(Supporting Information)
Insertion and Reduction Chemistry of Isocyanide with a Ditantalum Hydride Complex

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Experimental Section

General procedures

All manipulations were carried out using standard Schlenk techniques or in a glove-box under argon atmosphere. Anhydrous hexane and toluene were dried by passage through two columns of activated alumina and a Q-5 column, while anhydrous THF and DME were dried by passage through two columns of activated alumina. Anhydrous benzene and pentane were purchased from Kanto Chemical and used without further purification. Deuterated benzene (benzene-d_6) was dried and degassed over a potassium mirror prior to use. Deuterated tetrahydrofuran (THF-d_8) was distilled from calcium hydride prior to use. The starting complexes [{(OOCO)Ta}$_2$(μ-H)$_3$][M(DME)$_2$] (M = K (**1-K**), Na (**1-Na**)) were prepared by the literature procedure[1]. All other reagents were purchased and used without further purification. ^{1}H and ^{13}C NMR spectra were recorded on JEOL Lambda-400 and Lambda-500 spectrometers. All spectra were referenced to residual protiosolvent (^{1}H) or solvent (^{13}C) resonances. Elemental analyses (C, H, and N) were carried out on a YANACO MT-6 microanalyzer.

Synthesis of [{(OOCO)Ta}{(OOO)Ta}(μ-H)(μ-η^2:η^2-HCN*t*Bu)][K(DME)$_2$] (2-K)

To a THF (20 mL) solution of **1a** (1.00 g, 0.639 mmol) at −30 °C was added dropwise *t*BuNC (140 μL, 1.24 mmol) with stirring. The mixture was stored at −30 °C for 9 h and then all volatiles were removed in vacuo. The brown residue was extracted into DME (1 mL) and was filtrated. The filtrate was layered with hexane to give **2-K** as orange crystals (460 mg, 0.279 mmol, 44%).

^{1}H NMR (500 MHz, THF-d_8): δ 0.93 (s, 9H, *t*Bu), 0.94 (s, 9H, *t*Bu), 1.07 (s, 9H, *t*Bu), 1.20 (s, 9H, *t*Bu), 1.28 (s, 9H, *t*Bu), 1.42 (s, 9H, *t*Bu), 1.44 (s, 9H, *t*Bu), 2.09 (s, 3H, *p*Me), 2.11 (s, 3H,

*p*Me), 2.24 (s, 3H, *p*Me), 2.36 (s, 3H, *p*Me), 3.27 (s, 12H, DME), 3.29 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 3.43 (s, 8H, DME), 3.44 (d, $^2J_{HH}$ = 13 Hz, 2H, CH_2), 3.55 (s, 1H, CH), 4.75 (s, 1H, NCH), 4.81 (d, $^2J_{HH}$ = 13 Hz, 2H, CH_2), 4.82 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 5.70 (s, 1H, *m*H), 6.27 (s, 1H, *m*H), 6.48 (s, 1H, *m*H), 6.49 (s, 1H, *m*H), 6.67 (s, 2H, *m*H), 6.70 (s, 1H, *m*H), 6.88 (s, 1H, *m*H), 6.94 (s, 1H, *m*H), 6.96 (s, 1H, *m*H), 6.99 (s, 1H, *m*H), 7.01 (s, 1H, *m*H), 8.92 (s, 1H, TaH). ^{13}C NMR (126 MHz, THF-d_8): δ 21.2, 21.4, 21.9, 22.2 (*p*Me), 31.90, 31.92, 32.1, 32.3, 32.8, 33.0, 34.0 (C*Me*$_3$), 34.4 (CH_2), 35.0, 35.2 (*C*Me$_3$), 35.6 (CH_2), 35.7, 35.8, 36.0, 36.5 (*C*Me$_3$), 37.7 (CH_2), 59.3 (DME), 60.6 (N*C*Me$_3$), 71.2 (CH), 73.1 (DME), 123.5, 123.6, 124.8, 125.0, 125.7, 126.1, 126.56, 126.61, 127.3, 127.6, 128.6, 129.5, 129.77, 129.80, 129.85, 130.1, 130.2, 131.1, 131.2, 131.3, 131.7, 133.0, 133.1, 135.5, 139.9, 140.2, 140.3, 141.2, 146.6 (Ar), 147.8 (NCH), 148.3, 155.1, 155.4, 157.9, 159.5, 162.4, 163.6 (Ar). Anal. Calcd for $C_{81}H_{116}KNO_{10}Ta_2$: C, 58.44; H, 7.02; N, 0.84. found: C, 58.69; H, 7.22; N, 0.92.

