Supporting Information

The first CNS-active carborane: A novel $P2X_7$ receptor antagonist with antidepressant activity.

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Synthesis Experimental

General experimental

Melting points were determined using a Stuart SMP10 Melting Point Apparatus and are uncorrected.

Infrared absorption (IR) spectra were obtained using a Bruker ALPHA FT-IR spectrometer with (neat) samples pressed against a selenium-zinc cell (SeZn). Significant absorbance bands are reported in wavenumbers (cm^{-1}) and are described by the abbreviations: br = broad, s = strong, m = medium, w = weak. Where applicable, bands are also assigned their respective functional group.

^{1}H Nuclear Magnetic Resonance (^{1}H NMR) spectra were obtained on a Bruker DRX400 (400 MHz) or AVANCE III 500 Ascend (500 MHz) at 300 K unless otherwise stated. Chemical shift data is expressed in ppm relative to $\delta_{TMS} = 0$, using residual protons in deuterated solvent as an internal reference. The data is reported as chemical shift (δ), relative integral, observed multiplicity (s = singlet, d = doublet, dd = doublet of doublets, quart = quartet, quint = quintet, m = multiplet), coupling constant(s) (*J* Hz) and assignment (unassigned diastereotopic protons are allocated lower case letters e.g. a, b, etc.). All multiplicities and coupling constants are apparent. Assignment of signals was assisted by 2D multiplicity-edited HSQC and HMBC experiments where necessary.

^{13}C Nuclear Magnetic Resonance (^{13}C NMR) spectra were obtained on a Bruker DRX400 (100 MHz) or AVANCE III 500 Ascend (125 MHz) at 300 K with complete proton decoupling unless otherwise stated. Chemical shift data is expressed in ppm relative to $\delta_{TMS} = 0$, using deuterated solvent as an internal reference. The data is reported as chemical shift (δ), and assignment. The protonicity (CH_3 = primary, CH_2 = secondary, CH = tertiary, C_q = quaternary carbon, C_{Ar} = aromatic carbon) and assignment of signals was assisted by 2D multiplicity-edited HSQC and HMBC experiments where necessary.

Low resolution mass spectra (LRMS) was obtained from a ThermoQuest Finnigan LCQ Deca ion trap mass spectrometer with electro-spray ionization in either positive (+ESI) or negative (-ESI) mode or atmospheric-pressure chemical ionization in positive mode (+APCI). Gas chromatography with mass spectrometer (GC-MS) was performed on a ThermoQuest Finnigan PolarisQ mass spectrometer attached to a Trace 2000 GC. Data is expressed as observed mass (*m/z*), assignment (M = molecular ion), and relative intensity (%).

Elemental analysis was determined in duplicate using a PerkinElmer PE2400 Elemental Analyzer (CHNS) at Macquarie University, NSW, Australia and are uncorrected.

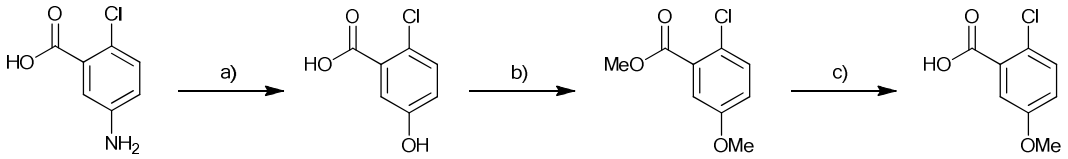
Analytical thin layer chromatography (TLC) was performed using 0.2 mm thick, aluminum-backed, pre-coated silica gel plates (Merck Silica gel 60 F_{254}). Compounds were visualized by short and long wavelength ultra-violet fluorescence and by staining with potassium permanganate (40 g potassium carbonate, 6 g potassium permanganate, 600 mL H_2O then 5 mL of 10% NaOH).

Normal-phase Flash chromatography was performed using Merck Silica gel 60 (230 – 400 mesh ASTM), under a positive pressure of N_2, with the indicated solvents. Solvent compositions were mixed volume per volume (v/v) as specified.

Evaporation or concentration under reduced pressure refers to evaporation using a rotary evaporator connected to a vacuum pump. Removal of residual solvent when necessary was achieved by evacuation (0.01 - 0.1 mm Hg) with a high stage oil sealed vacuum pump.

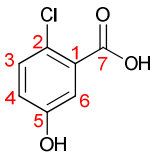
All solvents and reagents were dried and purified when necessary according to published standard procedures.[1] Tetrahydrofuran was dried over sodium wire (determined by benzophenone) and distilled. Dichloromethane and boron trifluoride diethyl etherate were distilled from calcium hydride at ambient pressure. Hexanes refers to hexanes (bp 65 – 69 °C) and brine refers to saturated aqueous sodium chloride solution. Moisture sensitive reactions were carried out in oven dried glassware under a dry inert atmosphere of N_2 or Ar. Reaction temperatures were controlled using ice : water (0 – 5 °C) cooling baths or oil heating baths (> room temperature). Room temperature ranges from 18-25 °C.

Synthesis of the 2-chloro-5-methoxybenzoic acid (9)

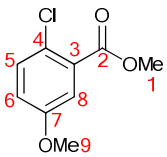


S1 S2 S3 9

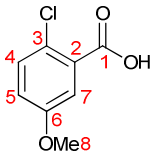
Scheme 1 Conditions: a) H_2SO_4, $NaNO_2$, <5 °C, 1h, then H_2O, 65 °C, 0.5 h, 72%; b) MeI, K_2CO_3, DMF, 40 °C, 95%; c) LiOH, THF/H_2O, Δ, 76%



2-Chloro-5-hydroxybenzoic acid (S2). 5-Amino-2-chlorobenzoic acid **S1** (2.60 g, 15.2 mmol) was suspended in aq. H_2SO_4 (1.25% *v*/*v*, 240 mL) and cooled below 5 °C with an ice bath. A solution of $NaNO_2$ (1.55 g, 225 mmol) in H_2O (45 mL) was added dropwise to maintain a reaction temperature below 5 °C. The reaction was stirred until all solids had dissolved and a clear solution resulted (~1 hr). The reaction was poured into hot H_2O (450 mL, 65 °C) and decolorizing charcoal (2 g) was added. The mixture was refluxed for 0.5 h, then cooled to room temperature and filtered. The filtrate was extracted with EtOAc (3 x 300 mL) and combined and dried over anhyd. Na_2SO_4. The solvent was removed by rotary evaporation to give crude 2-chloro-5-hydroxybenzoic acid **S2** (1.88 g, 71.5%) as a light brown solid. **M.P.** 174-177 °C (lit. M.P. 186-189 °C)[2]; **IR** (ZnSe cell): 3302 (broad, O-H), 1693, 1656, 1569, 1432, 1212, 660 cm^{-1}; **^{1}H NMR** (300 MHz, d_6-DMSO): δ 13.23 (1H, s, broad, CO_2H), 9.95 (1H, s, broad, ArOH), 7.29 (1H, d, $J_{H3\text{-}H4}$ 8.7, H3), 7.15 (1H, d, $^4J_{H4\text{-}H6}$ 3.0, H6), 6.90 (1H, dd, $^3J_{H3\text{-}H4}$ 8.7, $^4J_{H4\text{-}H6}$ 3.0, H4); **^{13}C NMR** (75 MHz, d_6-DMSO): δ 166.6 (C7), 156.1 (C5), 132.0 (C1), 131.5 (C3), 121.0 (C2), 119.6, 117.2; **LRMS (-ESI)**: *m/z* 343 ([2M-H]$^-$, 100%), 171 ([M-H]$^-$, 37%). The spectroscopic data matched that reported in the literature.[3]

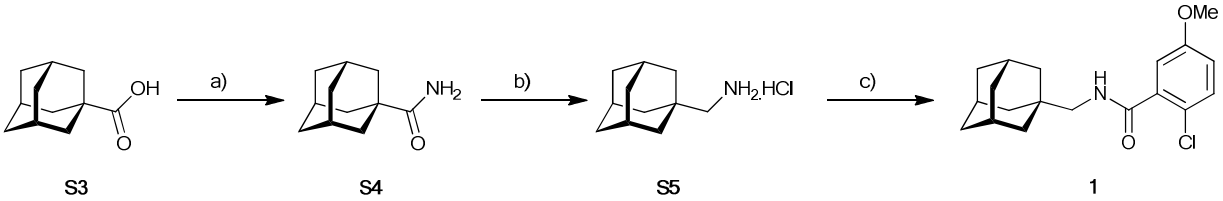


Methyl 2-chloro-5-methoxybenzoate (S3). Iodomethane (3.0 mL, 49 mmol) was added to 2-chloro-5-hydroxybenzoic acid **S2** (1.5 g, 8.7 mmol) and K_2CO_3 (5.3g, 39 mmol) in DMF (75 mL). The reaction mixture was heated at 40 °C for 20 h under a N_2 atmosphere, then cooled to RT and quenched with H_2O (150 mL). The ester was extracted with Et_2O (3 x 100 mL) with the organic layers combined and washed with aq. NaOH (0.1 M, 150 mL), then H_2O (2 x 100 mL), and then dried over anhyd. $MgSO_4$. The solvent was removed under vacuo to yield the brown oil of methyl 2-chloro-2-methoxybenzoate **S3** (1.67 g, 95%). **IR** (ZnSe cell): 2953 (w, C-H), 2841 (w, C-H), 1731 (s, C=O), 1478 (m), 1433 (m), 1288 (s), 1218 (s, C-O), 1114 (s), 1055 (s), 1025 (s), 816 (m), 777 (m), 642 (m, C-Cl) cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$): δ 7.35-7.28 (2H, m, H5, H8), 6.94 (1H, dd, $^3J_{H5\text{-}H6}$ 9.0, $^4J_{H6\text{-}H8}$ 3.0, H6), 3.91 (3H, s, H1), 3.80 (3H, s, H9); **^{13}C NMR** (75 MHz, $CDCl_3$): δ 166.1 (C2), 158.0 (C7), 131.9 (Ar-H), 130.7 (C3), 125.0 (C4), 119.0 (ArH), 116.1 (ArH), 55.8 (C9), 52.5 (C1) ppm.



