Experimental Details

General Procedure: All reactions were performed using oven-dried glassware under an atmosphere of dry argon. All reagents were purchased from Aldrich and used without further purification. Anhydrous THF was purchased from Aldrich and used without further drying. Chromatographic purification was run using Silicycle Silica Gel. NMR spectra were recorded on a Varian 300 operating at 300 MHz and 75 MHz for ^{1}H and ^{13}C respectively and referenced to internal standards. Diastereomeric ratios and extent of reaction were determined by comparison of total ion counts, using an Agilent Technologies Model 6890 gas chromatograph equipped with an Agilent Technologies Model 5773 mass spectrophotometer. The column used was a 30 m x 0.25 mm, 0.25 um film thickness, HP1 column with 1 mL/min flow rate. Combustion analysis was performed by Quantitative Technologies, Inc. and HRMS was performed by M-Scan Analysis.

Scheme 2

Procedure for the Addition of TMS-CN to (*R*)-Camphor (1).

2-Cyano-2-trimethylsilanyloxy-1,3,3-trimethyl-[2.2.1]-bicycloheptane (Characterized as a 96:4 Mixture of Major:Minor* Diastereomers) (2). A 500 mL, 2-neck, round bottom flask equipped with an argon inlet and a magnetic stir-bar was charged with 475 mg (12.5 mmol, 0.05 equiv) of lithium methoxide, 420 mL of anhydrous THF, and 25 g (0.25 mol, 1.2 equiv) of trimethylsilylcyanide. The resulting clear yellow solution was stirred at 22°C for 10 minutes and 32 g (0.21 mol) of (*R*)-(+)-camphor was added. The reaction mixture was stirred at rt until all the starting material was consumed. After completion, the reaction was quenched with 10% Na_2CO_3, and extracted with *tert*-butyl methyl ether. The combined organic layers were concentrated *in vacuo* to afford a crude liquid. The crude product was diluted with 400 mL of hexanes and washed once with 50 mL of acetonitrile. The hexane layer was separated, and concentrated to afford 50.0 g (95%) of the desired product as a colorless liquid. The diastereomeric excess was determined by GC. The temperature program used was 110/2/5/160/20 - retention times: Major 12.01, minor 12.15 min. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.22 (s, 9H), 0.88 (s, 3H), 1.09 (m, 1H), 1.20 (s, 6H), 1.36-1.43 (m, 1H), 1.60-1.90 (m, 4H), 2.08 (d, 1H, *J* = 14.1 Hz) 2.21 (ddd, 1H, *J* = 14.1, 3.5, 3.2 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 1.34, 18.07, 22.17, 25.95, 26.66, 29.73, 40.55, 43.66, 48.68, 54.41, 83.75, 120.18. MS (EI): *m/z* = 251 (M+). Anal. Calcd for $C_{14}H_{25}NOSi$: C, 66.87; H, 10.02; N, 5.57. Found: C, 66.84; H, 9.97; N, 5.48.

Procedure for the Synthesis of 2-aminomethyl-2-hydroxy-1,3,3-trimethyl-[2.2.1]-bicycloheptane HCl Salt (3). A 500 mL, 2-neck, round bottom flask equipped with an argon inlet and a magnetic stir-bar was charged with 25.1 g (100 mmol, 1.0 equiv) of 2-cyano-2-trimethylsilanyloxy-1,3,3-trimethyl-[2.2.1]-bicycloheptane **2**, 200 mL of anhydrous toluene, and 72 mL (0.24 mol, 2.4 equiv) of REDAL-H. The resulting solution was stirred at 70°C for 2 hours and at room temperature for 16 h. The reaction was quenched with 5N NaOH (100 mL x 2) and the toluene layer was washed with brine (100 mL). The organic layers were concentrated *in vacuo* to afford 22.1 g of the crude free-base as an oil. A 250 mL, 2-neck, round bottom flask equipped with an argon inlet and a magnetic stir-bar was charged with 1.6 g (100 mmol, 1.0 equiv) of 2-aminomethyl-2-hydroxy-1,3,3-trimethyl-[2.2.1]-bicycloheptane **2**, 20 mL of anhydrous toluene, and 50 mL (50 mmol, 5 equiv) of 1N HCl in ether. Methanol (25 mL) was added to the resulting gel and the mixture was stirred at reflux until a clear solution was obtained. The reaction mixture was stirred at room temperature for 16 h. The slurry was filtered, and the solid washed with toluene. The isolated solid was dried *in vacuo* to yield 1.3 g of the desired product as a white solid. ^{1}H NMR (300 MHz, CD_3OD): δ 0.88 (s, 9H), 0.92 (s, 3H), 1.09 (m, 1H), 1.15 (s, 3H), 1.43 (m, 3H), 1.75 (m, 2H), 1.91 (ddd, 1H, *J* = 13.1, 3.5, 3.5 Hz), 2.68 (dd, 1H, *J* = 17.3, 12.9 Hz), 4.9 (br s, 4H). ^{13}C NMR (75 MHz, CD_3OD): δ 11.64, 21.23, 22.04, 28.03, 31.14, 44.90, 46.47, 50.24, 50.86, 52.63, 81.65. Anal. Calcd for $C_{14}H_{25}NOSi$: C, 72.09; H, 11.55; N, 7.64. Found: C, 71.97; H, 11.63; N; 7.57.

Oxazolidinone (4)

A 100 mL, 2-neck, round bottom flask equipped with an argon inlet and a magnetic stir-bar was charged with 4 g (24 mmol, 1.0 equiv) of 2-cyano-2-trimethylsilanyloxy-1,3,3-trimethyl-[2.2.1]-bicycloheptane **2**, 4 g (24 mmol, 1.0 equiv) of carbodimidazole and 30 mL of anhydrous THF. The resulting solution was stirred at 70°C for 3 hours and at room temperature for 16 h. The reaction was diluted with 80 mL of ethyl

acetate and washed with 1N HCl (50 mL x 3), 0.1 N K_2CO_3 (50 mL) and brine (50 mL). The organic layers were concentrated *in vacuo* to afford 2.1 g of the crude product. The crude product was purified by silica gel chromatography (eluant 2:1 Hex:EtOAc) to yield the pure compound as a mix of diastereomers. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.79 (s, 3H), 0.81 (s, 3H), 1.09 (m, 2H), 1.49 (m, 1H), 1.60 (d, 1H, *J* = 13.8 Hz), 1.72 (m, 2H), 2.40 (m, 1H), 3.25 (d, 1H, *J* = 8.3 Hz), 3.70 (d, 1H, *J* = 8.3 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 10.13, 20.43, 20.54, 26.96, 29.36, 45.42, 47.46, 49.35, 49.92, 51.88, 91.64, 160.20.
NMR analysis (Hetcor, NOESY, COSEY indicated the major isomer was the endo addition product). The purified compound was crystallized from MeOH to give single crystals for X-ray analysis. X-ray data confirmed the stereochemistry (see pages 5-23).