Synthesis of [{(OOCO)Ta}{(OOO)Ta}(μ-H)(μ-η^2:η^2-HCN*t*Bu)] [Na(DME)$_2$] (2-Na)

A mixture of **1-Na** (500 mg, 0.319 mmol) and *t*BuNC (75.0 μL, 0.663 mmol) in (15 mL) THF was treated as described above to give 193 mg of **2-Na** (0.117 mmol, 37%).

^{1}H NMR (400 MHz, THF-d_8): δ 0.94 (s, 18H, *t*Bu), 1.08 (s, 9H, *t*Bu), 1.17 (s, 9H, *t*Bu), 1.29 (s, 9H, *t*Bu), 1.41 (s, 18H, *t*Bu), 2.10 (s, 3H, *p*Me), 2.11 (s, 3H, *p*Me), 2.25 (s, 3H, *p*Me), 2.37 (s, 3H, *p*Me), 3.27 (s, 12H, DME), 3.31 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 3.43 (s, 8H, DME), 3.47 (d, $^2J_{HH}$ = 13 Hz, 2H, CH_2), 3.65 (s, 1H, CH), 4.76 (s, 1H, NCH), 4.79 (d, $^2J_{HH}$ = 13 Hz, 2H, CH_2), 4.82 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 5.71 (s, 1H, *m*H), 6.29 (s, 1H, *m*H), 6.48 (s, 1H, *m*H), 6.50 (s, 1H, *m*H), 6.68 (s, 2H, *m*H), 6.70 (s, 1H, *m*H), 6.88 (s, 1H, *m*H), 6.95 (s, 1H, *m*H), 6.97 (s, 1H, *m*H), 7.00 (s, 1H, *m*H), 7.01 (s, 1H, *m*H), 8.94 (s, 1H, TaH). ^{13}C NMR (100 MHz, THF-d_8): δ 21.2, 21.4, 21.9, 22.2 (*p*Me), 31.9, 32.0, 32.1, 32.3, 32.8, 33.0, 33.8 (C*Me*$_3$), 35.0 (CH_2), 35.1, 35.2 (*C*Me$_3$), 35.5 (CH_2), 35.6, 35.8, 36.0, 36.5 (*C*Me$_3$), 37.6 (CH_2), 59.3 (DME), 60.6 (N*C*Me$_3$), 71.0 (CH), 73.1 (DME), 123.5, 123.8, 125.0, 125.2, 125.8, 126.2, 126.5, 126.5, 127.3, 127.6, 128.7, 129.8, 129.8, 130.0, 130.1, 130.2, 130.3, 131.0, 131.5, 131.7, 131.7, 132.9, 133.1, 135.5, 139.8, 140.1, 140.2, 141.1, 147.0 (Ar), 148.2 (NCH), 148.4, 154.9, 155.7, 157.8, 159.4, 162.3, 163.5 (Ar). IR (KBr, Nujol, cm^{-1}): 1139 (m). Anal. Calcd for $C_{81}H_{116}NNaO_{10}Ta_2$: C, 59.01; H, 7.09; N, 0.85. found: C, 58.78; H, 7.23; N, 0.86.

Formation of [{(OOCO)Ta}{(OOO)Ta}(μ-H)$_2$(CN*t*Bu)]$^-$ (3).

In a J. Young valve NMR tube, a THF-d_8 solution of **1-K** (12 mg, 7.6 μmol) was treated with *t*BuNC (2 μL, 18 μmol) at −30 °C. The mixture was stored at −30 °C for 12 h and then the NMR tube was inserted to the NMR probe precooled to −30 °C. According to NMR spectroscopy, formation of **3** as a sole product was noticed.