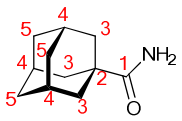
2-Chloro-5-methoxybenzoic acid (9). Methyl 2-chloro-5-methoxybezoate **S3** (1.62 g, 8.07 mmol) was dissolved in THF (30 mL) and aq. LiOH (1 M, 40 mL) was added. The solution was heated at reflux for 4 h, then stirred overnight (~16 h). The solution was reduced to half by rotary evaporation then aq. HCl (6 M) was added until pH 1. The resulting white precipitate was extracted into CH_2Cl_2 (2 x 150 mL). The organic layers were combined, dried over anhyd. $MgSO_4$ and evaporated to dryness. The resulting brown gum was recrystallized from H_2O (~200-250 mL) resulting in the tan-colored powder of 2-chloro-5-methoxybenzoic acid **9** (1.145 g, 76%). **M.P.** 178-180 °C (lit. M.P. 178-181 °C)[3]; **^{1}H NMR** (400 MHz, DMSO-d_6): δ 13.48 (1H, s, broad, CO_2H), 7.43 (1H, d, $J_{H4\text{-}H5}$ 8.8, H4), 7.28 (1H, d, $^4J_{H5\text{-}H7}$ 2.4, H7), 7.10 (1H, dd, $^3J_{H4\text{-}H5}$ 8.8, $^4J_{H5\text{-}H7}$ 2.8, H5), 3.78 (3H, s, H8); **^{13}C NMR** (75 MHz, DMSO-d_6): δ 166.7 (C1), 157.8 (C6), 132.4 (C2), 131.6 (C4), 122.7 (C3), 118.5 (C5), 115.5 (C7), 55.7 (C8) ppm. The spectroscopic data matched that reported in the literature.[3]

Synthesis of adamantanyl benzamide (1)

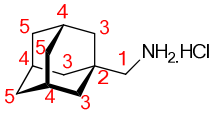


S3 S4 S5 1

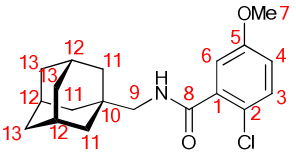
Scheme 2. a) CDI, THF, rt, 1 h, then 28% $NH_{3(aq)}$, rt, 4 h 97%; b) $LiAlH_4$, THF, reflux, 20 h, then HCl, 89%; c) 2-chloro-5-methoxybenzoic acid **9**, $(COCl)_2$, THF, rt, 1 h, then amine **S5**, THF, Et_3N, rt, 16 h, 72%



1-Adamantanecarboxamide (S4). 1-Adamantanecarboxylic acid **S3** (5.0 g, 27.7 mmol) and 1,1'-carbonyldiimidazole (5.4 g, 33.3 mmol) were stirred for 1 h at room temperature in THF (100 mL) under a N_2 atmosphere. The reaction was cooled on ice then aq. NH_3 (28%, 14 mL) was added. The reaction was stirred for 4 h, allowing the solution to warm to room temperature. The solvent was removed by rotary evaporation and the residue dissolved in CH_2Cl_2 (350 mL) and washed with NaOH (1 M, 150 mL), then HCl (1 M, 150 mL) and then H_2O (150 mL). The organic layer was dried over anhyd. $MgSO_4$ and evaporated to dryness to yield 1-adamantanecarboxamide **S4** (4.80 g, 97%) as a colorless solid. This product was used in the next step without any further purification. **M.P.** 190-192 °C (lit. M.P. 186-192 °C)[4]; **^{1}H NMR** (400 MHz, d_6-DMSO): δ 6.90 (1H, br s, NH), 6.64 (1H, br s, NH), 1.94 (3 H, br s, H4), 1.74 (6H, d, J = 2.8, H3), 1.64 (6H, m, H5) ppm; **^{13}C NMR** (100 MHz, d_6-DMSO): δ 179.2 (C1), 39.63 (C2), 38.74 (3C, C3), 36.16 (3C, C5), 27.66 (3C, C4) ppm.



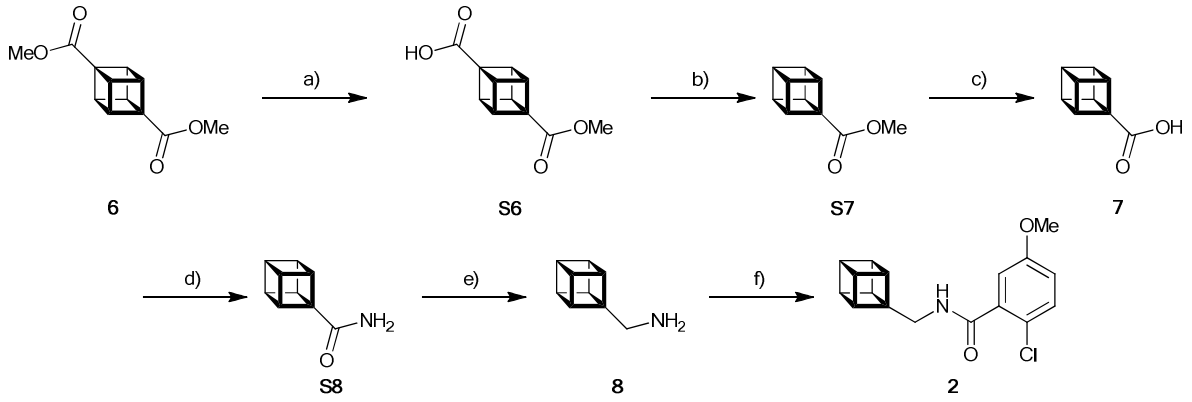
Adamantan-1-ylmethylamine hydrochloride (S5). A solution of 1-adamantanecarboxamide **S4** (4.78 g, 26.7 mmol) in THF (110 mL) was treated with $LiAlH_4$ (3.92 g, 103 mmol) at 0 ºC and stirred under a N_2 atmosphere whilst warming to room temperature. After 2 h, the reaction was heated at reflux for 16 h and then cooled on ice. Chilled H_2O (4 mL) was added dropwise, with vigorous stirring, and then followed by NaOH (15% w/v, 4 mL) and additional H_2O (12 mL). The solution was left stirring at room temperature until effervescence had ceased and the grey powder had turned white (30 min). The solution was dried with anhyd. $MgSO_4$ and filtered. The precipitate was washed with CH_2Cl_2 (2 x 100 mL). The filtrate in each case were combined and evaporated by rotary evaporation to yield a yellow-orange oil (4.20 g). This residue was dissolved in Et_2O (30 mL), washed with NaOH (1 M, 10 mL) and then H_2O (10 mL) before drying over anhyd. $MgSO_4$. The $MgSO_4$ was removed by filtration and the resulting filtrate was treated with HCl in dioxane (4 M, 10 mL). The resulting white precipitate was collected by filtration and washed with Et_2O to yield adamantan-1-ylmethylamine hydrochloride **S5** (4.811 g, 89%) as a colorless powder. **M.P.** 360-370 °C; **IR** (ZnSe cell): 2899, 2845 (C-H), 1093 (C-N); **^{1}H NMR** (300 MHz, $CDCl_3$): δ 8.29 (3H, br s, NH_3^+), 2.65 (2H, q, J = 6 Hz, H1), 2.03 (3H, br s, H4), 1.80-1.55 (12 H, m, H3, H5) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ 51.3 (C1), 39.8 (3C), 36.6 (3C), 32.2 (C2), 28.0 (3C) ppm; **HRMS** (+ESI): Calc. for $C_{11}H_{20}N$ [M-Cl]+: 166.15903, found 166.15903; **LRMS** (+ESI): 166 ([M-Cl]+, 100).



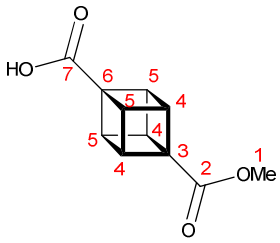
***N*-(Adamantan-1-ylmethyl)-2-chloro-5-methoxybenzamide (1).** Oxalyl chloride (500 μL, 5.91 mmol) was added to 2-chloro-5-methoxybenzoic acid **9** (500 mg, 2.68 mmol) in THF (10 mL). A drop of DMF was added to the solution which resulted in effervescence and the reaction mxture was left stirring at room temperature under a N_2 atmosphere. After 1 h, the solvent was removed by a stream of N_2 gas and the acid chloride dried under a high vacuum (0.5 h). The acid chloride was dissolved in THF (10 mL) and added to adamantan-1-ylmethylamine hydrochloride **S5** (540 mg, 2.68 mmol) with THF washings (5 mL). Et_3N (800 μL, 5.74 mmol) was added to the reaction mixture which was then left stirring at room temperature under a N_2 atmosphere overnight (18 h). The solvent was removed by rotary evaporation and the residue partitioned between EtOAc (100 mL) and aq. NaOH (1 M, 50 mL). The organic layer was collected and washed with $HCl_{(aq)}$ (1 M, 50 mL) before being dried over anhyd. $MgSO_4$ and evaporated to dryness. The resulting off-white solid was subjected to flash chromatography (1:3, EtOAc: hexane) to yield pure *N*-(adamantan-1-ylmethyl)-2-chloro-5-methoxybenzamide **1** (640 mg, 72 %) as a colorless solid. **M.P.** 115-118 °C; **IR** (thin film): 3447, 3288 (N−H), 1645 (C=O), 1599, 1450, 1292, 808, 617 cm^{-1}; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 7.34-7.22 (2H, m, H3, H6), 6.88 (1H, dd, ^3J = 9.0 Hz, ^4J = 3.0 Hz, H4), 6.35 (1H, br s, NH), 3.81 (3H, s, H7), 3.16 (2H, d, J = 6.0 Hz, H9), 1.99 (3H, br s, H12), 1.68 (6H, m, H13), 1.58 (6H, d, J = 2.0 Hz, H11) ppm; **^{13}C NMR** (126 MHz, $CDCl_3$): δ 166.4 (C8), 158.6 (C5), 135.9 (C1), 131.2 (C3), 121.7 (C2), 118.0 (C4), 115.3 (C6), 55.8 (C7), 51.9 (C9), 40.4 (C11), 37.1 (C13), 34.0 (C10), 38.4 (C12) ppm;

HRMS (+ESI) Calc. for $C_{19}H_{24}O_2N^{35}Cl$ $[M+H]^+$: 334.1571, found: 334.1562; **LRMS** (+ESI): 691/689 ($[2M+Na]^+$, 66/100), 334.1 ($[M+H]^+$, 26); **Anal.** ($C_{19}H_{24}ClNO_2$): calc, C 68.35, H 7.25, N 4.20; found, C 68.47, H 7.24, N 4.26.

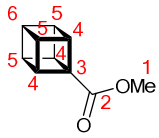
Synthesis of cubanyl benzamide (2)



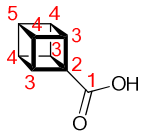
Scheme 3. a) NaOH (1 equiv.), MeOH, THF, rt, 16 h, then $HCl_{(aq)}$, 88%; b) $(COCl)_2$, CH_2Cl_2, rt, 1 h, then 2-mercaptopyridine *N*-oxide sodium salt, *hv*, DMAP, $CHCl_3$, reflux, 1 h, 78%; c) NaOH, MeOH, reflux, 1 h, 97%; d) CDI, THF, rt, 2 h, then $NH_4OH_{(aq)}$, 4 °C to rt, 5 h, 85%; e) $LiAlH_4$, THF, 0 °C to reflux, 16 h, 51%; f) 2-chloro-5-methoxybenzoic acid **9**, $(COCl)_2$, CH_2Cl_2, rt, 1 h, then amine **8**, CH_2Cl_2, rt, 16 h, 66%



4-Methoxycarbonylcubane carboxylic acid (S6).[5] A solution of methanolic NaOH (2.5 M, 8.5 mL, 21 mmol) was added dropwise to a solution of dimethyl 1,4-cubanedicarboxylate **6** (4.5 g, 20 mmol) in THF (140 mL) at ambient temperature. The mixture was stirred for 16 h and then concentrated under reduced pressure with minimal heating. The solid residue was diluted with H_2O (50 mL) and washed with $CHCl_3$ (3 × 25 mL). The aqueous layer was acidified to pH 3 by the dropwise addition of conc. HCl (2.5 mL) and then extracted with $CHCl_3$ (1 × 80 mL; 2 × 40 mL). The combined organic extracts were dried over anhyd. Na_2SO_4 and evaporated *in vacuo* to yield the half ester **S6** (3.7 g, 88%) as a colorless solid. **M.P.** 184–186 °C (lit. M.P. 182–183 °C)[5]; **^{1}H NMR** (200 MHz, $CDCl_3$): δ 4.27 (6H, m, H4, H5), 3.72 (3H, s, H1) ppm. The spectroscopic data matched that reported in the literature.[5]