Table 2:
Entry 2
General Procedure for the Addition of TMS-CN to Hindered Ketones using LiTGMM.
(1*R*)-2-Cyano-6,6-dimethyl-2-trimethylsilanyloxy-bicyclo[3.1.1]heptane. A 25mL, 2-neck, round bottom flask equipped with an argon inlet and a magnetic stir-bar was charged with 7.14 mL of anhydrous THF, 7.14 µL (5 mol%) of tri(ethylene glycol) monomethyl ether, 111 µL (0.18 mmol, 0.05 equiv) of 1.6 M *n*-butyllithium solution in hexanes, and 0.57 mL (4.28 mmol, 1.2 equiv) of trimethylsilylcyanide. The resulting clear yellow solution was stirred at 22°C for 30 minutes. The solution was cooled to 0°C and 400 mg (3.57 mmol) of (*R*)-(+)-nopinone was added. The reaction mixture was stirred at 22°C until all the starting material was consumed. After completion, the reaction was quenched with 10% Na_2CO_3, and extracted twice with *tert*-butyl methyl ether. The combined organic layers were concentrated *in vacuo* to afford a crude liquid. The crude product was diluted with 20 mL of hexanes and washed once with 2 mL of acetonitrile. The hexane layer was separated, dried over anhydrous $MgSO_4$, filtered and concentrated to afford 650 mg (95%) of the desired product as a single isomer. ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.26 (s, 9H), 1.04 (s, 3H), 1.27 (s, 3H), 1.29 (d, 1H, *J* = 10.2 Hz), 1.84 to 2.66 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 1.5, 22.7, 24.3, 27.5, 28.0, 33.1, 38.0, 40.4, 51.8, 74.6, 123.8; MS (EI): *m/z* = 237 (M^+); Anal. Calcd for $C_{13}H_{23}NOSi$: C, 65.77; H, 9.76; N, 5.90. Found: C, 65.42; H, 9.63; N, 5.70.

Entry 1
2-Cyano-2-trimethylsilanyloxy-1,7,7-trimethyl-[2.2.1]-bicycloheptane (Characterized as a 93:7 Mixture of Major:Minor* Diastereomers). ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.25 (s, 9H), 0.90 (s, 3H), 0.98 (s, 3H), 1.01 (s, 3H), 1.10-1.23 (m, 1H), 1.61-1.86 (m, 4H), 2.08 (d, 1H, *J* = 14.1 Hz) 2.21 (ddd, 1H, *J* = 14.1, 3.4, 3.0 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 1.04, 10.56, 20.46, 21.12, 26.51, 31.67, 45.16, 47.84, 48.70, 54.06, 78.51, 121.90. MS (EI): *m/z* = 251 (M+). Anal. Calcd for $C_{14}H_{25}NOSi$: C, 66.87; H, 10.02; N, 5.57. Found: C, 66.84; H, 9.97; N, 5.48.

Entry 3
1-Cyano-1-trimethylsilanyloxy-2-methyl-cyclohexane (Characterized as a 77:23 Mixture of Major:Minor* Diastereomers). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.23 and 0.24* (s, 9H), 1.03* and 1.07 (d, 3H, *J* = 6.6* and 6.6 Hz), 1.15 to 2.19 (m, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 1.1*, 1.5, 16.4, 20.2*, 23.8, 24.5*, 24.9, 28.3*, 31.6, 38.2*, 39.8, 40.8*, 43.2, 71.6*, 76.0, 120.1, 122.5*. MS (EI): *m/z* = 211(M^+), 196, 169; Anal. Calcd for $C_{11}H_{21}NOSi$: C, 62.50; H, 10.01; N, 6.63. Found: C, 62.10; H, 10.01; N, 6.27.

Entry 4
1-Cyano-1-trimethylsilanyloxy –(3*R*)-methylcyclohexane (Characterized as a 85:15 Mixture of Major:Minor* Diastereomers). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.25 and 0.26* (s, 9H), 0.74 to 0.90 (m, 1H), 0.90* and 0.96 (d, 3H, *J* = 6.6* and 6.3 Hz), 1.16 to 2.19 (m, 8H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 1.3*, 1.7, 20.2*, 21.9, 22.0*, 23.0, 26.5*, 30.3, 33.4, 33.6*, 37.8*, 39.4, 46.3*, 48.0, 68.2*, 71.8, 122.0, 123.1*. MS (EI): *m/z* = 211 (M^+), 196, 169. Anal. Calcd for $C_{11}H_{21}NOSi$: C, 62.50; H, 10.01; N, 6.63. Found: C, 62.80; H, 9.89; N, 6.60.

Entry 5
1-Cyano-1-trimethylsilanyloxy-2-methylcyclopentane (Characterized as a 82:18 Mixture of Major:Minor* Diastereomers). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.23 (s, 9H), 1.06* and 1.12 (d, 3H, *J* = 6.6* and 6.9 Hz), 1.29 to 1.44 (m, 1H), 1.73 to 2.29 (m, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 1.0*, 1.2, 12.7*, 15.6, 20.1,

21.4*, 29.5, 30.2*, 39.9, 40.7*, 46.5, 46.9*, 75.8*, 79.8, 120.9, 122.2*. MS (EI): *m/z* = 197 (M^+), 182, 169, 155.

Entry 6

1-Trimethylsilanyloxy-1-cyano-2-methyl-5-isopropenylcylohexene (Characterized as a 97:3 Mixture of Major:Minor* Diastereomers). ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.35 (s, 9H), 1.76 (s, 3H), 1.84 (s, 3H), 1.98 (m, 1H), 2.20 (m, 1H), 2.38 (m, 1H), 2.57 (m, 1H), 4.81 (dd, 2H, *J* =12.4, 1.0 Hz), 5.63 (dd, 1H, *J* = 4.3, 2.1 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 1.32, 17.43, 20.49, 25.63, 26.96, 30.65, 39.18, 42.37, 67.91, 110.03, 121.16, 127.01, 133.77, 147.05. MS (EI): m/z = 249 (M+).

Entry 7

Ethyl 3-trimethylsilanyloxy-3-cyanobutanoate.

NOTE: It was found ethyl 3-trimethylsilanyloxy-3-cyanopentanoate desilated during work-up if base was used to quench the reaction. A modified procedure was run for this compound.