For **3**: ^{1}H NMR (500 MHz, THF-d_8, −30 °C): δ 0.18 (s, 9H, *t*Bu), 1.02 (s, 9H, *t*Bu), 1.12 (s, 9H, *t*Bu), 1.18 (s, 9H, *t*Bu), 1.322 (s, 9H, *t*Bu), 1.328 (s, 9H, *t*Bu), 1.393 (s, 9H, *t*Bu), 2.05 (s, 3H, *p*Me), 2.07 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 2.13 (s, 3H, *p*Me), 2.16 (s, 3H, *p*Me), 2.17 (s, 3H, *p*Me), 3.24 (s, 1H, CH), 3.32 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 3.41 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 4.05 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 4.47 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 4.57 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 6.07 (s, 1H, *m*H), 6.49 (s, 1H, *m*H), 6.59 (s, 2H, *m*H), 6.70 (s, 1H, *m*H), 6.71 (s, 1H, *m*H), 6.74 (s, 1H, *m*H), 6.75 (s, 1H, *m*H), 6.81 (s, 1H, *m*H), 7.04 (s, 1H, *m*H), 7.07 (s, 1H, *m*H), 7.35 (s, 1H, *m*H), 9.18 (d, $^2J_{HH}$ = 9 Hz, 1H, TaH), 10.87 (d, $^2J_{HH}$ = 9 Hz, 1H, TaH). ^{13}C NMR (126 MHz, THF-d_8, −30 °C): δ 21.1, 21.35, 21.38, 22.0 (*p*Me), 29.7, 30.8, 31.1, 32.1, 32.5, 32.7 (C*Me$_3$*), 32.8 (CH$_2$), 33.5 (C*Me$_3$*), 33.8 (CH$_2$), 35.2, 35.5, 35.6, 35.8, 36.4, 36.5 (*C*Me$_3$), 37.3 (CH$_2$), 74.6 (N*C*Me$_3$), 78.3 (CH), 123.1, 123.7, 124.3, 124.9, 125.1, 125.2, 126.1, 126.4, 126.5, 127.1, 127.8, 128.1, 129.3, 129.8, 129.9, 130.6, 130.8, 130.9, 131.3, 132.7, 133.6, 134.7, 136.2, 137.0, 139.0, 140.86, 140.92, 144.4, 150.0, 150.1, 160.7, 161.0, 162.0, 162.8, 163.0, 163.1, 164.8 (Ar + *C*N*t*Bu).

Synthesis of [{(OOCO)Ta}{(OOO)Ta}(μ-η^1:η^2-CN*t*Bu)][K(18-crown-6)(THF)] (4-K).

To a THF (5 mL) solution of **2-K** (145 mg, 87.1 μmol) at room temperature was added 18-crown-6 (30 mg, 0.11 mmol) with stirring. Slow effervescence occurred, the solution changing from orange to dark yellow within 10 minutes. The mixture was layered with hexane to give **4-K** as dark brown crystals (147 mg, 80.8 μmol, 93%).

^{1}H NMR (396 MHz, THF-d_8): δ 0.88 (s, 9H, *t*Bu), 1.02 (s, 9H, *t*Bu), 1.21 (s, 18H, *t*Bu), 1.25 (s, 9H, *t*Bu), 1.34 (s, 9H, *t*Bu), 1.35 (s, 9H, *t*Bu), 2.07 (s, 3H, *p*Me), 2.09 (s, 3H, *p*Me), 2.12 (s, 3H, *p*Me), 2.15 (s, 3H, *p*Me), 3.01 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 3.04 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 3.40 (s, 1H, CH), 3.42 (s, 24H, 18-crown-6), 3.48 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 4.48 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 5.07 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 5.27 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 6.21 (s, 1H, *m*H), 6.50 (s,