Methyl cubanecarboxylate (S7).[6] Oxalyl chloride (4.6 mL, 58.65 mmol) was added to the half ester **S6** (5.70 g, 27.6 mmol) in CH_2Cl_2 (140 mL). A drop of DMF was added to which resulted in effervescence and the reaction left stirring at room temperature under a N_2 atmosphere. After 1 h, the solvent was removed by a stream of N_2 gas and the acid chloride dried under a high vacuum for at least 1 h. The acid chloride was dissolved in $CHCl_3$ (140 mL) added to a solution of mercaptopyridine *N*-oxide sodium salt (5.07 g, 34.0 mmol) and *N*,*N*-dimethylpyridine (340 mg, 2.78 mmol) in $CHCl_3$ (140 mL). The combined solution was refluxed by irradiation from a 500 W tungsten filament lamp, placed *ca.* 2 cm from the reaction vessel. After 1 h, the solvent was removed by rotary evaporation and the resulting residue dissolved in Et_2O (500 mL) and washed with HCl (1 M, 2 x 500 mL), aq. Na_2CO_3 (sat., 500 mL) and then brine (500 mL). The organic layer was dried over anhyd. $MgSO_4$ and evaporated to dryness by rotary evaporation. The crude ester was subjected to flash chromatography (1:9, Et_2O:hexanes) to yield pure mono ester **S7** (3.48 g, 78%) as a sweet-smelling, low melting point, colorless solid. **M.P.** 51-52 °C (lit. M.P. 51.2-52.9 °C)[6]; **^{1}H NMR** (400 MHz, $CDCl_3$): δ 4.28-4.21 (3 H, m, H4), 4.04-3.95 (4H, m, H5, H6), 3.69 (3 H, s, H1) ppm; **^{13}C NMR** (100 MHz, $CDCl_3$): δ 173.0 (C2), 55.8 (C3), 51.5 (C1), 49.6 (C4), 48.0 (C6), 45.3 (C5) ppm. The spectroscopic data matched that reported in the literature.[6]



Cubanecarboxylic acid (7).[6] The monoester **S7** (2.95 g, 18.2 mmol) was refluxed in methanolic NaOH (1.25 M, 40 mL) for 2 h and then the solvent was removed by rotary evaporation. The residue was dissolved in H_2O (50 mL) and washed with Et_2O (50 mL). The aqueous layer was acidified to pH 1 with conc. HCl resulting in a white precipitate that was extracted into CH_2Cl_2 (100 mL). The aqueous layer was extracted once more with CH_2Cl_2 (100 mL) and the two organic layers were combined, dried over anhyd. $MgSO_4$ and evaporated to dryness to yield cubanecarboxylic acid **7** (2.62 g, 97%) as a light yellow solid. **M.P.** 126-128 °C (lit. M.P. 124-125 °C)[5]; **^{1}H NMR** (500 MHz, $CDCl_3$): δ 4.18-4.4.11 (3 H, m,

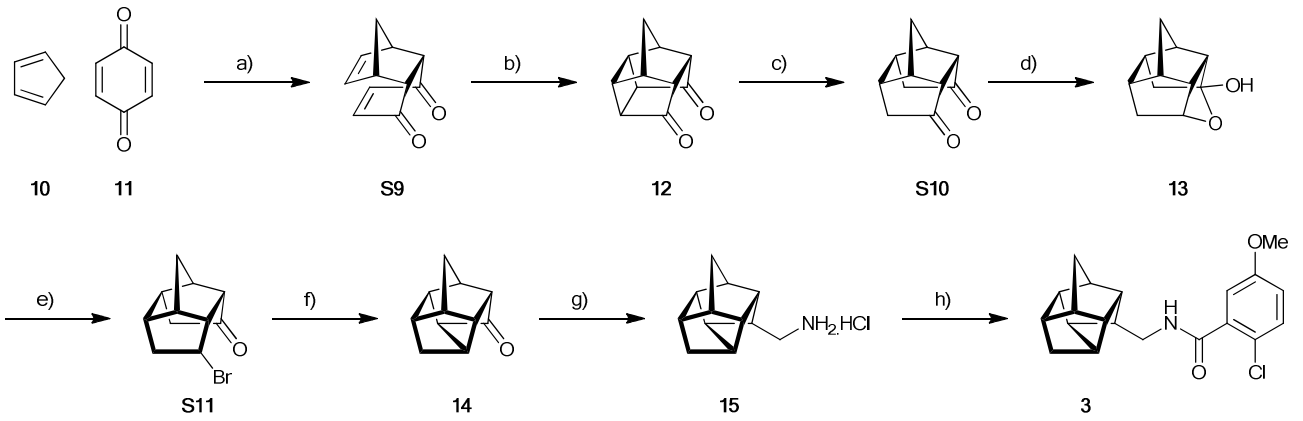
H3), 4.00-3.93 (4H, m, H4, H5) ppm; **^{13}C NMR** (125 MHz, $CDCl_3$): δ 172.8 (C1), 55.3 (C2), 48.5 (C3), 47.0 (C5), 44.1 (C4) ppm. The spectroscopic data matched that reported in the literature.[5]

Cubanecarboxamide (S8). Cubanecarboxylic acid **7** (2.60 g, 17.6 mmol) and 1,1'-carbonyldiimidazole (3.41 g, 21.0 mmol) were stirred for 2 h at room temperature in THF (65 mL) under a N_2 atmosphere. The reaction was cooled on ice then aq. NH_3 (28%, 9 mL) was added. The reaction was stirred for 5 h, allowing the solution to warm to room temperature. The solvent was removed by rotary evaporation and the residue dissolved in hot $CHCl_3$ (300 mL) and washed with aq. Na_2CO_3 (sat., 100 mL) and then HCl (1 M, 100 mL). The organic layer was dried over anhyd. $MgSO_4$ and evaporated to dryness to yield cubanecarboxamide **S8** (2.206 g, 85%). **M.P.** 209-211 °C (lit. M.P. 209-212 °C)[7]; **^{1}H NMR** (500 MHz, d_6-DMSO): δ 7.15 (1H, br s, NH_a), 6.87 (1H, br s, NH_b), 4.12 (3H, m, H3), 3.96 (1H, m, H5), 3.92 (3H, m, H4) ppm; **^{13}C NMR** (125 MHz, d_6-DMSO): δ 172.3 (C1), 55.8 (C2), 48.5 (C3), 46.8 (C5), 43.8 (C4) ppm.

Cubane-1-methylamine (8). Powdered $LiAlH_4$ (0.45 g, 12 mmol) was added to a solution of cubanecarboxamide **S8** (0.37g, 2.5 mmol) in THF (60 mL) at 0 °C with stirring. After warming to ambient temperature, the suspension was heated at reflux for 16 h before cooling to 0 °C. Aq. NaOH (15% *w*/*v*, 2 mL) was added dropwise with vigorous stirring to quench the excess $LiAlH_4$ after which the mixture was stirred at ambient temperature for 1 h. The granular precipitate was filtered off and washed with CH_2Cl_2 (70 mL). The combined organic portions were dried over anhyd. Na_2SO_4 and evaporated under reduced pressure. The crude product was purified by flash chromatography (90:10:0.5, CH_2Cl_2/MeOH/$NH_{3(aq)}$) to yield the amine **8** (287 mg, 70%) as a light brown oil. $\mathbf{R}_f$ 0.14 (90:10:0.5, CH_2Cl_2/MeOH/$NH_{3(aq)}$); **^{1}H NMR** (300 MHz, $CDCl_3$): δ 4.03 (1H, m, H5), 3.92–3.75 (6H, m, H3, H4), 2.82 (2H, s, H1) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ 59.7, 48.9, 47.0 (3C), 44.5, 44.3 (3C) ppm. The spectroscopic data matched that reported in the literature.[7]

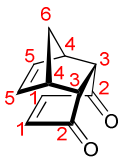
2-Chloro-*N*-(cuban-1-ylmethyl)-5-methoxybenzamide (2). Oxalyl chloride (740 μL, 8.63 mmol) was added to 2-chloro-5-methoxybenzoic acid **9** (800 mg, 4.29 mmol) in THF (13 mL). A drop of DMF was added to which resulted in effervescence and the reaction left stirring at room temperature under a N_2 atmosphere. After 1 h, the solvent was removed by a stream of N_2 gas and the acid chloride dried under a high vacuum. In a separate flask, Et_3N (600 μL, 4.29 mmol) was added to freshly prepared cubane-1-methylamine **8** (293 mg, 2.20 mmol) dissolved in THF (10 mL). The amine solution was added to the neat acid chloride with THF washes (2 x 1.5 mL) resulting in a white precipitate. The reaction was left stirring at room temperature under a N_2 atmosphere overnight (18 h). The solvent was removed by rotary evaporation and the residue partitioned between EtOAc (150 mL) and aq. NaOH (1 M, 50 mL). The organic layer was collected and washed again with NaOH (1 M, 50 mL) and then aq. HCl (1 M, 50 mL). The organic layer was collected, dried over anhyd. $MgSO_4$ and evaporated to dryness. The resulting off-white solid was subjected to flash chromatography (1:3, EtOAc: hexane) to yield **2** (435 mg, 66 %) as an off-white solid. The benzamide **2** was crystallized from EtOAc and hexane. **M.P.** 160-161 °C, $\mathbf{R}_f$ 0.24 (1:3, EtOAc: hexane); **IR** (thin film): 3313 (N–H), 3192, 3074, 3065, 2986, 2966, 2924, 2851, 2835, 2733, 2696, 2258, 2231, 2193, 2131, 1638 (C=O), 1605, 1545, 1298, 1136, 1024, 818, 519, 509 cm-1; **^{1}H NMR** (400 MHz, d_6-DMSO): δ 8.45 (1H, br t, J = 5.8 Hz, NH), 7.38 (1H, d, J = 8.8 Hz, H3), 7.00 (1H, dd, ^3J = 8.8 Hz, ^4J = 2.8 Hz, H4), 6.91 (1H, d, ^4J = 2.8 Hz, H6), 4.00 (1H, m, H13), 3.92-3.83 (6H, m, H11, H12), 3.77 (3H, s, H7), 3.45 (2H, d, J = 6.0 Hz, H9) ppm ; **^{13}C NMR** (101 MHz, d_6-DMSO): δ 166.2 (C8), 157.8 (C5), 138.1 (C1), 130.4 (C3), 120.8 (C2), 116.1 (C4), 114.0 (C6), 57.1 (C10), 55.6 (C7), 47.5 (C13), 47.1 (C11), 43.3 (C12), 40.8 (C9) ppm; **HRMS** (+ESI) Calc. for $C_{17}H_{16}O_2N^{35}Cl$ $[M + Na]^+$: 324.0764, found: 324.0756; **LRMS** (+ESI): 627/625 ($[2M + Na]^+$, 84/100), 324 ($[M + Na]^+$, 95), 302 ($[M + H]^+$, 30); **Anal.** ($C_{17}H_{16}ClNO_2$): calc. C 67.66, H 5.34, N 4.64; found C 67.89, H 5.48, N 4.49.

Synthesis of trishomocubanyl benzamide (3)

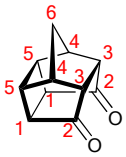


10 11 S9 12 S10 13 S11 14 15 3

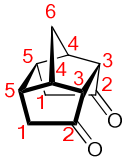
Scheme 4. a) PhMe, -10 °C to rt, 3 h, 78%; b) *hv*, Me_2CO/hexanes, 8 h, 92%; c) Zn, AcOH, rt, 5 h, 90%; d) $NaBH_4$, EtOH/H_2O, rt, 3 h, quant.; e) 33% HBr in CH_3CO_2H, sealed tube, 100 °C, 16 h; f) *t*-BuOK, Et_2O, rt, 16 h, 70% over 2 steps; g) TosMIC, *t*-BuOK, $(MeOCH_2)_2$, EtOH, 5-35 °C, then $LiAlH_4$, Et_2O, reflux, 16 h, then anhyd. HCl, Et_2O (70% over 3 steps); h) 2-chloro-5-methoxybenzoic acid **9**, EDC.HCl, HOBt, NMM, CH_2Cl_2, 0 °C to rt, 16 h, 80%.