A 25mL, 2-neck, round bottom flask equipped with an argon inlet and a magnetic stir-bar was charged with 16 mL of anhydrous THF, 380 μL (5 mol%) of LiOEt (1 M in THF), and 0.95 g (9.57 mmol, 1.2 equiv) of trimethylsilylcyanide. The resulting clear yellow solution was stirred at 22°C for 30 minutes and 1.0 g (7.68 mmol) of ethyl acetoacetate was added. The reaction mixture was stirred at 22°C until all the starting material was consumed. After completion, the reaction was quenched with 10 mL of water, and extracted with 30 mL of *tert*-butyl methyl ether. The organic layer was washed with water (1x10 mL), dried (Na_2SO_4), and concentrated *in vacuo* to afford the desired product as yellow oil (1.70 g, 96.5 % yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.30 (s, 9H 1.33 (t, J=7.0 Hz, 3H), 1.75 (s, 3H), 2.79 (s, 1H), 2.81 (s, 1H), 4.25 (q, 2H, J= 7.0 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 1.15, 14.13, 29.15, 47.43, 60.97, 66.62, 121.03, 167.93. MS (EI): m/z = (M+).

Entry 8

Cyano-cyclohexyl-trimethylsilanyloxy-acetic acid ethyl ester. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.26 (s, 9H), 1.24 (m, 5H), 1.38 (m, 5H), 1.38 (t, 3H, *J* = 7.1 Hz), 1.56-1.98 (m, 6H), 4.32 (q, 2H, *J* = 7.1 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 0.81, 14.29, 25.90, 25.97, 26.44, 27.11, 47.07, 63.08, 118.07, 168.11. MS (EI): *m/z* = 283 (M+).

Entry 9

Cyclohexyl-phenyl-trimethylsilanyloxy-acetonitrile. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.12 (s, 9H), 1.17 (m, 5H), 1.40 (m, 1H), 1.73 (m, 4H), 2.05 (m, 1H), 7.40 (m, 3H), 7.49 (m, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): δ 1.05, 26.12, 27.45, 27.54, 50.85, 79.75, 120.43, 126.01, 128.42, 128.66, 140.36. MS (EI): *m/z* = 287.

Entry 10

2-Phenyl-2-trimethylsilanyloxy-propionitrile. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.17 (s, 9H), 1.85 (s, 3H), 7.40 (m, 3H), 7.54 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$): δ -3.39, 29.11, 67.14, 117.17, 120.14, 128.18, 137.51. MS (EI): *m/z* = 219 (M+). Anal. Calcd for $C_{12}H_{17}NOSi$: C, 65.61; H, 7.81; N, 6.39. Found: C, 65.20; H, 7.54; N, 5.99.

Entry 11

1-(4-Aminophenyl)-1-trimethylsilanyloxy-1-cyanoethane. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.15 (s, 9H), 1.85 (s, 3H), 3.75 (br s, 2H), 6.67 (d, 2H, *J* = 9.0 Hz), 7.32 (d, 2H, *J* = 9.0 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ 1.20, 33.20, 71.50, 114.80, 122.14, 126.05, 131.40, 147.03. MS (EI): m/z =234 (M+) 219, 120. Anal. Calcd for $C_{12}H_{18}N_2OSi$: C,61.50; H,7.74; N,11.95. Found: C, 61.45; H, 7.67; N; 11.83.

Entry 12

Phenyl-trimethylsilanyloxy-acetonitrile. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.22 (s, 9H), 5.49 (s, 1H), 7.38 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$): δ -4.69, 59.20, 114.79, 121.92, 124.50, 124.90, 131.87. MS (EI): *m/z* = 205 (M+).

Entry 13

(+)-O-Trimethylsilyl-dehydro-isoandrosterone cyanohydrin. ^{1}H NMR (300 MHz, $CDCl_3$): δ 0.15 (s, 9H), 0.26 (s, 9H), 0.81 (s, 3H), 1.03 (s, 3H), 1.13 (m, 2H), 1.51-1.99 (m, 14H), 2.17-2.47 (m, 3H), 3.50 (m, 1H), 5.33 (d, 1H, *J* = 5.2 Hz). ^{13}C NMR (75 MHz, $CDCl_3$): δ -4.24, -3.25, 7.60, 14.92, 16.12, 18.91, 26.83, 27.40, 28.01, 28.58, 32.06, 32.84, 33.48, 38.12, 43.10, 45.07, 67.64, 77.19, 116.19, 117.88, 136.91. MS (EI): *m/z* = 459 (M+). Anal. Calcd for $C_{26}H_{45}NO_2Si_2$: C, 67.91; H, 9.86; N, 3.05. Found: C, 67.65; H, 9.99; N; 2.95.

Table 3

Entry 1

2--(*tert*-Butyl-dimethylsilanyloxy)-2-cyano-1,3,3-trimethyl-[2.2.1]-bicycloheptane (Characterized as a 88:12 Mixture of Major:Minor* Diastereomers). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.26 (s, 3H), 0.28 (s, 3H), 0.86 (m, 1H), 0.95 (s, 9H), 0.97 (s, 3H), 1.07 (s, 3H), 1.15 (s, 3H), 1.30-1.42 (m, 1H), 1.65-1.75 (m, 2H), 2.18 (m, 1H), 2.61 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ -3.86, -3.17, 18.36, 21.17, 21.40, 25.85, 27.22, 27.76, 45.33, 45.92, 48.83, 55.83, 121.9. MS (EI): *m/z* = 293 (M+), 236, 209.

Entry 2

1-(*tert*-Butyl-dimethyl-silanyloxy)-1-cyano-2-methylcyclohexane (Characterized as a 93:7 Mixture of Major:Minor* Diastereomers). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.22 and 0.25* (s, 3H), 0.28 and 0.29* (s, 3H), 0.92 and 0.95* (s, 9H), 1.08* and (d, 3H, *J* = 6.6* and 6.6 Hz), 1.89 to 2.22 (m, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ -3.8, -2.7, 16.6, 18.2, 23.9, 25.0, 25.7, 31.7, 39.8, 43.5, 76.2, 120.4. MS (EI): *m/z* = 253 (M^+), 238, 196, 169.

