol). To this solution were added 1-bromo-2-chloroethane (249 µL, 431 mg, 3.01 mmol) and NaI (56 mg, 0.373 mmol). The mixture was stirred under nitrogen for 10 days at 60 ºC. Water was added and the mixture was allowed to stir for 30 min. The resulting precipitate was washed with water and hexane and dried in vacuo. Trituration in THF afforded **3f** as a white powder (90 mg, 43%). Rf 0.58 (CH_2Cl_2); mp. >320°C; ^{1}H NMR (400 MHz, DMSO-d_6) δ) 7.48 (s, 2H), 6.85 (s, 2H), 6.69 (s, 4H), 4.76 (s, 4H), 3.76 (s, 6H), 2.51 (s, embedded in solvent signal), 1.89 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ) 158.8, 141.3, 141.2, 138.1, 134.7, 134.4, 133.9, 130.9, 126.8, 112.9, 107.8, 55.32, 40.7, 22.1, 21.3; HRMS (ESI) Calcd for $C_{36}H_{36}N_4O_2Na$ (M + Na^+) 579.2736, found 579.2746.

6,11-Dihydro-1,16-dibromo-3,14-dimethylbisbenzimidazo[1,2-*b*:2',1'-*d*][2,5]benzo-diazocine 5a. To a solution of 4,4'-dibromo-6,6'-dimethyl-BBI[7] (568 mg, 1.19 mmol) in DMF (100 mL) was added K_2CO_3 (512 mg, 3.89 mmol) and the mixture was allowed to stir 15 min before α,α'-dibromo-*o*-xylene (658 mg, 2.39 mmol) was added. The mixture was headed to 60 ºC for 21 h, then cooled to room temperature. Water (100 mL) was then added and the mixture was stirred for 30 min, during which time a white, gelatinous precipitate formed. The precipitate was filtered and washed with water and hexane and dried *in vacuo* to yield **5** as a dull white powder (621 mg, 88%). Rf (compound does not elute in hexane, toluene, $CHCl_3$, CH_2Cl_2, EtOAc, MeOH, EtOH, or water). m.p. >300ºC; ^{1}H NMR (300 MHz, $CDCl_3$) δ) 7.46 (m, 4H), 7.41 (s, 2H), 7.15 (s, 2H), 5.13 (br s, 4H), 2.49 (s, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ) 140.5, 138.5, 135.8, 133.7, 129.8, 129.3, 127.9, 108.7, 67.2, 59.0, 48.7, 21.5; LRMS (EI): m/z = 522.0 100% (M^+). Anal. Calcd for $C_{24}H_{18}Br_2H_4$: C, 55.2; H, 3.47; N, 10.73. Found: C, 54.68; H, 3.36; N, 10.68.

7,8-Dihydro-1,13-dibromo-3,11-dimethyl-6*H*-benzimidazo[2',1':3,4][1,4]diazepino-[1,2-*a*]benzimidazole 5b. To a solution of 4,4'-dibromo-6,6'-dimethyl-BBI[7] (1.52 g, 3.59 mmol) in DMF (300 mL) was added K_2CO_3 (1.54 g, 10.8 mmol) and the mixture was allowed to stir 15 min before 1,3-dibromopropane (1.8 mL, 18 mmol) was added. The mixture was headed to 60 ºC for 22 h, then cooled to room temperature. Water (250 mL) was then added and the mixture was stirred for 30 min, during which time a white, gelatinous precipitate formed. The precipitate was filtered and washed with water and hexane and dried *in vacuo* to yield **5b** as a dull white powder (1.11 g, 67%). Rf (compound does not elute in hexane, toluene, $CHCl_3$, CH_2Cl_2, EtOAc, MeOH, EtOH, or

water). m.p. >300ºC; ^{1}H NMR (300 MHz, DMSO-d_6, δ) 7.52 (s, 2H), 7.40 (s, 2H), 4.53 (t, 4H, J = 4.0 Hz), 2.58 (m, 2H) 2.49 (s, 6H); ^{13}C NMR (100 MHz, DMF-d_7, sample dissolved at 90 ºC, spectra acquired immediately at 300 K, δ) 137.9, 137.2, 128.1, 126.0, 122.5, 113.7, 111.2, 45.6, 27.8, 21.8; LRMS (EI): m/z = 460.1 100% (M^+).