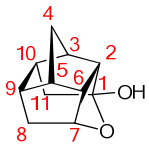
***Endo-cis*-1,4,4a,8a-tetrahydro-1,4-methanonaphthalene-5,8-dione (S9)**.[8] A solution of cold, freshly cracked cyclopentadiene **10** (17.2 mL, 210 mmol) in PhMe (20 mL) was added dropwise over 30 min to a mixture of recrystallized 1,4-benzoquinone **11** (24 g, 220 mmol) in PhMe (200 mL) at -10 °C. The reaction mixture was stirred at ambient temperature for 3 h and then evaporated *in vacuo*. The crude residue was recrystallized from MeOH to give the desired adduct **S9** (28.5 g, 78%) as yellow crystals. **^{1}H NMR** (200 MHz, $CDCl_3$): δ 6.56 (2H, s, H1), 6.07 (2H, br s, H5), 3.55 (2H, br s, H4), 3.22 (2H, br s, H3), 1.54 (1H, dt, *J* = 8.8, 1.6 Hz, H), 1.42 (1H, br d, *J* = 8.8 Hz, $H6_b$) ppm. The spectroscopic data matched that reported in the literature.[8]



Pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-dione (Cookson diketone, 12).[8] The *endo* adduct **S9** (13.0 g, 74 mmol) was dissolved in degassed $(CH_3)_2CO$/hexane (1:9 v/v,250 mL) under an Ar atmosphere and irradiated with a 350 W Hanovia mercury lamp (*ca.* 10 cm distance) for 8 h. After completion of photocyclization, the solvent was evaporated *in vacuo* to give crude Cookson diketone **12** (12.0 g, 92%) as a colorless solid. Recrystallisation from EtOAc/hexane (20:80) gave analytically pure **12** as colorless crystals. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 3.14 (2H, br s), 2.91 (2H, br s), 2.78 (2H, br s), 2.67 (2H, br s), 2.02 (1H, d, *J* = 11.3 Hz, $H6_a$), 1.85 (1H, d, *J* = 11.3 Hz, $H6_b$) ppm. The spectroscopic data matched that reported by the Aldrich Library and in the literature.[8]

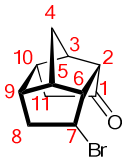


Tetracyclo[6.3.0.0^{4,11}.0^{5,9}]undecane-2,7-dione (S10).[9] A mixture of Cookson diketone **12** (10 g, 57 mmol) and zinc dust (40 g, 612 mmol) in glacial AcOH (400 mL) was stirred at ambient temperature for 5 h. The reaction was then poured into ice H_2O (400 mL) and extracted with CH_2Cl_2 (2 × 200 mL, 2 × 150 mL). The combined organic extracts were washed with dilute NaOH (1.2 M, 4 × 200 mL), H_2O (200 mL), dried over anhyd. Na_2SO_4 and evaporated *in vacuo* to give the diketone **S10** (9.0 g, 90%) as a colorless solid. This was used in the next step without further purification. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 2.80 (2H, br s, H3), 2.75–2.65 (4H, m, H1), 2.29–2.20 (4H, m, H4, H5), 1.98 (1H, d, *J* = 7.4 Hz, $H6_a$), 1.85 (1H, d, *J* = 7.4 Hz, $H6_b$) ppm. The spectroscopic data matched that reported in the literature.[9]

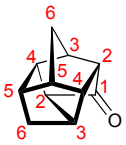


Octahydro-2,3,5-ethanylylidene-2*H*-pentaleno[1,6-*bc*]furan-2-ol (13).[10] To a solution of the diketone **S10** (9.0 g, 51 mmol) in aq. EtOH (1:1 v/v, 600 mL) was added $NaBH_4$ (3.2 g, 85 mmol) at ambient temperature in one portion, and the resulting mixture was stirred for 3 h. The reaction mixture was concentrated *in vacuo* and H_2O (300 mL) was added to the residue, followed by extraction with CH_2Cl_2 (1 × 150 mL, 2 × 100 mL, 1 × 50 mL). The combined organic extracts were washed with H_2O (2 × 100 mL), dried over anhyd. Na_2SO_4 and evaporated by rotary evaporation to give lactol **13** (9.1 g, >98%) as a colorless solid. This was used in the next step without further purification. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 4.63 (1H, t, *J* = 6.6 Hz), 4.26 (1H, m), 2.90–2.83 (1H, m), 2.47 (1H, br s), 2.39–2.33 (1H, m), 2.20 (3H, br s), 2.07 (1H, d, *J* =13.2 Hz), 1.95 (1H, m), 1.85 (1H, d, *J* = 14.1 Hz), 1.79 (1H, d, *J* =10.8 Hz), 1.66 (2H, d, *J* = 10.8 Hz) ppm. The spectroscopic data matched that reported in the literature.[10]

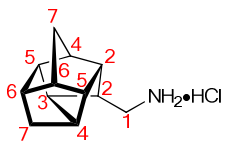
8-Bromohexahydro-1,2,4-[1]propanyl[3]ylidenepentalen-5(1*H*)-one (S11).[10] The lactol **13** (9.1 g, 51 mmol) was treated with a solution of HBr in AcOH (33%, 40 mL) and heated to 100 ºC with stirring in a sealed tube overnight, and then stirred at ambient temperature for a further 16 h. The reaction was quenched by carefully pouring into a mixture of sat. $NaHCO_{3(aq.)}$ and CH_2Cl_2 (3:1 v/v, 600 mL). The aqueous layer was removed and extracted with additional CH_2Cl_2 washes (3 × 50 mL). The combined organic layers were washed sequentially with aq. $NaHCO_3$ (sat., 3 × 100 mL), H_2O (2 × 100 mL), dried over anhyd. Na_2SO_4 and then evaporated *in vacuo* to give the crude bromoketone **S11** (12 g, 95%) as a brown oil. This was used in the next step without further purification. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 4.29 (1H, dd, *J* = 7.2, 3.6 Hz), 3.00 (1H, s), 2.78–2.71 (1H, m), 2.65 (1H, br s), 2.53–2.36 (4H, m), 2.34–2.25 (1H, m), 2.20–2.12 (2H, m), 1.79 (2H, dd, J = 26.0, 11.1 Hz) ppm. The spectroscopic data matched that reported in the literature.[10]



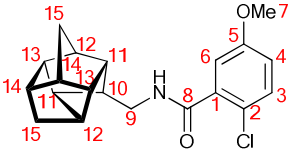
(±)-(D_3)-Trishomocubanone (Pentacyclo[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecan-4-one, 14).[11] A suspension of the bromoketone **S11** and KO^{t}Bu (8.5 g, 76 mmol) in Et_2O (150 mL) was stirred at ambient temperature for 16 h. The reaction was acidified to pH 6 with 1 M HCl and then concentrated *in vacuo*. Water (150 mL) was added, followed by extraction with CH_2Cl_2 (4 × 80 mL). The combined organic extracts were washed with H_2O (2 × 100 mL), dried over anhyd. Na_2SO_4 and evaporated by rotary evaporation. The crude product was purified by flash chromatography (9:1, hexane:Et_2O) to yield the desired ketone **14** (5.9 g, 70% over 2 steps) as a colorless solid. **^{1}H NMR** (300 MHz, $CDCl_3$): δ 2.45 (2H, br s), 2.40–2.35 (4H, m), 1.80–1.76 (2H, m), 1.69 (2H, dd, *J* = 10.4, 1.5 Hz), 1.42 (2H, d, *J* = 10.2 Hz) ppm. The spectroscopic data matched that reported in the literature.[11]



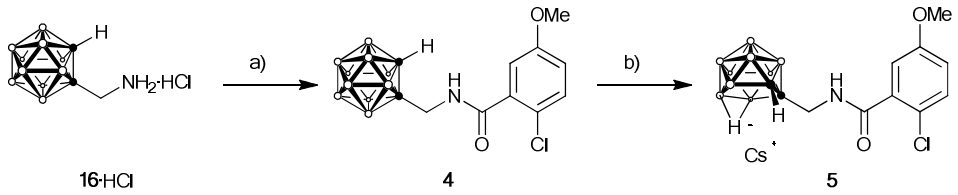
D_3-Trishomocubylmethylamine hydrochloride (15). A solution of D_3-trishomocubanone **14** (4.0 g, 25 mmol), *p*-tosylmethylisocyanide (7.2 g, 37 mmol) and EtOH (3 mL) in freshly distilled 1,2-dimethoxyethane (100 mL) was cooled to 5 ºC and solid KO^{t}Bu (7.6 g, 68 mmol) was added in several portions over 10 min, maintaining the temperature between 5 and 10 ºC. The mixture was then stirred at ambient temperature for 45 min and for a further 30 min at 35─40 ºC. After cooling, the precipitate was filtered off and washed with 1,2-dimethoxyethane (100 mL). The combined organic layers were concentrated *in vacuo* to *ca.* 3 mL and chromatographed through a short column of neutral alumina (hexane) to give 4-cyano-D_3-trishomocubane as a colorless oil which was used in the next step without further purification. A solution of the 4-cyano-D_3-trishomocubane in Et_2O (200 mL) was treated with $LiAlH_4$ (1.5 g, 39 mmol) at 0 ºC and subsequently heated at reflux for 16 h. After cooling to 0 ºC, aq. NaOH (15% *w*/*v* 20 mL) was added dropwise with vigorous stirring. After 1 h of stirring at 0 ºC, the mixture was filtered and the precipitate was washed with CH_2Cl_2 (500 mL). The combined organic layers were dried over anhyd. Na_2SO_4 and re-dissolved in Et_2O (80 mL). A solution of HCl in Et_2O (2M, 15 mL) was added dropwise at 0 °C and the mixture was stirred for 2 h at ambient temperature. Evaporation of the solvent furnished the desired amine salt **15** (3.7 g, 70% over 2 steps) as a colorless solid. **^{1}H NMR** (300 MHz, D_2O): δ 3.02 (2H, d, *J* = 7.7 Hz, H1), 2.35 (1H, br s, H2), 2.24–1.90 (7H, m), 1.48–1.24 (5H, m) ppm.



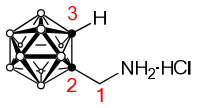
***N*-(D_3-trishomocuban-4-ylmethyl)-2-chloro-5-methoxybenzamide (3).** A mixture of the 2-chloro-5-methoxybenzoic acid **9** (522 mg, 2.80 mmol), D_3-trishomocubylmethylamine hydrochloride **15** (593 mg, 2.80 mmol), *N*-(3-dimethylaminopropyl)-*N*′-ethylcarbodiimide hydrochloride (700 mg, 3.65 mmol), and 1-hydroxybenzotriazole hydrate (14% H_2O content, 572 mg, 3.64 mmol) in CH_2Cl_2 (10 mL) was treated with *N*-methylmorpholine (925 μL, 8.41 mmol) dropwise at 0 °C. After stirring for 16 h at ambient temperature, aq. $NaHCO_3$ (sat., 30 mL) and CH_2Cl_2 (20 mL) were added. The organic layer was separated and the aqueous layer was further extracted with CH_2Cl_2 (2 × 30 mL). The combined organic layers were washed sequentially with aq. $NaHCO_3$ (sat., 40 mL) and brine (40 mL), dried over anhyd. Na_2SO_4, and then concentrated *in vacuo*. Subjecting the crude amide **3** to flash column chromatography (4:1, hexane:EtOAc) resulted in pure benzamide **3** (770 mg, 80%) as a colorless solid. **M.P.** 117–123 °C; **R$_f$** 0.38 (7:3, hexane/EtOAc); **IR** (KBr) 3277, 2955, 2867, 1645 (C=O), 1537, 1318, 1240, 1033, 857 cm^{-1}; **^{1}H NMR** (600 MHz, $CDCl_3$): δ 7.27 (1H, d, *J* = 8.4 Hz, H3), 7.24 (1H, d, ^4J = 3.0, H3, H6), 6.88 (1H, dd, ^3J = 9.0, ^4J = 3.0 Hz, H4), 6.32 (1H, br s, NH), 3.81 (3H, s, H7), 3.42 (2H, m, H9), 2.40 (1H, br s, H10), 2.20–1.96 (8H, m), 1.43 (2H, d, *J* = 10.2 Hz), 1.33 (2H, m) ppm; **^{13}C NMR** (75 MHz, $CDCl_3$): δ 166.1 (C8), 158.6 (C5), 135.9 (C1), 131.2 (C3), 121.8 (C2), 117.9 (C4), 115.1 (C6), 55.8 (C7), 49.8, 47.6, 45.9, 42.8, 40.6, 33.4, 33.1 ppm; **HRMS** (+ESI): Calc. for $C_{20}H_{23}{}^{37}ClNO_2$ [M + H]$^+$: 346.13823, found: 346.13844, Calc. for $C_{20}H_{23}{}^{35}ClNO_2$ [M + H]$^+$: 344.14118, found: 344.14125; **LRMS** (+ESI): 344 ([M+H]+, 100); **Anal.** ($C_{20}H_{22}ClNO_2$): calc, C 69.86, H 6.45, N 4.07; found, C 70.12, H 6.64, N 4.10.