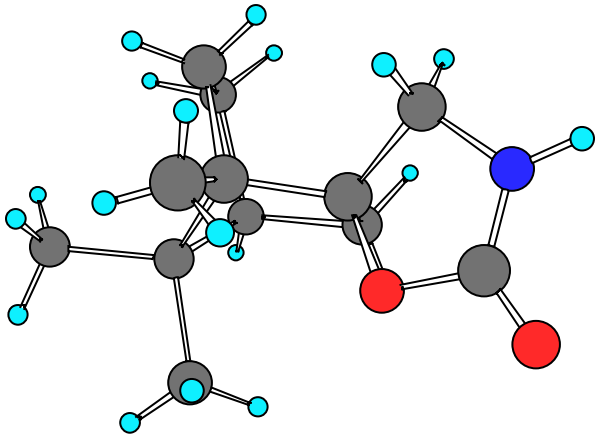
Entry 3

1-(*tert*-Butyl-dimethylsilanyloxy)-1-cyano-(3*R*)-methylcyclohexane (Characterized as a 96:4 Mixture of Major:Minor* Diastereomers). ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.22 (s, 3H), 0.23 (s, 3H), 0.86 (m, 1H), 0.88 (s, 9H), 0.95 (d, 3H, *J* = 6.3 Hz), 1.13 to 2.16 (m, 8H). ^{13}C NMR (75 MHz, $CDCl_3$) δ -3.23, -3.17, 18.0, 21.8, 23.0, 25.6, 30.2, 33.4, 39.4, 47.9, 71.6, 121.9. MS (EI): *m/z* = 253 (M+), 196, 169.

Entry 4

Cyclohexylphenyl-*tert*-butyl-dimethylsilanyloxy acetonitrile. ^{1}H NMR (300 MHz, $CDCl_3$): δ –0.18 (s, 3H), 0.19 (s, 3H), 0.98 (s, 9H), 1.17 (m, 5H), 1.80 (m, 3H),), 2.05 (m, 1H), 7.40 (m, 3H), 7.53 (m, 2H). ^{13}C NMR (300 MHz, $CDCl_3$): δ -3.95, -3.58, 18.64, 25.97, 26.14, 27.58, 27.65, 51.03, 80.03, 120.30, 126.13, 128.39, 128.74, 140.27. Anal. Calcd for $C_{20}H_{31}NOSi$: C, 72.89; H, 9.48; N, 4.25. Found: C, 72.77; H, 9.31; N, 4.18.

X-RayData



4

Data Collection

A colorless needle crystal of $C_{12}H_{19}O_2N$, having approximate dimensions of 0.15 x 0.15 x 0.25 mm was mounted on a glass fiber. All measurements were made on a Rigaku AFC5R diffractometer with graphite monochromated Cu-Ka radiation and a rotating anode generator.

Cell constants and an orientation matrix for data collection obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections in the range $63.50 < 2q < 79.49^o$, corresponded to a primitive orthorhombic cell with dimensions:

a = 15.090(1) Å
b = 11.3613(9) Å
c = 13.549(2) Å
V = 2322.8(3) $Å^3$

For Z = 8 and F.W. = 209.29, the calculated density is 1.20 g/cm^3. The systematic absences of:

h00: h ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_12_12$ (#18)

The data were collected at a temperature of 23 ± 1^oC using the w-2q scan technique to a maximum 2q value of 127.0^o. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.32^o with a take-off angle of 6.0^o. Scans of $(1.73 + 0.35 \tan q)^o$ were made at a speed of 16.0^o/min (in w). The weak reflections (I < 15.0s(I)) were re-scanned (maximum of 6 scans) and the counts were accumulated to ensure good counting statistics. Stationary background counts were recorded on each side of the reflection. The ratio of peak counting time to background counting time was 2:1. The diameter of the incident beam collimator was 1.0 mm, the crystal to detector distance was 285 mm, and the detector aperture was 6.0 x 6.0 mm (horizontal x vertical).

Data Reduction

A total of 2171 reflections was collected. The intensities of three representative reflections were measured after every 150 reflections. Over the course of data collection, the standards increased by 0.0%. A linear correction factor was applied to the data to account for this phenomenon.

The linear absorption coefficient, m, for Cu-Ka radiation is 6.5 cm^{-1}. An empirical absorption correction based on azimuthal scans of several reflections was applied, which resulted in transmission factors ranging from 0.96 to 1.00. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction was applied (coefficient = 2.35896e-006).

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included, but not refined. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 1415 observed reflections and 272 variable parameters and converged (largest parameter shift was 0.03 times its esd) with unweighted and weighted agreement factors of:

$$R1 = S \, ||Fo| - |Fc|| \, / \, S \, |Fo| = 0.051$$

$$wR2 = [\, S \, (\, w \, (Fo^2 - Fc^2)^2 \,) / \, S \, w(Fo^2)^2]^{1/2} = 0.175$$

The standard deviation of an observation of unit weight[4] was 1.24. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.20 and -0.23 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Df' and Df'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the teXsan[9] crystallographic software package of Molecular Structure Corporation except for refinement, which was performed using SHELXL-97[10].

References

(1) SIR97: Altomare, A.; Cascarano, M.; Giacovazzo, C.; Guagliardi, A. (**1993**). *J. Appl. Cryst*., *26*, 343.

(2) DIRDIF94: Beurskens, P.T.; Admiraal, G.; Beurskens, G.; Bosman, W.P.; de Gelder, R.; Israel, R. and Smits, J.M.M.(1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized: (SHELXL97)

$$Sw(F_o^2 - F_c^2)^2 \text{ where}$$

$$w = 1/ [\, s^2(Fo^2) + (0.1000 \cdot P)^2 + 0.0000 \cdot P \,]$$
$$P = (Max(Fo^2,0) + 2Fc^2)/3$$

(4) Standard deviation of an observation of unit weight:

$$[S w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

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EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{12}H_{19}O_2N$
Formula Weight	209.29
Crystal Color, Habit	colorless, needle
Crystal Dimensions	0.15 X 0.15 X 0.25 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2q range)	25 (63.5 - 79.5^o)
Omega Scan Peak Width at Half-height	0.32^o
Lattice Parameters	a = 15.090(1) Å b = 11.3613(9) Å c = 13.549(2) Å V = 2322.8(3) Å^3
Space Group	$P2_12_12$ (#18)
Z value	8
D_{calc}	1.197 g/cm^3
F_{000}	912.00
m(CuKa)	6.46 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku AFC5R
Radiation	CuKa (l = 1.54178 Å) graphite monochromated
Attenuator	Ni foil (factors = 1.00, 3.65, 13.29, 49.39)

Take-off Angle	6.0^{o}
Detector Aperture	6.0 mm horizontal 6.0 mm vertical
Crystal to Detector Distance	285 mm
Voltage, Current	50kV, 160mA
Temperature	23.0^{o}C
Scan Type	w-2q
Scan Rate	16.0^{o}/min (in w) (up to 6 scans)
Scan Width	$(1.73 + 0.35 \tan q)^{o}$
$2q_{max}$	127.0^{o}
No. of Reflections Measured	Total: 2171
Corrections	Lorentz-polarization Absorption (trans. factors: 0.9641 - 1.0000) Decay (0.00% increase) Secondary Extinction (coefficient: 2.35896e-006)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$S\ w\ (Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\ s^2(Fo^2) + (0.1000 \cdot P)^2 +$ $+ 0.0000 \cdot P\ ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
p-factor	0.0075
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00s(I))	1415
No. Variables	272