7,8-Dihydro-1,13-dibromo-3,11-dimethyl-6*H*-benzimidazo [2',1':3,4][1,4]diazino[1,2-*a*]benzimidazole 5c. To a solution of 4,4'-dibromo-6,6'-dimethyl-BBI[7] (49.8 mg, 0.12 mmol) in DMF (10 mL) was added K_2CO_3 (54 mg, 0.36 mmol) and the mixture was allowed to stir 15 min before 1-bromo-2-chloroethane (0.05 mL, 0.6 mmol) was added. The mixture was headed to 60 ºC for 4 d, then cooled to room temperature. Water (50 mL) was then added and the mixture was stirred for 30 min, during which time a white, gelatinous precipitate formed. The precipitate was filtered and washed with water and hexane and dried *in vacuo* to yield **5b** as a dull white powder (32 mg, 6%). Rf (compound does not elute in hexane, toluene, $CHCl_3$, CH_2Cl_2, EtOAc, MeOH, EtOH, or water). m.p. >300ºC; ^{1}H NMR (300 MHz, DMF-d_7, sample dissolved at 90 ºC, spectra acquired immediately at 300 K, δ) 7.58 (s, 2H), 7.43 (s, 2H), 4.91 (s, 4H), 2.53 (s, 6H), 2.58 (s, 6H); ^{13}C NMR (100 MHz, DMF-d_7, sample dissolved at 90 ºC, spectra acquired immediately at 300 K, δ) 136.6, 135.3, 130.8, 128.4, 120.8, 113.8, 111.2, 43.8, 21.8; LRMS (EI): m/z = 446.0 100% (M^+).

Crystal data

Compound **3a** (obtained from CH_2Cl_2/hexane): $C_{38}H_{32}N_4O_2$, M = 576.70, space group: $P\bar{1}$ (triclinic), a = 10.2157(3) Å, b = 11.9363(5) Å, c = 12.9452(5) Å, α = 66.518(2)º, β = 89.148(2)º, γ = 86.525(2)º, V = 1445.05(9) Å^3, Z = 2, F(000) = 608, μ(Mo $K\alpha$) = 0.0830 mm^{-1}, Dx = 1.325 g cm^{-3}, $2\theta_{(max)}$ = 60º, T = 160 K, 36774 measured reflections, 8408 independent reflections, 5233 reflections with $I > 2\sigma(I)$, refinement on F^2 with SHELXL97, 402 parameters, $R(F)$ [$I > 2\sigma(I)$ reflections] = 0.0615, $wR(F^2)$ [all data] = 0.1543, goodness of fit = 1.047, $\Delta\rho_{max}$ = 0.33 e Å^{-3}.

Compound **3b** · acetone (obtained from CH_2Cl_2/acetone): $C_{45}H_{46}N_4O_3$, M = 690.88, space group: $P2_1/c$ (monoclinic), a = 21.4622(3) Å, b = 7.4031(1) Å, c = 23.7326(3) Å, β = 94.2256(9)º, V = 3760.55(9) Å^3, Z = 4, F(000) = 1472, μ(Mo $K\alpha$) =0.0767 mm^{-1}, Dx = 1.220 g cm^{-3}, $2\theta_{(max)}$ = 55º, T = 160 K, 86943 measured reflections, 8633 independent reflections, 5874 reflections with $I > 2\sigma(I)$, refinement on F^2 with SHELXL97, 480 parameters, $R(F)$ [$I > 2\sigma(I)$ reflections] = 0.0770, $wR(F^2)$ [all data] = 0.2173, goodness of fit = 1.066, $\Delta\rho_{max}$ = 0.66 e Å^{-3}.

Compound **3c** · CH_2Cl_2 (obtained from CH_2Cl_2/hexane): $C_{34}H_{32}Cl_2N_4O_2$, M = 599.56, space group: $P2_1/c$ (monoclinic), a = 12.7522 (2) Å, b = 22.9038(4) Å, c = 10.1324(2) Å, β = 106.800(1)º, V = 2833.11(9) Å^3, Z = 4, F(000) = 1256, μ(Mo $K\alpha$) = 0.269 mm^{-1} Dx = 1.406 g cm^{-3}, $2\theta_{(max)}$ = 55º, T = 160 K, 57343 measured reflections, 6474 independent reflections, 4378 reflections with $I > 2\sigma(I)$, refinement on F^2 with SHELXL97, 384 parameters, $R(F)$ [$I > 2\sigma(I)$ reflections] = 0.0703, $wR(F^2)$ [all data] = 0.2237, goodness of fit = 1.036, $\Delta\rho_{max}$ = 1.20 e Å^{-3}.