1H, *m*H), 6.62 (s, 1H, *m*H), 6.63 (s, 1H, *m*H), 6.66 (s, 1H, *m*H), 6.68 (s, 1H, *m*H), 6.75 (s, 1H, *m*H), 6.77 (s, 1H, *m*H), 6.78 (s, 1H, *m*H), 6.98 (s, 1H, *m*H), 7.04 (s, 1H, *m*H), 7.06 (s, 1H, *m*H). ^{13}C NMR (99 MHz, THF-d_8): δ 21.4, 21.5, 21.5, 21.7 (*p*Me), 31.3, 32.0, 32.1, 32.2 32.5, 33.1, 33.9 (C*Me*$_3$), 35.0, 35.4, 35.4, 35.5, 35.8, 36.2 (*C*Me$_3$), 37.2 , 37.4, 38.1 (CH$_2$), 63.9 (N*C*Me$_3$), 65.9 (CH), 123.6, 123.7, 123.8, 124.5, 124.69, 124.71, 124.9, 124.9, 125.6, 125.6, 126.16, 126.21, 129.7, 130.2, 130.6, 130.93, 130.93, 130.95, 130.0, 130.18, 130.24, 131.6, 132.7, 136.7, 139.0, 139.3, 140.2, 141.5, 142.4, 148.5, 151.6, 161.5, 163.7, 164.8, 165.2, 165.7 (Ar), 247.9 (Ta=C). IR (KBr, Nujol, cm^{-1}): 1105 (m). Anal. Calcd for $C_{89}H_{126}KNO_{13}Ta_2$: C, 58.77; H, 6.98; N, 0.77. found: C, 58.85; H, 7.28; N, 0.89.

Reaction of [{(OOCO)Ta}{(OOO)Ta}(μ-η^1:η^2-CN*t*Bu)][Na(18-crown-6)(THF)$_2$] (4-Na).

The same procedure as used for **4-K** was followed. Addition of 18-crown-6 (40 mg, 0.15 mmol) to THF (5 mL) solution of **2-Na** (100 mg, 60.7 μmol) afforded **4-Na** as dark brown crystals (102 mg, 52.4 μmol, 86%).

^{1}H NMR (396 MHz, THF-d_8): δ 0.88 (s, 9H, *t*Bu), 1.02 (s, 9H, *t*Bu), 1.21 (s, 18H, *t*Bu), 1.25 (s, 9H, *t*Bu), 1.34 (s, 9H, *t*Bu), 1.35 (s, 9H, *t*Bu), 2.07 (s, 3H, *p*Me), 2.09 (s, 3H, *p*Me), 2.12 (s, 3H, *p*Me), 2.15 (s, 3H, *p*Me), 3.01 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 3.04 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 3.40 (s, 25H, 18-crown-6 + CH), 3.48 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 4.48 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 5.07 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 5.27 (d, $^2J_{HH}$ = 13 Hz, 1H, CH$_2$), 6.21 (s, 1H, *m*H), 6.50 (s, 1H, *m*H), 6.62 (s, 1H, *m*H), 6.63 (s, 1H, *m*H), 6.66 (s, 1H, *m*H), 6.68 (s, 1H, *m*H), 6.75 (s, 1H, *m*H), 6.77 (s, 1H, *m*H), 6.78 (s, 1H, *m*H), 6.98 (s, 1H, *m*H), 7.04 (s, 1H, *m*H), 7.06 (s, 1H, *m*H). ^{13}C NMR (99 MHz, THF-d_8): δ 21.4, 21.5, 21.5, 21.7 (*p*Me), 31.3, 32.0, 32.1, 32.2 32.5, 33.1, 33.9 (C*Me*$_3$), 35.0, 35.38, 35.39, 35.5, 35.8, 36.2 (*C*Me$_3$), 37.2 , 37.4, 38.1 (CH$_2$), 63.9 (N*C*Me$_3$), 65.9 (CH), 123.6, 123.7, 123.8, 124.5, 124.70, 124.76, 124.92, 124.97, 125.6, 125.6, 126.15, 126.22, 129.7, 130.2, 130.6, 130.92, 130.92, 130.95, 130.0, 130.18, 130.25, 131.6, 132.7, 136.7, 139.0, 139.3, 140.2, 141.5, 142.4, 148.4, 151.6, 161.5, 163.7, 164.8, 165.2, 165.7 (Ar), 247.9 (Ta=C). Anal. Calcd for $C_{93}H_{134}NNaO_{14}Ta_2$: C, 59.57; H, 7.20; N, 0.75. found: C, 59.69; H, 7.10; N, 0.87.