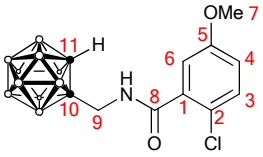
Synthesis of *closo o*-carboranyl benzamide 4 and *nido o*-carboranyl benzamide (5)



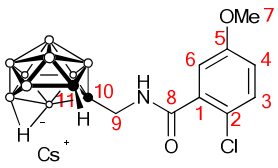
Scheme 5. a) 2-chloro-5-methoxybenzoic acid **9**, $(COCl)_2$, CH_2Cl_2, rt, 1 h, then amine **16**, THF, Et_3N, rt, 16 h, 72%; b) CsF (3 equiv.), EtOH, reflux, 24 h, 95%.



***Closo* 1,2-carboranylmethylamine hydrochloride (16•HCl).** The carboranylmethylamine hydrochloride **16**•HCl was prepared according to that reported by Wilson and Soloway.[12] The spectroscopic data matched that reported in the literature.[12]



***N*-(*closo*-1,2-carboranylmethyl)-2-chloro-5-methoxybenzamide (4).** Oxalyl chloride (100 μL, 1.15 mmol) was added to 2-chloro-5-methoxybenzoic acid **9** (100 mg, 0.536 mmol) suspended in CH_2Cl_2 (2 mL) under N_2 atmosphere. A drop of DMF was added (resulting in effervescence) and the solution stirred at room temperature for an hour. The solvent and excess oxalyl chloride were removed under high vacuum and the resulting oil was dissolved in CH_2Cl_2 (1 mL). In a separate flask, Et_3N (320 μL) was added to *N*-(*closo*-1,2-carboranyl)methylamine hydrochloride **16**•HCl (100 mg, 0.477 mmol) suspended in CH_2Cl_2 (1 mL) at room temperature under a N_2 atmosphere. The acid chloride solution was added by means of a cannula to this solution with CH_2Cl_2 washing (0.5 mL). The reaction mixture was left to stir at ambient temperature for 16 h under N_2 atmosphere. The reaction was diluted with $CHCl_3$ (25 mL) and washed with HCl (1 M, 2 x 10 mL), NaOH (1 M, 2 x 10 mL), and then H_2O (10 mL). The organic layer was dried over anhyd. $MgSO_4$ then evaporated to dryness. The resulting brown oil (137 mg) was subjected to flash chromatography (4:1, hexane : EtOAc) to afford *N*-(*closo*-1,2-carboranylmethyl)-2-chloro-5-methoxybenzamide **4** (105 mg, 64%) as an off-white solid. Recrystallization from ^{i}PrOH/ H_2O yielded white needles. **M.P.** 160-162 °C; **IR** (ZnSe cell): 3320 (N-H), 3040 (C-H), 2584 (B-H), 2554 (B-H), 1645 (C=O), 1533, 1234, 1019, 813, 642, 598 cm^{-1}; **^{1}H{^{11}B} NMR** (400 MHz, d_6-DMSO): δ 9.18 (1H, t, J 6.4, NH), 7.43 (1H, d, J = 8.8, H3), 7.07 (1H, dd, J = 8.8, ^4J = 2.8, H4), 6.97 (1H, d, ^4J = 2.8, H6), 5.09 (1H, s, H11), 4.02 (2H, d, J = 6.8, H9), 3.79 (3H, s, H7), 2.50-1.70 (10H, m, BH); **^{13}C NMR** (100 MHz, d_6-DMSO): δ 166.2 (C8), 157.8 (C5), 136.2 (C1), 130.9 (C3), 121.1 (C2), 116.7 (C4), 114.5 (C6), 75.9 (C11), 62.1 (C9), 55.7 (C7), 43.5 (C10); **^{11}B NMR** (128 MHz, d_6-DMSO): δ -2.90, -5.67, -9.72, -11.67, -12.57; **LRMS** (+ESI) m/z: 364 ([M+Na]$^+$, 100%), 470 (36%), 540 (54%); **HRMS** (+ESI) m/z: 361.2187 (8%, $C_{11}H_{20}{}^{10}B_5{}^{11}B_5{}^{35}ClNNaO_2$ [M+Na]$^+$ gives 361.2192), 362.2151 (29%, $C_{11}H_{20}{}^{10}B_4{}^{11}B_6{}^{35}ClNNaO_2$ [M+Na]$^+$ gives 362.2156), 363.2114 (66%, $C_{11}H_{20}{}^{10}B_3{}^{11}B_7{}^{35}ClNNaO_2$ [M+Na]$^+$ gives 363.2120), 364.2078 (100%, $C_{11}H_{20}{}^{10}B_2{}^{11}B_8{}^{35}ClNNaO_2$ [M+Na]$^+$ gives 364.2083), 365.2042 (90%, $C_{11}H_{20}{}^{10}B_1{}^{11}B_9{}^{35}ClNNaO_2$ [M+Na]$^+$ gives 365.2047), 366.2049 (38%, $C_{11}H_{20}{}^{10}B_2{}^{11}B_8{}^{37}ClNNaO_2$ [M+Na]$^+$ gives 366.2054), 367.2012 (30%, $C_{11}H_{20}{}^{10}B_1{}^{11}B_9{}^{37}ClNNaO_2$ [M+Na]$^+$ gives 367.2018), 368.1976 (11%, $C_{11}H_{20}{}^{11}B_{10}{}^{37}ClNNaO_2$ [M+Na]$^+$ gives 368.1981); **Anal.** ($C_{11}H_{20}B_{10}ClNO_2$): calc, C 38.65, H 5.90, N 4.10; found, C 70.12, H 6.64, N 4.10.



Cesium *N*-(*nido*-7,8-carboranylmethyl)-2-chloro-5-methoxybenzamide (5). A solution of *N*-(*closo-o*-carboranylmethyl)-2-chloro-5-methoxybenzamide **4** (100 mg, 0.293 mmol) and CsF (133 mg, 0.878 mmol) in EtOH (20 mL) was refluxed under a N_2 atmosphere for 24 h. The solvent was removed by rotary evaporation and $(CH_3)_2CO$ (20 mL) was added to the white residue. The borate salts were removed by filtration and the filtrate evaporated to dryness to yield cesium *N*-(*nido*-7,8-carboranylmethyl)-2-chloro-5-methoxybenzamide Cs·**5** (129 mg, 95%) as a colorless solid. **M.P.** 194-196 °C; **IR** (ZnSe cell): 3417 (w, Ar-H), 2513 (s, B-H), 1704 (m), 1644 (s, C=O), 1526 (m), 1472 (m), 1232 (m), 1024 (s) cm^{-1}; **^{1}H NMR** (400 MHz, d_6-DMSO): δ 8.37 (1H, t, J = 5.8, NH), 7.36 (1H, d, J 8.8, H3), 6.99 (1H, dd, ^3J = 8.6, ^4J = 3.0, H4), 6.91 (1H, d, ^4J = 2.8, H6), 3.78 (3H, s, H7), 3.31 (2H, m, H9), 1.92 (1H, br s, H11), 2.50-0.00 (9H, br, m, BH), -2.68 (1H, br d, ^1J 58, hydride); **^{13}C NMR** (100 MHz, d_6-DMSO): δ 165.3 (C8), 157.6 (C5), 137.9 (C1), 130.4 (C3), 121.2 (C2), 116.0 (C4), 114.3 (C6), 58.4 (br, carborane C), 55.6 (C7), 46.3 (C9), 45.1 (br, carborane C); **^{11}B NMR** (128 MHz, d_6-DMSO): δ -10.5, -14.7, -17.6, -22.6, -33.4, -37.4; **LRMS** (-ESI) m/z: 328 c 11%), 329 ([M-Cs]$^-$, 27%), 330, ([M-Cs]$^-$, 66%), 331 ([M-Cs]$^-$, 92%), 332 ([M-Cs]$^-$, 100%), 333 ([M-Cs]$^-$, 62%), 334 ([M-Cs]$^-$, 32%), 335 ([M-Cs]$^-$, 15%); **HRMS** (-ESI) m/z:

327.2204 (5%, $C_{11}H_{20}{}^{10}B_5{}^{11}B_4{}^{35}ClNO_2$ [M-Cs]$^-$ gives 327.2202), 328.2170 (20%, $C_{11}H_{20}{}^{10}B_4{}^{11}B_5{}^{35}ClNO_2$ [M-Cs]$^-$ gives 328.2165), 329.2133 (59%, $C_{11}H_{20}{}^{10}B_3{}^{11}B_6{}^{35}ClNO_2$ gives [M-Cs]$^-$ 329.2129), 330.2096 (100%, $C_{11}H_{20}{}^{10}B_2{}^{11}B_7{}^{35}ClNO_2$ [M-Cs]$^-$ gives 330.2093), 331.2059 (90%, $C_{11}H_{20}{}^{10}B_1{}^{11}B_7{}^{35}ClNO_2$ [M-Cs]$^-$ gives 331.2056), 332.2012 (38%, $C_{11}H_{20}{}^{11}B_9{}^{35}ClNO_2$ [M-Cs]$^-$ gives 332.2020), 333.2031 (30%, $C_{11}H_{20}{}^{10}B_1{}^{11}B_8{}^{37}ClNO_2$ [M-Cs]$^-$ gives 333.2027), 334.1996 (14%, $C_{11}H_{20}{}^{11}B_9{}^{37}ClNO_2$ [M-Cs]$^-$ gives 334.1991).

Lipophilicity measurements

General Experimental

High Performance Liquid Chromatography was performed on a Waters 2695 Separations module equipped with the Waters Alliance Series Column Heater (set at 30 °C) and Waters 2996 Photodiode Array (PDA) Detector. Samples were resolved on a Waters Sunfire™ C18 5 µm column (2.1 x 150 mm) using an isocratic flow of 65 v/v% methanol in 50 mM sodium phosphate buffer (pH 7.4) at a flowrate of 0.3 mL/min. Standards and samples were dissolved in the elution solvent mixture and then filtered prior to injection. Data acquisition and processing was performed with the Waters Empower 2 software and Microsoft Excel™ was used for data analysis. All standards and samples were analyzed in triplicate.

Standards calibration curve

The retention times (RET) of acetone ($logD_{7.4}$ -0.24), aniline ($logD_{7.4}$ 0.90), phenol ($logD_{7.4}$ 1.50), toluene ($logD_{7.4}$ 2.730), cumene ($logD_{7.4}$ 3.66), triphenylamine ($logD_{7.4}$ 5.74), and hexachlorobenzene ($logD_{7.4}$ 6.35) were plotted against their literature $logD_{7.4}$ values[13] to obtain a calibration curve. An exponential curve was obtained (**Figure 1**) which was fitted to the equation [$LogD_{7.4}$ = 0.8401ln(RET)+1.8781] with an R^2 of 0.97.