Reflection/Parameter Ratio	5.20
Residuals: R1; wR2	0.051 ; 0.175
Goodness of Fit Indicator	1.24
Max Shift/Error in Final Cycle	0.03
Maximum peak in Final Diff. Map	0.20 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.23 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	1.1932(2)	0.3152(3)	0.8332(3)	4.92(8)
O(2)	1.0691(2)	0.3947(3)	0.7705(3)	6.8(1)
O(3)	0.7761(2)	0.2100(3)	0.6833(2)	4.09(7)
O(4)	0.9001(2)	0.1532(3)	0.7588(3)	5.90(9)
N(1)	1.0844(2)	0.1970(4)	0.7916(3)	4.7(1)
N(2)	0.8802(2)	0.3420(3)	0.7039(3)	4.24(9)
C(1)	1.2823(3)	0.1834(5)	0.9406(3)	4.3(1)
C(2)	1.3009(4)	0.0508(5)	0.9495(5)	6.2(2)
C(3)	1.3671(4)	0.0244(5)	0.8703(5)	7.1(2)
C(4)	1.3823(3)	0.1472(5)	0.8229(4)	5.5(1)
C(5)	1.2999(3)	0.1750(5)	0.7641(3)	5.5(1)
C(6)	1.2293(3)	0.1968(4)	0.8470(3)	3.8(1)
C(7)	1.3756(3)	0.2323(5)	0.9131(4)	4.8(1)
C(8)	1.2375(4)	0.2358(6)	1.0296(4)	6.7(2)
C(9)	1.4482(3)	0.2093(6)	0.9889(4)	8.1(2)
C(10)	1.3783(3)	0.3636(5)	0.8858(5)	6.9(2)
C(11)	1.1455(3)	0.1192(4)	0.8380(4)	4.8(1)
C(12)	1.1091(3)	0.3082(5)	0.7950(4)	4.5(1)
C(13)	0.6478(3)	0.3311(4)	0.6325(3)	3.37(9)
C(14)	0.6320(3)	0.4502(4)	0.5800(4)	4.8(1)
C(15)	0.6427(3)	0.4209(5)	0.4677(4)	6.2(2)
C(16)	0.6654(3)	0.2907(5)	0.4714(3)	4.8(1)
C(17)	0.7598(3)	0.2801(5)	0.5136(4)	4.5(1)
C(18)	0.7482(3)	0.3109(4)	0.6231(3)	3.3(1)
C(19)	0.6074(3)	0.2444(4)	0.5556(3)	3.7(1)
C(20)	0.6113(3)	0.3269(5)	0.7369(3)	5.0(1)
C(21)	0.6215(3)	0.1124(4)	0.5803(4)	5.0(1)
C(22)	0.5071(3)	0.2584(5)	0.5395(4)	5.6(1)
C(23)	0.8080(3)	0.4085(4)	0.6632(4)	4.3(1)
C(24)	0.8584(3)	0.2311(4)	0.7205(4)	4.0(1)
H(1)	1.3256	0.0356	1.0147	7.842
H(2)	1.2486	0.0075	0.9426	7.842
H(3)	1.4211	-0.0048	0.8981	8.654
H(4)	1.3441	-0.0295	0.8251	8.654
H(5)	1.4362	0.1540	0.7861	6.958
H(6)	1.3079	0.2428	0.7253	6.480
H(7)	1.2837	0.1101	0.7242	6.480
H(8)	1.1910	0.2861	1.0070	7.840
H(9)	1.2139	0.1745	1.0679	7.840
H(10)	1.2793	0.2798	1.0655	7.840

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(11)	1.5016	0.1935	0.9563	9.721
H(12)	1.4540	0.2767	1.0300	9.721
H(13)	1.4312	0.1436	1.0285	9.721
H(14)	1.3780	0.3718	0.8168	8.155
H(15)	1.3290	0.4023	0.9141	8.155
H(16)	1.4317	0.3965	0.9122	8.155
H(17)	1.0318	0.1711	0.7609	5.576
H(18)	1.1250	0.0957	0.9016	6.037
H(19)	1.1565	0.0515	0.7991	6.037
H(20)	0.5740	0.4780	0.5921	5.785
H(21)	0.6745	0.5057	0.5987	5.785
H(22)	0.5884	0.4352	0.4326	7.506
H(23)	0.6883	0.4667	0.4385	7.506
H(24)	0.6572	0.2501	0.4099	5.766
H(25)	0.7831	0.2041	0.5058	5.540
H(26)	0.7986	0.3364	0.4831	5.540
H(27)	0.6173	0.4034	0.7658	6.113
H(28)	0.5518	0.3042	0.7363	6.113
H(29)	0.6456	0.2726	0.7749	6.113
H(30)	0.5730	0.0685	0.5539	5.922
H(31)	0.6750	0.0858	0.5519	5.922
H(32)	0.6230	0.1024	0.6498	5.922
H(33)	0.4802	0.2867	0.5978	6.602
H(34)	0.4972	0.3129	0.4870	6.602
H(35)	0.4824	0.1843	0.5220	6.602
H(36)	0.9373	0.3740	0.7174	5.120
H(37)	0.7783	0.4534	0.7131	5.137
H(38)	0.8264	0.4593	0.6120	5.137

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.038(2)	0.044(2)	0.105(3)	0.002(2)	-0.008(2)	0.018(2)
O(2)	0.050(2)	0.072(2)	0.136(4)	0.013(2)	-0.006(2)	0.045(3)
O(3)	0.040(2)	0.044(2)	0.071(2)	-0.003(1)	-0.013(1)	0.008(2)
O(4)	0.050(2)	0.075(2)	0.099(3)	-0.002(2)	-0.026(2)	0.026(2)
N(1)	0.037(2)	0.061(3)	0.080(3)	0.001(2)	-0.010(2)	-0.002(3)
N(2)	0.040(2)	0.052(2)	0.069(3)	-0.008(2)	-0.015(2)	0.009(2)
C(1)	0.047(3)	0.060(3)	0.056(3)	0.007(3)	0.005(2)	-0.003(3)
C(2)	0.068(3)	0.070(4)	0.097(5)	0.014(3)	-0.012(4)	0.027(4)
C(3)	0.064(4)	0.064(4)	0.140(6)	0.017(3)	-0.006(4)	-0.012(4)
C(4)	0.049(3)	0.083(4)	0.078(4)	0.014(3)	0.006(3)	-0.015(3)
C(5)	0.069(3)	0.086(4)	0.052(3)	0.001(3)	-0.005(3)	-0.012(3)
C(6)	0.037(2)	0.038(2)	0.069(3)	0.000(2)	-0.000(2)	0.012(3)
C(7)	0.045(3)	0.071(4)	0.065(3)	0.002(3)	-0.002(2)	-0.006(3)
C(8)	0.074(4)	0.104(5)	0.076(4)	-0.003(4)	0.015(3)	-0.014(4)
C(9)	0.058(3)	0.143(6)	0.105(4)	0.004(4)	-0.023(3)	-0.035(5)