Synthesis of [{(OOCO)Ta}{(OOO)Ta}(μ-η^2:η^2-tBuNCCHN*t*Bu)(η^2-HN*C*tBu)][K(THF)] (5-K).

To a toluene (10 mL) solution of **2a** (473 mg, 0.284 mmol) at room temperature was added dropwise *t*BuNC (100 μL, 0.88 mmol). The mixture was stirred for 24 h at room temperature. After removal of volatiles under vacuum, the residue was extracted into pentane (2 mL) and filtrated. A drop of THF was added to the filtrate. The resulting filtrate was left to stand at room temperature for 1 day, after which pale yellow crystal of **5-K**·2pentane deposited (327 mg, 0.175 mmol, 62%). Prolong drying under vacuum resulted in partial loss of pentane solvates, giving **5-K**·pentane.

^{1}H NMR (400 MHz, C_6D_6): δ 0.10 (s, 9H, *t*Bu), 0.87 (t, $^3J_{HH}$ = 7 Hz, 6H, pentane), 0.98 (s, 9H, *t*Bu), 1.23 (m, 6H, penatne), 1.36 (s, 9H, *t*Bu), 1.38 (s, 9H, *t*Bu), 1.40 (m, 4H, THF), 1.45(s, 9H, *t*Bu), 1.46 (s, 9H, *t*Bu), 1.56 (s, 9H, *t*Bu), 1.59 (s, 9H, *t*Bu), 1.80 (s, 9H, *t*Bu), 2.12 (s, 3H, *p*Me), 2.23 (s, 3H, *p*Me), 2.27 (s, 3H, *p*Me), 2.41 (s, 3H, *p*Me), 3.10 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 3.30 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 3.55 (m, 4H, THF), 3.58 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 3.86 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 4.18 (s, 1H, CH), 4.32 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 4.75 (d, $^2J_{HH}$ = 13 Hz, 1H, CH_2), 5.57 (s, 1H, NCH), 6.84 (s, 1H, *m*H), 6.85 (s, 2H, *m*H), 6.91 (s, 1H, *m*H), 6.93 (s, 1H, *m*H), 6.95 (s, 1H, *m*H), 7.02 (s, 1H, *m*H), 7.08 (s, 1H, *m*H), 7.55 (s, 1H, *m*H), 7.57 (s, 1H, *m*H), 10.81 (s, 1H, N=CH). Two of the signals of the *m*H of the phenyl rings are overlapped in the residual signal of C_6D_6. ^{13}C NMR (100 MHz, C_6D_6): δ 20.66, 20.68, 20.8, 21.4 (*p*Me), 28.6, 29.9, 30.8, 31.1, 31.3, 31.9, 32.1, 32.2, 33.1(C*Me*$_3$), 33.2 (CH_2), 34.4, 34.6, 34.7, 35.3, 35.4 (*C*Me_3), 35.7 (CH_2), 36.1 (*C*Me_3), 37.3 (CH_2), 59.8 (N*C*Me_3), 61.1 (N*C*Me_3), 61.3 (N*C*H), 63.5 (N*C*Me_3), 64.7 (CH), 124.0, 124.5, 124.7, 125.0, 125.4, 125.9, 126.4, 126.8, 127.1, 128.3, 128.46, 128.51, 128.53, 129.2, 129.6, 129.7, 129.9, 130.1, 130.5, 130.7, 130.7, 132.4, 132.7, 133.7, 135.3, 137.9, 138.3, 140.7, 142.3, 143.1, 144.9, 150.2, 159.9, 160.1, 160.9, 161.0 (Ar), 217.9 (N=CH), 233.4(N=C). IR (KBr, Nujol, cm^{-1}): 1574 (m), 1132 (m), 819 (s). Anal. Calcd for $C_{92}H_{134}KN_3O_7Ta_2$: C, 61.56; H, 7.52; N, 2.34. found: C, 61.67; H, 7.77; N, 2.35.