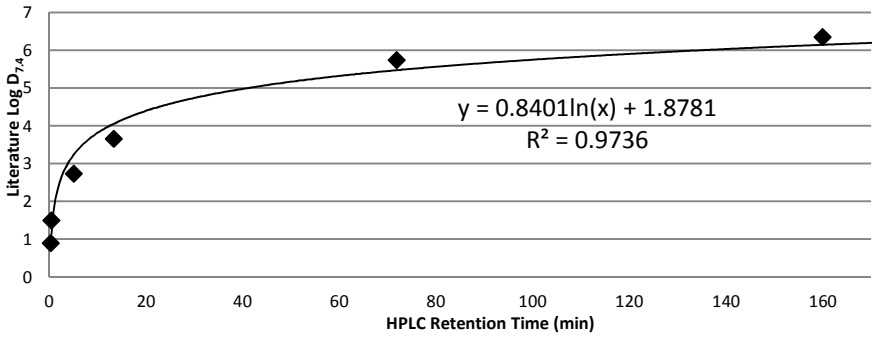


Figure 1. Calibration curve of standards with known $logD_{7.4}$ values against their HPLC retention times.

Sample lipophilicity measurements

The RET from three injections was averaged (**Table 1**) with the $logD_{7.4}$ value extrapolated from the equation found in **Figure 1**.

Table 1. Average RET and $logD_{7.4}$ values for the benzamide **1**-Cs·**5** series

Benzamide	Average RET (min)[a]	$logD_{7.4}$[b]
1	17.856 ± 0.005	4.30
2	6.271 ± 0.004	3.42
3	18.035 ± 0.005	4.31
4	17.724 ± 0.023	4.29
Cs·**5**	0.593 ± 0.005	1.44

[a] average retention time ± standard error based on the conditions outlined in the general experimental. [b] $LogD_{7.4}$ calculated from the equation $LogD_{7.4}$ = 0.8401ln(RET)+1.8781

Dye uptake assay

General experimental

Human monocytic leukaemia cells (THP-1) were obtained from the American Type Culture Collection (ATCC). Roswell Park Memorial Institute (RPMI) 1640 medium was purchased from Invitrogen.

Cell Culture

The human monocytic leukaemia cells (THP-1) were grown in RPMI 1640 medium (Invitrogen), supplemented with penicillin (100 U/mL), streptomycin (100 μg/mL) and 10% (*v*/*v*) heat-inactivated fetal bovine serum (FBS). Cells were cultured in 75 cm^2 flasks and incubated at 37 °C in a 5% CO_2 humidified atmosphere, maintained in log phase of growth and passaged when cell density reached 1 x 10^6 cells/mL (every 2-3 days).[14]

Yo-Pro Uptake Assay for Pore Formation

$P2X_7$ receptor pore formation was assessed by agonist-induced uptake of the Yo-Pro dye. Cells were plated in 96-well black walled, clear bottom plates in the presence of lipopolysaccharide (LPS, 25 ng/mL) and interferon-gamma (IFNγ, 10 ng/ml). Following an overnight incubation (16 h) required for THP-1 differentiation, cells were pre-incubated with various compound concentrations (diluted in DMSO) or control in culture medium (DMSO final concentration of 0.1%) for 30 min. Final treatments (in triplicate) were made up in phosphate buffered saline (PBS), without divalent cations, containing PRO®-1 dye (2 μM). Cells were washed once in pre-warmed PBS and treatments were added to cells, with or without 2′(3′)-O-(4-benzoylbenzoyl)adenosine 5′-triphosphate (BzATP, 100 μM) as agonist. Dye uptake was measured at 37 °C using a fluorescence plate reader (BMG Labtech); with data read at $\lambda_{Ex485nm}/\lambda_{Em520nm}$ at 30 second intervals over a 70 min period. Maximal intensity was determined by the EC_{70} value of agonist concentration (100 μM BzATP) and each compound concentration was expressed as a percentage of this maximal intensity to account for any variability between single experiments. IC_{50} values were determined from concentration-response curves derived from a four-parameter variable slope, analyzed in GraphPad Prism® (San Diego, CA).

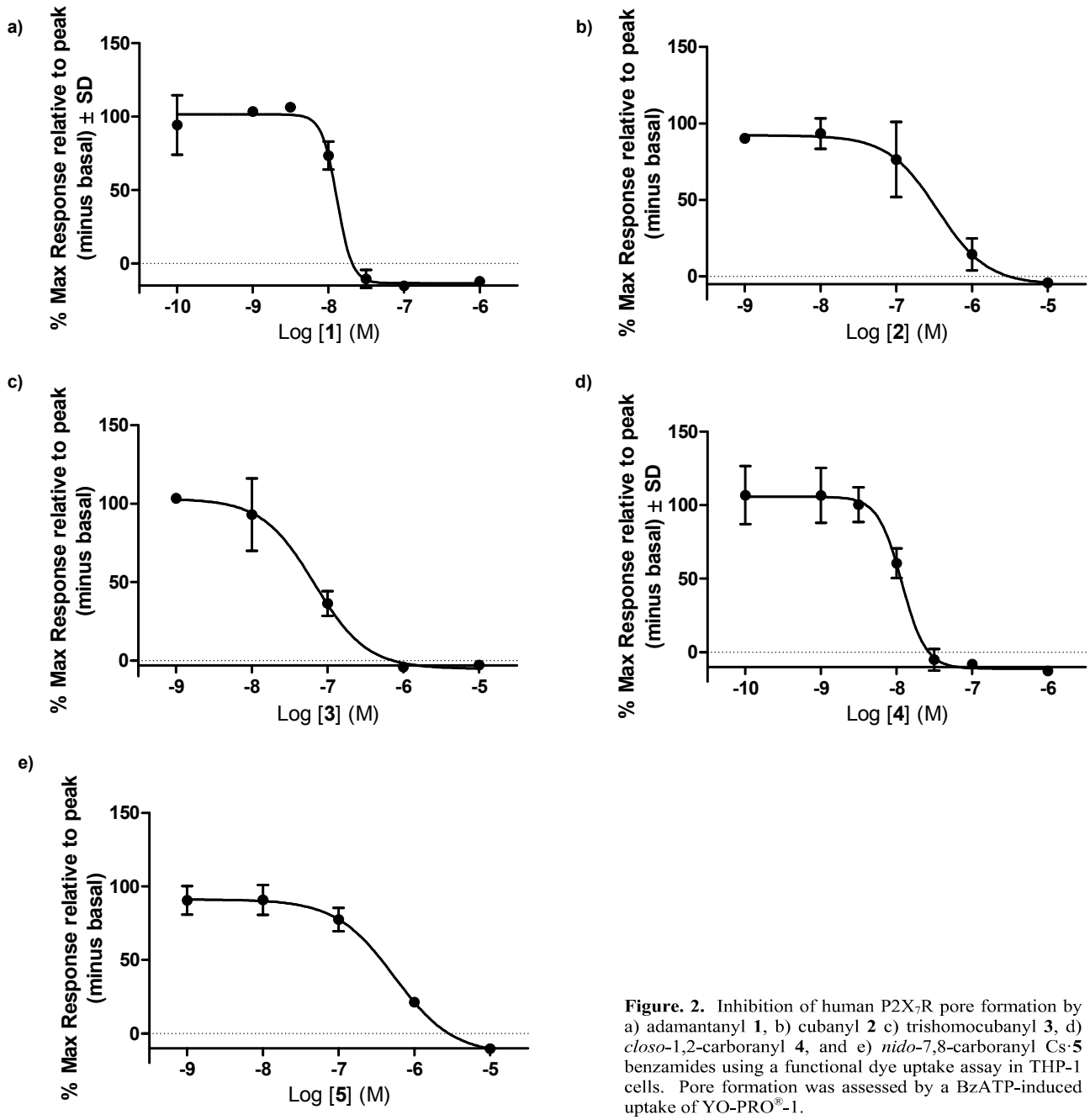


Figure. 2. Inhibition of human $P2X_7R$ pore formation by a) adamantanyl **1**, b) cubanyl **2** c) trishomocubanyl **3**, d) *closo*-1,2-carboranyl **4**, and e) *nido*-7,8-carboranyl Cs·**5** benzamides using a functional dye uptake assay in THP-1 cells. Pore formation was assessed by a BzATP-induced uptake of YO-PRO®-1.

Forced Swim Test

Animals and drug treatment

Wildtype (WT) and $P2X_7^{-/-}$ mice (C57BL6 background) were bred within our facilities at the Brain and Mind Research Institute. Female adult (12-16 weeks) mice were used for all experiments. All animals were group housed in cages of 2-5 under a reversed 12:12 h light-dark cycle with food and water available *ad libitum*. Before all behavioral testing, mice were handled for 3 days to reduce stress and were habituated to the testing room for 1 h before each experiment. All procedures were approved by "the University of Sydney animal ethics committee" and were in accordance with "the Australian code of practice for the care and use of animals for scientific purposes". During both the tolerability study and the FST, animals were separated into different cohorts of mice to ensure they were all aged-matched. Each cohort included all groups of animals (i.e. WT_{VEH}, $WT_{compounds\mathbf{1\text{-}5}}$, and $P2X_7^{-/-}{}_{VEH}$) to avoid the possible confounding effect of different times of testing.

All compounds were dissolved in a vehicle (VEH) suitable for *in vivo* studies that consisted of 8% EtOH, 8% TWEEN® 80 and 84% saline. Drug and VEH treatments were injected intraperitoneally (i.p) at a volume of 10 ml/kg, 20 mins prior to testing in the FST on all 3 days. Compounds **1**, **2**, **4** and **5** were given at the maximal dose studied of 20 mg/kg while **3** was only given at 1 mg/kg as this compound did not completely dissolve at higher doses.