C(10)	0.052(3)	0.078(4)	0.132(5)	-0.017(3)	0.011(4)	0.003(4)
C(11)	0.046(3)	0.050(3)	0.084(4)	-0.003(2)	-0.007(3)	-0.001(3)
C(12)	0.038(3)	0.058(3)	0.076(3)	0.002(3)	0.001(3)	0.015(3)
C(13)	0.036(2)	0.044(2)	0.048(3)	0.002(2)	-0.002(2)	-0.002(2)
C(14)	0.050(3)	0.052(3)	0.082(4)	0.007(3)	-0.009(3)	0.012(3)
C(15)	0.063(3)	0.091(4)	0.083(4)	-0.010(3)	-0.017(3)	0.033(4)
C(16)	0.060(3)	0.079(4)	0.042(3)	-0.001(3)	-0.005(2)	0.002(3)
C(17)	0.042(2)	0.071(4)	0.057(3)	-0.001(3)	0.007(2)	0.004(3)
C(18)	0.039(2)	0.038(2)	0.048(3)	0.005(2)	-0.005(2)	0.005(2)
C(19)	0.035(2)	0.052(3)	0.054(3)	0.003(2)	-0.005(2)	-0.006(2)
C(20)	0.057(3)	0.078(4)	0.056(3)	0.000(3)	0.016(3)	-0.013(3)
C(21)	0.057(3)	0.053(3)	0.079(4)	-0.002(3)	-0.011(3)	-0.011(3)
C(22)	0.038(2)	0.083(4)	0.093(4)	0.001(3)	-0.012(3)	-0.003(3)
C(23)	0.045(3)	0.049(3)	0.069(3)	-0.008(2)	-0.006(3)	0.006(3)
C(24)	0.035(2)	0.058(3)	0.060(3)	0.002(2)	-0.009(2)	0.007(3)

The general temperature factor expression:
$exp(-2p^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
O(1)	C(6)	1.470(7)	O(1)	C(12)	1.366(7)
O(2)	C(12)	1.211(7)	O(3)	C(18)	1.467(7)
O(3)	C(24)	1.359(7)	O(4)	C(24)	1.206(7)
N(1)	C(11)	1.422(8)	N(1)	C(12)	1.319(9)
N(1)	H(17)	0.95	N(2)	C(23)	1.445(7)
N(2)	C(24)	1.326(7)	N(2)	H(36)	0.95
C(1)	C(2)	1.532(9)	C(1)	C(6)	1.509(8)
C(1)	C(7)	1.550(8)	C(1)	C(8)	1.485(9)
C(2)	C(3)	1.51(1)	C(2)	H(1)	0.95
C(2)	H(2)	0.95	C(3)	C(4)	1.55(1)
C(3)	H(3)	0.95	C(3)	H(4)	0.95
C(4)	C(5)	1.503(9)	C(4)	C(7)	1.566(9)
C(4)	H(5)	0.95	C(5)	C(6)	1.560(8)
C(5)	H(6)	0.95	C(5)	H(7)	0.95

C(6)	C(11)	1.538(8)	C(7)	C(9)	1.524(9)
C(7)	C(10)	1.53(1)	C(8)	H(8)	0.95
C(8)	H(9)	0.95	C(8)	H(10)	0.95
C(9)	H(11)	0.95	C(9)	H(12)	0.95
C(9)	H(13)	0.95	C(10)	H(14)	0.95
C(10)	H(15)	0.95	C(10)	H(16)	0.95

Table 3. Bond Lengths(Å) (continued)

atom	atom	distance	atom	atom	distance
C(11)	H(18)	0.95	C(11)	H(19)	0.95
C(13)	C(14)	1.551(8)	C(13)	C(18)	1.529(7)
C(13)	C(19)	1.548(8)	C(13)	C(20)	1.513(8)
C(14)	C(15)	1.55(1)	C(14)	H(20)	0.95
C(14)	H(21)	0.95	C(15)	C(16)	1.53(1)
C(15)	H(22)	0.95	C(15)	H(23)	0.95
C(16)	C(17)	1.546(8)	C(16)	C(19)	1.536(8)
C(16)	H(24)	0.95	C(17)	C(18)	1.543(9)
C(17)	H(25)	0.95	C(17)	H(26)	0.95
C(18)	C(23)	1.527(8)	C(19)	C(21)	1.547(9)
C(19)	C(22)	1.543(8)	C(20)	H(27)	0.95
C(20)	H(28)	0.95	C(20)	H(29)	0.95
C(21)	H(30)	0.95	C(21)	H(31)	0.95
C(21)	H(32)	0.95	C(22)	H(33)	0.95
C(22)	H(34)	0.95	C(22)	H(35)	0.95
C(23)	H(37)	0.95	C(23)	H(38)	0.95

Table 4. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle

Table 5. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
O(1)	C(6)	C(1)	C(2)	171.1(5)	O(1)	C(6)	C(1)	C(7)	-82.4(6)
O(1)	C(6)	C(1)	C(8)	46.4(8)	O(1)	C(6)	C(5)	C(4)	122.1(6)
O(1)	C(6)	C(11)	N(1)	17.6(7)	O(1)	C(12)	N(1)	C(11)	7.4(8)
O(2)	C(12)	O(1)	C(6)	-174.9(7)	O(2)	C(12)	N(1)	C(11)	-172.1(8)
O(3)	C(18)	C(13)	C(14)	172.4(5)	O(3)	C(18)	C(13)	C(19)	-83.4(6)
O(3)	C(18)	C(13)	C(20)	47.2(7)	O(3)	C(18)	C(17)	C(16)	119.5(5)
O(3)	C(18)	C(23)	N(2)	22.7(6)	O(3)	C(24)	N(2)	C(23)	6.8(8)
O(4)	C(24)	O(3)	C(18)	-169.1(6)	O(4)	C(24)	N(2)	C(23)	-174.6(7)
N(1)	C(11)	C(6)	C(1)	141.2(6)	N(1)	C(11)	C(6)	C(5)	-98.9(6)
N(1)	C(12)	O(1)	C(6)	5.6(7)	N(2)	C(23)	C(18)	C(13)	144.3(5)
N(2)	C(23)	C(18)	C(17)	-94.8(6)	N(2)	C(24)	O(3)	C(18)	9.6(7)
C(1)	C(2)	C(3)	C(4)	1.5(8)	C(1)	C(6)	O(1)	C(12)	-140.6(5)
C(1)	C(6)	C(5)	C(4)	2.6(7)	C(1)	C(7)	C(4)	C(3)	-55.1(6)
C(1)	C(7)	C(4)	C(5)	55.1(6)	C(2)	C(1)	C(6)	C(5)	-72.9(6)
C(2)	C(1)	C(6)	C(11)	53.2(7)	C(2)	C(1)	C(7)	C(4)	55.8(6)
C(2)	C(1)	C(7)	C(9)	-59.4(8)	C(2)	C(1)	C(7)	C(10)	174.3(7)
C(2)	C(3)	C(4)	C(5)	-72.5(8)	C(2)	C(3)	C(4)	C(7)	34.7(7)
C(3)	C(2)	C(1)	C(6)	70.8(7)	C(3)	C(2)	C(1)	C(7)	-37.5(8)
C(3)	C(2)	C(1)	C(8)	-164.6(6)	C(3)	C(4)	C(5)	C(6)	69.5(7)
C(3)	C(4)	C(7)	C(9)	62.9(7)	C(3)	C(4)	C(7)	C(10)	-174.7(6)