X-ray Crystallography

Crystallographic data of **2-Na**, **4-Na**, and **5-K** are summarized in Table S1. Single crystals of **2-Na**, **4-Na**, and **5-K** were obtained from DME/hexane, THF/hexane, and THF/pentane, respectively. Crystals of these complexes were immersed in mineral oil on nylon loop and transferred to a Rigaku Saturn CCD system (for **4-Na**) or a Rigaku Mercury CCD system (for **2-Na**

and **5-K)** equipped with a Rigaku GNNP low-temperature device. Data were collected under cold nitrogen stream (123 K) using graphite-monochromated Mo-Kα radiation (λ = 0.7107 Å). Equivalent reflections were merged, and the images were processed with the CrystalClear (Rigaku) Program. Corrections for Lorentz-polarization effects and absorption were performed.

All calculations were performed using SHELXS[1] and SHELXL[2]. The structures were solved by Patterson and Fourier transform methods. For **4-Na** and **5-K**, the methylene carbons of the THF molecules coordinated to Na and K were disordered over two positions and refined isotropically. For **2-Na**, two sites occupied by hexane were identified in the unit cell. These were considerably disordered and were treated by SQUEEZE as a diffuse contribution.[4,5] In the resulting void space, a contribution of 134 e^- per unit cell was found and taken to represent 0.5 hexane molecules in the asymmetric unit. For **4-Na**, three sites occupied by THF were identified in the asymmetric unit. Two sites were ordered and fully occupied. The third site was considerably disordered and was treated by SQUEEZE again. In the resulting void space, a contribution of 51 e^- per unit cell was found and taken to represent 0.5 THF molecules for each Ta_2 complex, giving a total of 2.5THF in the asymmetric unit. Crystals of **5-K** contained two pentane molecules per asymmetric unit, and these solvate molecules were ordered and fully occupied. All solvate molecules of **4-Na** and **5-K** were refined isotropically. The remaining non-hydrogen atoms were refined anisotropycally. The bridging hydride in **2-Na** was found in the Fourier map and refined with isotropic thermal parameters. The other hydrogen atoms were included in the model at geometrically calculated positions and refined using a riding model.

References

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Table S1. Crystallographic Data for **2-Na**, **4-Na**, and **5-K**.

	2-Na	**4-Na**	**5-K**
formula	$C_{84}H_{123}NNaO_{10}Ta_2$	$C_{103}H_{155}NNaO_{16.5}Ta_2$	$C_{97}H_{146}KN_3O_7Ta_2$
M	1691.72	2056.17	1867.17
T/K	123(2)	123(2)	123(2)
color	orange	black	colorless
crystal size/mm^3	0.35 x 0.27 x 0.15	0.17 x 0.06 x 0.05	0.15 x 0.12 x 0.11
crystal system	Monoclinic	Triclinic	Triclinic
space group	$P2/a$ (no. 13)	P-1 (no. 2)	P-1 (no. 2)
a/Å	22.166(3)	15.8296(18)	16.441(5)
b/Å	13.5778(11)	16.0469(18)	17.510(5)
c/Å	29.333(4)	23.802(3)	17.751(5)
α/°	90	95.3351(8)	99.9870(15)
β/°	103.516(6)	103.7628(8)	95.320(3)
γ/°	90	112.4524(15)	109.979(3)
V/Å^3	8583.7(18)	5312.2(10)	4666(2)
Z	4	2	2
D_c/g cm^{-3}	1.306	1.285	1.329
μ/mm^{-1}	2.605	2.122	2.441
reflections collected	86847	66441	37406
independent reflections (R_{int})	19555 (0.0377)	24217 (0.0364)	20495 (0.0356)
refined parameters	891	1065	956
goodness-of-fit on F^2	1.089	1.081	1.106
$R1$ [$I > 2\sigma(I)$][a]	0.0350	0.0507	0.0508
wR_2 (all data)[b]	0.1078	0.1365	0.0816
largest diff. peak and hole/e Å^{-3}	2.374 and −1.214	3.803 and −1.905	1.306 and −0.903

[a] $R1 = \Sigma||F_o| - |F_c|| / \Sigma|F_o|$, [b] $wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma[w(F_o^2)^2]]^{0.5}$