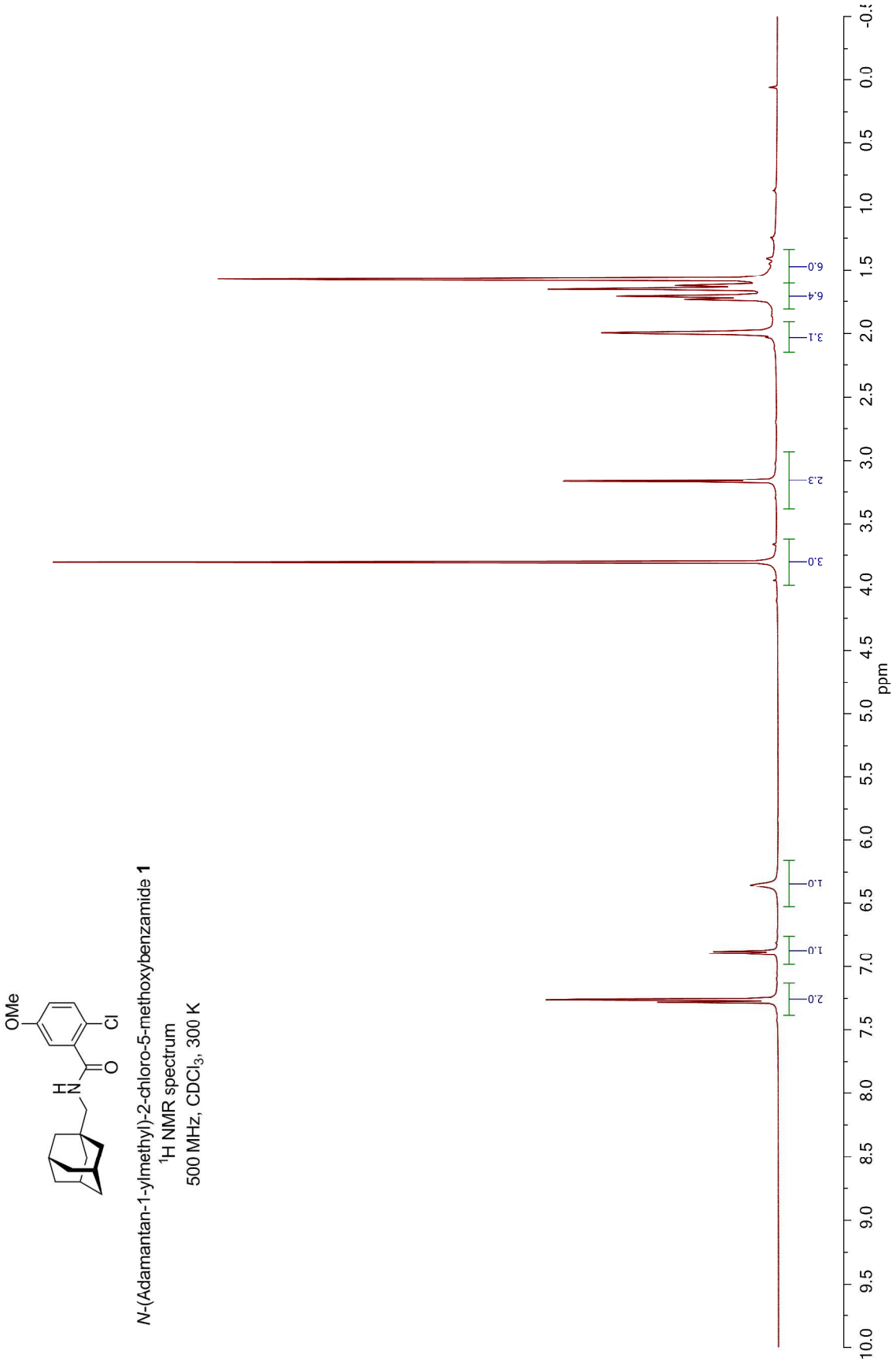
Tolerability Study

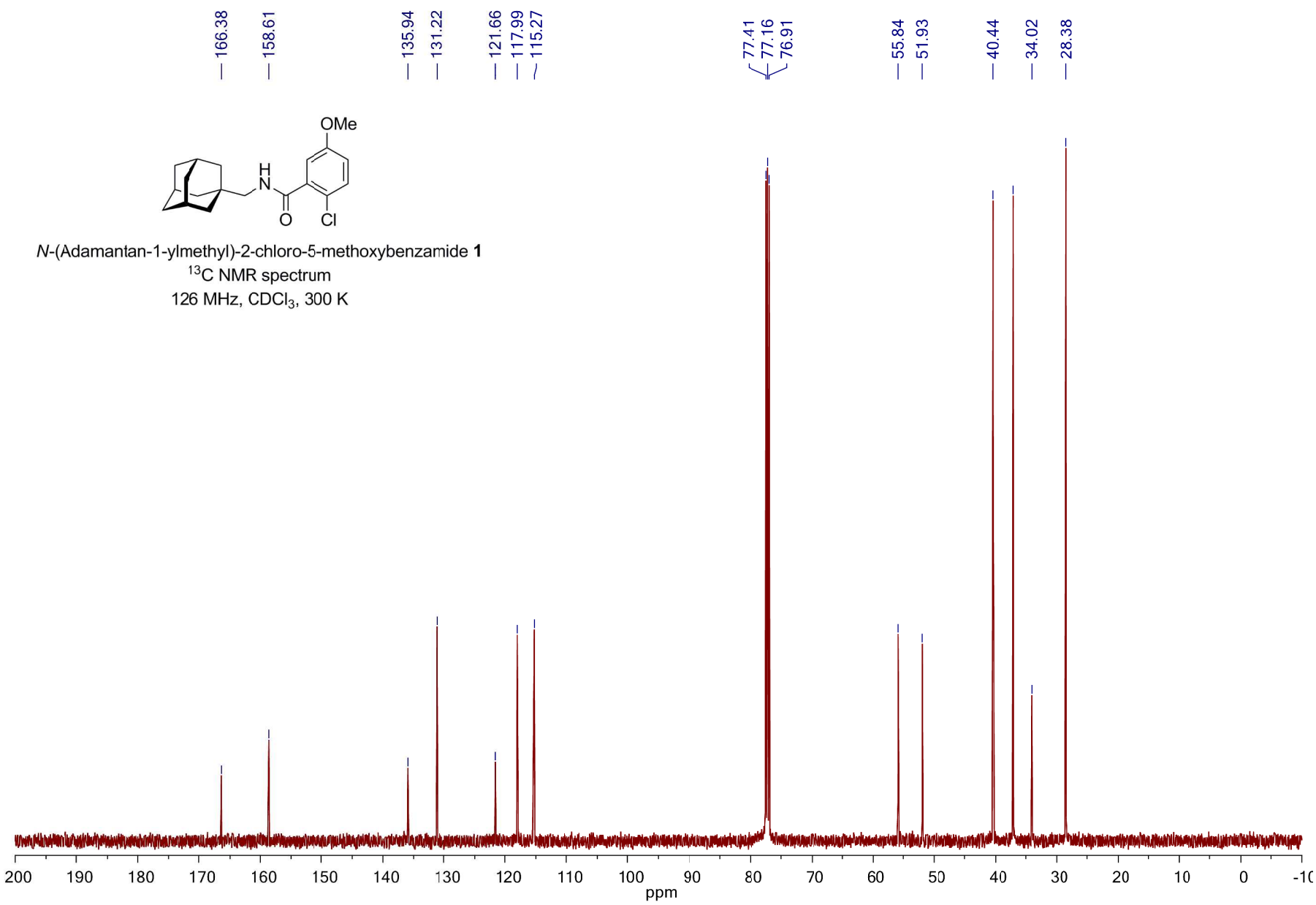
Before assessing the compounds in a large number of animals in our behavioural model of depression, tolerability was assessed in single mice at escalating doses (0.1, 0.5, 1, 5, 10 and 20 mg/kg) compared to a VEH-treated mouse. Animals were weighed and injected with each compound starting from the lowest dose and then monitored closely during a 30 min Normal Behavioral Assessment (NBA) in their home cage. This assessment included recording any abnormalities in posture, activity, gait, respiratory pattern, hydration and bodily condition. At the end of the NBA, body temperature was taken using a mouse rectal probe connected to a thermocouple.[15] Locomotor activity was then assessed for 2 h, before body temperature was taken again. Locomotion was assessed in a perspex open field activity chamber (45 cm x 45 cm x 26 cm) under red light. Distance travelled was recorded by the computer software Trackmate 2.1 (Motion Mensura, Australia).[16] All mice were further monitored for adverse effects for 48 h, including weighing at 24 h.

Forced Swim Test (FST)

Mice were tested in the FST using the same model employed to reveal the antidepressant phenotype of $P2X_7^{-/-}$ mice.[16] Each trial involved the placement of each mouse in a cylindrical container (height: 41 cm; diameter: 17 cm) with a water depth of 10 cm at ambient temperature (24-25 °C). The test duration was 6 min and immobility time was measured as the proportion of time spent in a passive immobile posture, allowing for minimal movements necessary to keep the animals' heads above water.[17] The computer software, Trackmate 2.1 (Motion Mensura, Australia) measured immobility time, which was used as a marker of behavioural despair. The total immobility time (s) each mouse remained stationary over the final 3 min of the Forced-Swim Test (FST) was averaged and compared against the WT cohort using a Mann-Whitney U-test analysis.[18]

^{1}H and ^{13}C NMR spectra

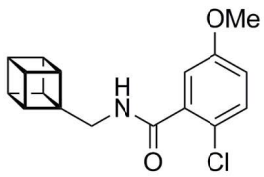




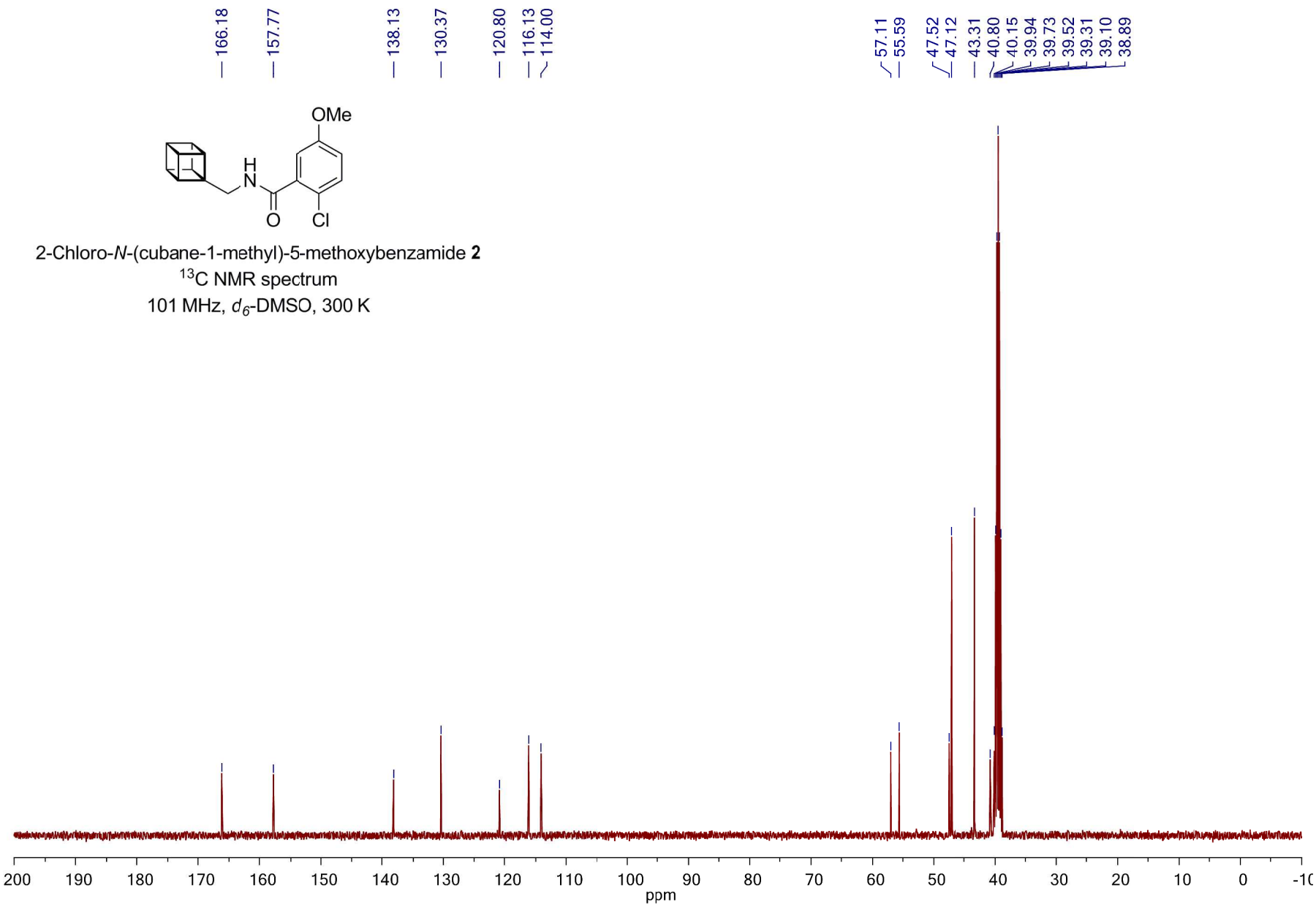
N-(Adamantan-1-ylmethyl)-2-chloro-5-methoxybenzamide **1**

^{13}C NMR spectrum

126 MHz, $CDCl_3$, 300 K



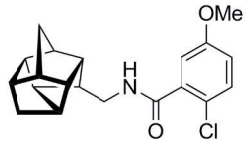
2-Chloro-*N*-(cubane-1-methyl)-5-methoxybenzamide **2**
^{1}H NMR spectrum
400 MHz, d_6-DMSO, 300 K