Table 5. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(4)	C(5)	C(6)	C(11)	-124.6(6)	C(4)	C(7)	C(1)	C(6)	-53.9(6)
C(4)	C(7)	C(1)	C(8)	-179.9(6)	C(5)	C(4)	C(7)	C(9)	173.1(6)
C(5)	C(4)	C(7)	C(10)	-64.5(7)	C(5)	C(6)	O(1)	C(12)	106.5(6)
C(5)	C(6)	C(1)	C(7)	33.6(6)	C(5)	C(6)	C(1)	C(8)	162.4(7)
C(6)	C(1)	C(7)	C(9)	-169.1(6)	C(6)	C(1)	C(7)	C(10)	64.7(8)
C(6)	C(5)	C(4)	C(7)	-37.3(7)	C(6)	C(11)	N(1)	C(12)	-16.1(8)
C(7)	C(1)	C(6)	C(11)	159.8(5)	C(8)	C(1)	C(6)	C(11)	-71.5(8)
C(8)	C(1)	C(7)	C(9)	64.9(9)	C(8)	C(1)	C(7)	C(10)	-61.4(9)
C(11)	C(6)	O(1)	C(12)	-14.8(7)	C(13)	C(14)	C(15)	C(16)	-1.6(7)
C(13)	C(18)	O(3)	C(24)	-145.8(5)	C(13)	C(18)	C(17)	C(16)	1.3(7)
C(13)	C(19)	C(16)	C(15)	-57.5(5)	C(13)	C(19)	C(16)	C(17)	53.1(6)
C(14)	C(13)	C(18)	C(17)	-71.2(6)	C(14)	C(13)	C(18)	C(23)	56.0(7)
C(14)	C(13)	C(19)	C(16)	54.2(5)	C(14)	C(13)	C(19)	C(21)	173.7(5)
C(14)	C(13)	C(19)	C(22)	-64.8(6)	C(14)	C(15)	C(16)	C(17)	-70.3(7)
C(14)	C(15)	C(16)	C(19)	37.1(6)	C(15)	C(14)	C(13)	C(18)	73.4(6)
C(15)	C(14)	C(13)	C(19)	-33.7(6)	C(15)	C(14)	C(13)	C(20)	-161.2(5)
C(15)	C(16)	C(17)	C(18)	72.4(7)	C(15)	C(16)	C(19)	C(21)	-177.2(6)
C(15)	C(16)	C(19)	C(22)	61.8(7)	C(16)	C(17)	C(18)	C(23)	-127.0(6)
C(16)	C(19)	C(13)	C(18)	-53.1(5)	C(16)	C(19)	C(13)	C(20)	178.7(5)
C(17)	C(16)	C(19)	C(21)	-66.6(7)	C(17)	C(16)	C(19)	C(22)	172.3(6)

Table 5. Torsion Angles(^{o}) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(17)	C(18)	O(3)	C(24)	101.4(6)	C(17)	C(18)	C(13)	C(19)	33.0(6)
C(17)	C(18)	C(13)	C(20)	163.6(6)	C(18)	C(13)	C(19)	C(21)	66.4(7)
C(18)	C(13)	C(19)	C(22)	-172.1(6)	C(18)	C(17)	C(16)	C(19)	-35.2(7)
C(18)	C(23)	N(2)	C(24)	-19.1(7)	C(19)	C(13)	C(18)	C(23)	160.1(5)
C(20)	C(13)	C(18)	C(23)	-69.2(7)	C(20)	C(13)	C(19)	C(21)	-61.8(7)
C(20)	C(13)	C(19)	C(22)	59.7(7)	C(23)	C(18)	O(3)	C(24)	-20.5(6)

Table 6. Non-bonded Contacts out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
O(1)	H(37)	3.1170	76502	O(1)	H(1)	3.2555	75704
O(1)	H(24)	3.4164	55603	O(2)	H(36)	2.1304	1
O(2)	H(36)	2.7193	76502	O(2)	H(37)	2.9788	76502
O(2)	N(2)	3.049(7)	1	O(2)	H(38)	3.1392	76502
O(2)	O(2)	3.157(9)	76502	O(2)	N(2)	3.210(7)	76502
O(2)	H(24)	3.2443	55603	O(2)	C(23)	3.250(8)	76502
O(2)	H(13)	3.4480	45703	O(3)	H(23)	3.2582	64604
O(3)	H(4)	3.3426	75502	O(3)	H(10)	3.4085	45703
O(3)	H(19)	3.5065	75502	O(4)	H(17)	1.9941	1
O(4)	H(19)	2.5370	75502	O(4)	N(1)	2.864(6)	1
O(4)	H(12)	3.0815	45703	O(4)	H(10)	3.0986	45703
O(4)	C(11)	3.350(8)	75502	O(4)	H(18)	3.4472	75502
O(4)	H(22)	3.5867	64604	N(1)	H(24)	3.0018	55603
N(1)	H(12)	3.1353	45703	N(1)	H(36)	3.1660	1
N(1)	H(22)	3.3901	55603	N(1)	C(24)	3.575(8)	1
N(2)	H(17)	3.0953	1	N(2)	H(35)	3.4385	55603
N(2)	H(34)	3.5899	55603	C(2)	H(8)	3.0766	74704
C(2)	H(15)	3.1749	74704	C(2)	H(10)	3.3325	74704
C(3)	H(3)	3.2178	85502	C(3)	H(8)	3.2999	74704
C(3)	H(32)	3.3358	75502	C(3)	H(11)	3.3770	85502