Compound **3d** · 2 CH_2Cl_2 (obtained from CH_2Cl_2/hexane): $C_{39}H_{42}Cl_4N_4O_2$, M = 740.60, space group: $Pbca$ (orthorhombic), a = 20.2497(2) Å, b = 14.7082(2) Å, c = 24.9430(4) Å, V = 7428.9(2) Å^3, Z = 8, F(000) = 3104, μ(Mo $K\alpha$) = 0.538 mm^{-1} Dx = 1.324 g cm^{-3}, $2\theta_{(max)}$ = 55º, T = 160 K, 89418 measured reflections, 8506 independent reflections, 6208 reflections with $I > 2\sigma(I)$, refinement on F^2 with SHELXL97, 451 parameters, $R(F)$ [$I > 2\sigma(I)$ reflections] = 0.0538, $wR(F^2)$ [all data] = 0.1457, goodness of fit = 1.057, $\Delta\rho_{max}$ =

0.40 *e* Å^{-3}.

Compound **3e** (obtained from DMF): $C_{32}H_{28}N_4O_2$, M = 500.60, space group: $C2/c$ (monoclinic), a = 23.0441(8) Å, b = 12.4981(5) Å, c = 17.4616(6) Å, β = 98.296(3)º, V = 4976.4(3) Å^3, Z = 8, F(000) = 2112, μ(Mo $K\alpha$) = 0.0850 mm^{-1} Dx = 1.336 g cm^{-3}, $2\theta_{(max)}$ = 50º, T = 160 K, 30928 measured reflections, 4389 independent reflections, 3012 reflections with $I > 2\sigma(I)$, refinement on F^2 with SHELXL97, 348 parameters, $R(F)$ [$I > 2\sigma(I)$ reflections] = 0.0575, $wR(F^2)$ [all data] = 0.1522, goodness of fit = 1.073, $\Delta\rho_{max}$ = 0.29 *e* Å^{-3}.

Compound **3f** (obtained from layering ether above an NMR sample in $CDCl_3$): $C_{36}H_{36}N_4O_2$, M = 556.71, space group: $P2_1/c$ (monoclinic), a = 8.5899(4) Å, b = 27.008(1) Å, c = 12.9677(5) Å, β = 104.886(3)º, V = 2907.5(2) Å^3, Z = 4, F(000) = 1184, μ(Mo $K\alpha$) = 0.0797 mm^{-1} Dx = 1.272 g cm^{-3}, $2\theta_{(max)}$ = 50º, T = 160 K, 42093 measured reflections, 5103 independent reflections, 3203 reflections with $I > 2\sigma(I)$, refinement on F^2 with SHELXL97, 388 parameters, $R(F)$ [$I > 2\sigma(I)$ reflections] = 0.0933, $wR(F^2)$ [all data] = 0.2685, goodness of fit = 1.041, $\Delta\rho_{max}$ = 1.22 *e* Å^{-3}.

Compound **5a** (obtained from EtOAc/CH_2Cl_2): $C_{24}H_{18}Br_2N$, M = 522.24, space group: $C2/c$ (monoclinic), a = 17.1485(4) Å, b = 15.7133(6) Å, c = 8.9323(5) Å, β = 119.689(2)º, V = 2090.9(1) Å^3, Z = 4, F(000) = 1040, μ(Mo $K\alpha$) = 3.908 mm^{-1} Dx = 1.659 g cm^{-3}, $2\theta_{(max)}$ = 55º, T = 160 K, 22598 measured reflections, 2396 independent reflections, 1952 reflections with $I > 2\sigma(I)$, refinement on F^2 with SHELXL97, 137 parameters, $R(F)$ [$I > 2\sigma(I)$ reflections] = 0.0669, $wR(F^2)$ [all data] = 0.1859, goodness of fit = 1.103, $\Delta\rho_{max}$ = 1.42 *e* Å^{-3}.