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
C(4)	H(28)	3.3263	65501	C(4)	H(3)	3.5260	85502
C(5)	H(24)	3.3155	55603	C(5)	H(26)	3.3629	55603
C(8)	H(29)	3.0097	55703	C(8)	H(2)	3.1196	75704
C(8)	H(4)	3.5476	75704	C(8)	H(1)	3.5844	75704
C(9)	H(3)	3.2950	85502	C(10)	H(27)	3.1162	76502
C(10)	H(28)	3.3771	65501	C(10)	H(2)	3.4285	75704
C(11)	H(12)	3.5991	45703	C(12)	H(36)	2.9024	1
C(12)	H(24)	2.9446	55603	C(12)	H(37)	3.3818	76502
C(12)	H(12)	3.4721	45703	C(14)	H(20)	3.2195	66502
C(14)	H(25)	3.3691	65604	C(14)	H(33)	3.4544	66502
C(14)	H(34)	3.5553	66502	C(15)	H(31)	3.3426	65604
C(15)	H(7)	3.3821	45603	C(15)	H(25)	3.4219	65604
C(16)	H(7)	3.3811	45603	C(16)	H(6)	3.4381	45603
C(17)	H(6)	3.3312	45603	C(17)	H(35)	3.4127	55603
C(17)	H(7)	3.4701	45603	C(20)	H(9)	3.0546	45703
C(20)	H(5)	3.3734	45501	C(20)	H(14)	3.5870	76502
C(21)	H(38)	3.2315	64604	C(21)	H(23)	3.3307	64604
C(21)	H(26)	3.4719	64604	C(21)	H(4)	3.4898	75502
C(21)	H(7)	3.5034	75502	C(21)	H(30)	3.5953	65502
C(22)	H(20)	3.3143	66502	C(22)	H(26)	3.3415	45603

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
C(22)	H(25)	3.4629	45603	C(23)	H(31)	3.5569	65604
C(24)	H(17)	2.7601	1	C(24)	H(10)	3.1445	45703
C(24)	H(19)	3.3875	75502	C(24)	H(34)	3.5349	55603
H(1)	H(8)	2.8612	74704	H(1)	H(15)	2.9444	74704
H(1)	H(10)	3.4835	74704	H(2)	H(15)	2.5639	74704
H(2)	H(10)	2.6237	74704	H(2)	H(8)	2.7611	74704
H(2)	H(16)	3.5876	74704	H(3)	H(3)	2.3832	85502
H(3)	H(11)	2.5644	85502	H(3)	H(5)	3.1326	85502
H(3)	H(8)	3.1874	74704	H(3)	H(13)	3.2518	85502
H(4)	H(32)	2.5642	75502	H(4)	H(29)	2.8492	75502
H(4)	H(8)	3.1378	74704	H(4)	H(10)	3.2188	74704
H(4)	H(11)	3.4715	85502	H(5)	H(28)	2.5318	65501
H(5)	H(33)	3.0364	65501	H(5)	H(32)	3.4210	65501
H(5)	H(29)	3.4392	65501	H(5)	H(32)	3.5630	75502
H(6)	H(24)	2.9217	55603	H(6)	H(26)	2.9668	55603
H(6)	H(33)	3.1612	65501	H(6)	H(25)	3.2110	55603
H(6)	H(21)	3.3430	76502	H(7)	H(23)	2.7732	55603
H(7)	H(26)	2.8815	55603	H(7)	H(32)	2.9709	75502
H(7)	H(24)	3.0764	55603	H(7)	H(31)	3.2849	75502
H(8)	H(11)	2.9107	45703	H(8)	H(29)	3.1061	55703

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
H(9)	H(29)	2.4411	55703	H(9)	H(27)	2.8258	55703
H(9)	H(15)	3.1696	74704	H(9)	H(37)	3.4440	55703
H(9)	H(11)	3.5521	45703	H(10)	H(29)	3.0167	55703
H(11)	H(28)	3.3230	65501	H(11)	H(29)	3.4024	65501
H(11)	H(18)	3.5932	55703	H(12)	H(18)	3.1015	55703
H(12)	H(17)	3.1234	55703	H(13)	H(36)	3.4501	55703
H(14)	H(27)	2.6468	76502	H(14)	H(28)	2.9422	65501
H(14)	H(21)	3.3599	76502	H(14)	H(37)	3.3891	76502
H(14)	H(33)	3.4806	65501	H(14)	H(20)	3.5639	76502
H(15)	H(27)	3.0936	76502	H(15)	H(18)	3.3984	75704
H(15)	H(37)	3.5684	76502	H(16)	H(27)	3.1061	76502
H(16)	H(16)	3.1270	86502	H(16)	H(28)	3.1723	65501
H(16)	H(27)	3.4325	65501	H(16)	H(18)	3.4963	75704
H(17)	H(36)	2.7743	1	H(17)	H(22)	3.0108	55603
H(17)	H(24)	3.1203	55603	H(17)	H(34)	3.4050	55603
H(19)	H(23)	3.2616	55603	H(19)	H(22)	3.3065	55603
H(20)	H(20)	2.2889	66502	H(20)	H(33)	2.7969	66502
H(20)	H(34)	2.9709	66502	H(20)	H(22)	3.4131	66502
H(21)	H(25)	2.7381	65604	H(21)	H(31)	3.1871	65604
H(21)	H(33)	3.3182	66502	H(22)	H(22)	3.0475	66502

Table 6. Non-bonded Contacts out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
H(22)	H(34)	3.2255	66502	H(23)	H(31)	2.4715	65604
H(23)	H(25)	2.8348	65604	H(23)	H(32)	3.4519	65604
H(24)	H(38)	3.3257	64604	H(25)	H(34)	3.2381	55603
H(25)	H(35)	3.2849	55603	H(25)	H(33)	3.2907	55603
H(26)	H(35)	2.7844	55603	H(26)	H(31)	2.9014	65604
H(26)	H(33)	3.2660	55603	H(26)	H(30)	3.3106	65604
H(26)	H(34)	3.4673	55603	H(30)	H(30)	2.6969	65502
H(30)	H(38)	2.9821	64604	H(30)	H(35)	3.0223	65502
H(31)	H(38)	2.6460	64604	H(35)	H(36)	3.3795	45603
H(35)	H(38)	3.3912	45603	H(36)	H(36)	3.4310	76502

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB, and TC are the crystal lattice translation digits along cell edges a, b, and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9, and thus, $\pm$4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure is given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

(1)	+X,	+Y,	+Z	(2)	-X,	-Y,	+Z
(3)	1/2+X,	1/2-Y,	-Z	(4)	1/2-X,	1/2+Y,	-Z