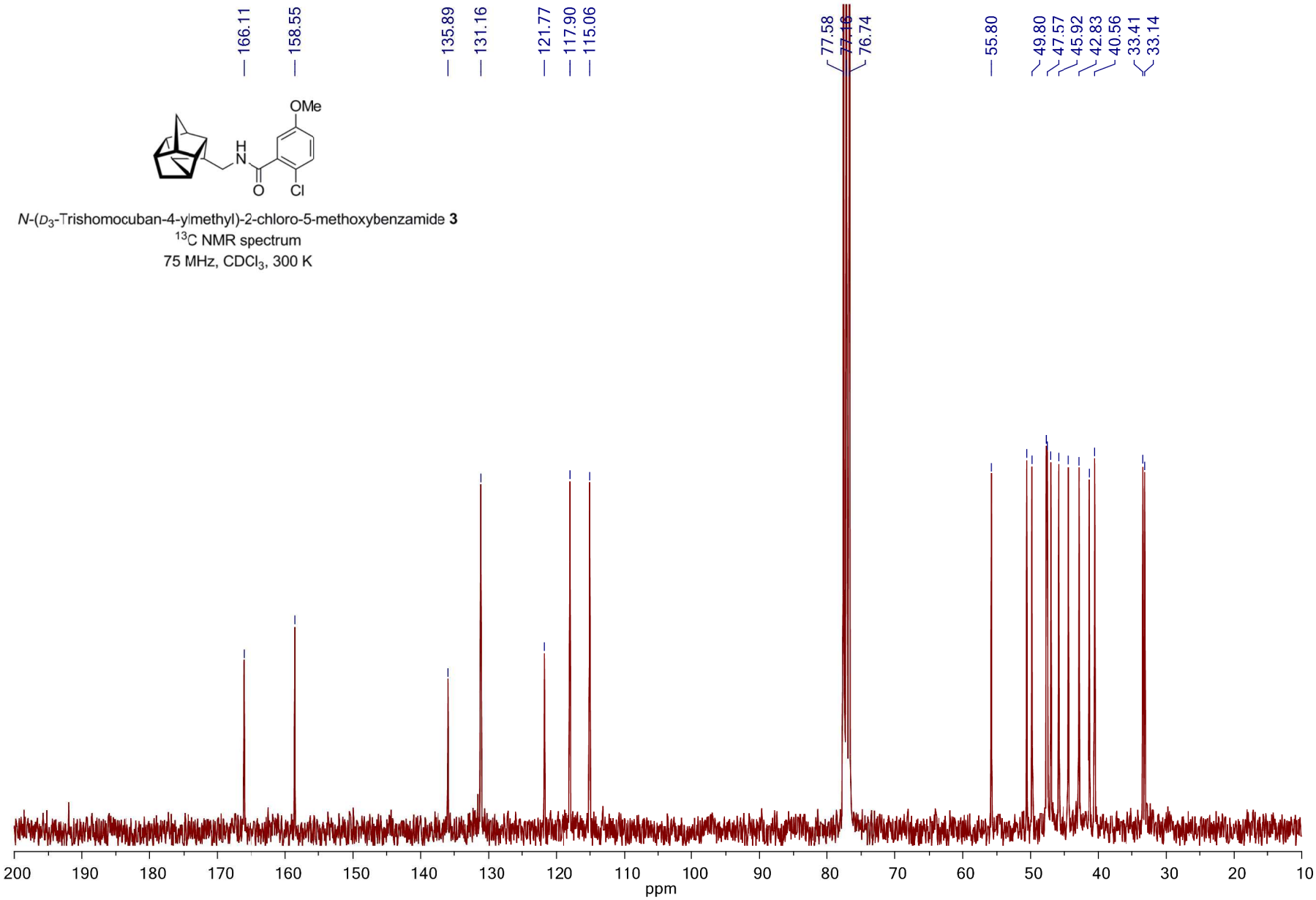
2-Chloro-*N*-(cubane-1-methyl)-5-methoxybenzamide **2**

^{13}C NMR spectrum

101 MHz, d_6-DMSO, 300 K



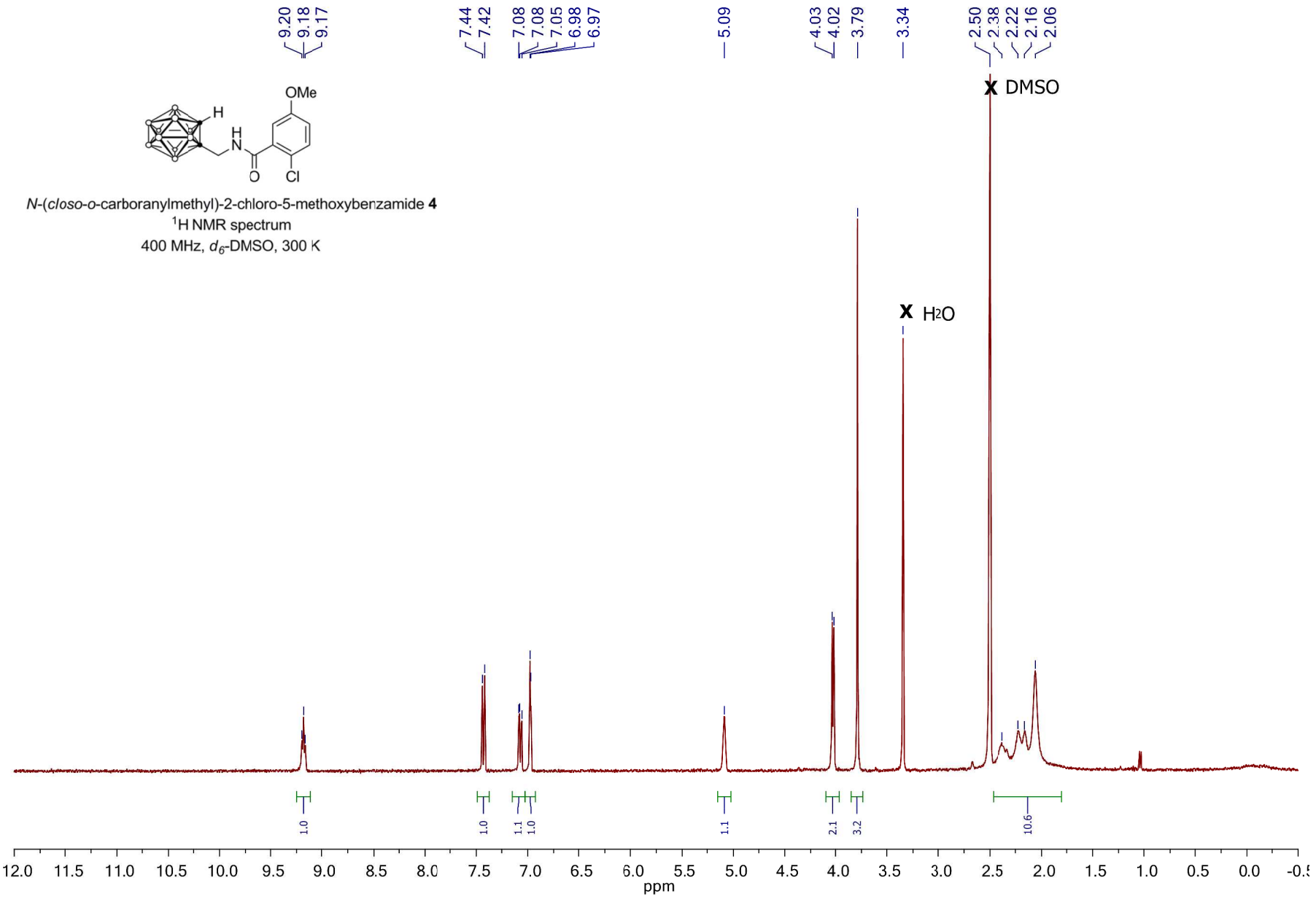
N-(D_3-Trishomocuban-4-ylmethyl)-2-chloro-5-methoxybenzamide **3**
^{1}H NMR spectrum
600 MHz, $CDCl_3$, 303 K



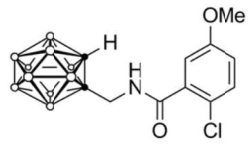
N-(D_3-Trishomocuban-4-ylmethyl)-2-chloro-5-methoxybenzamide **3**

^{13}C NMR spectrum

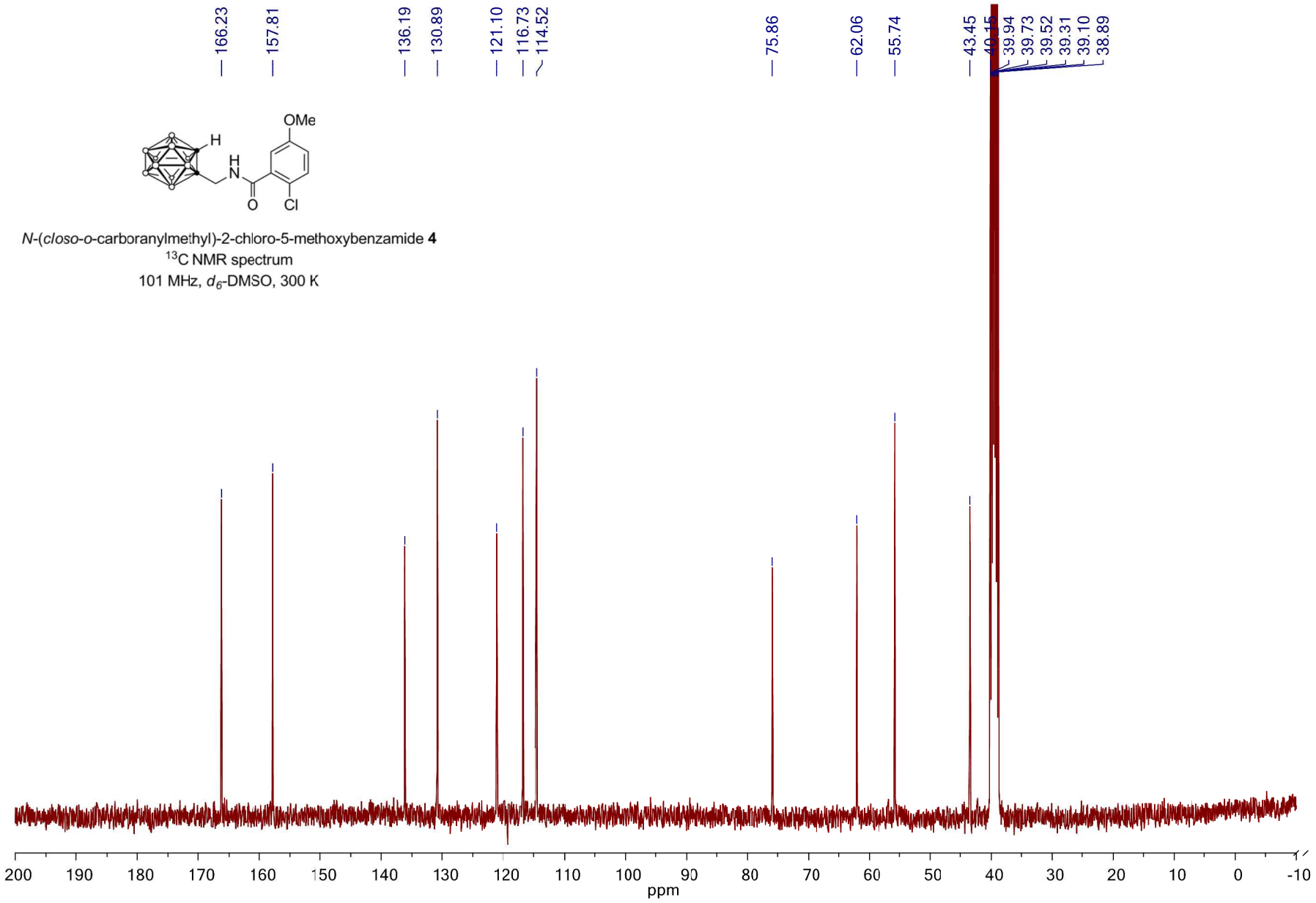
75 MHz, $CDCl_3$, 300 K



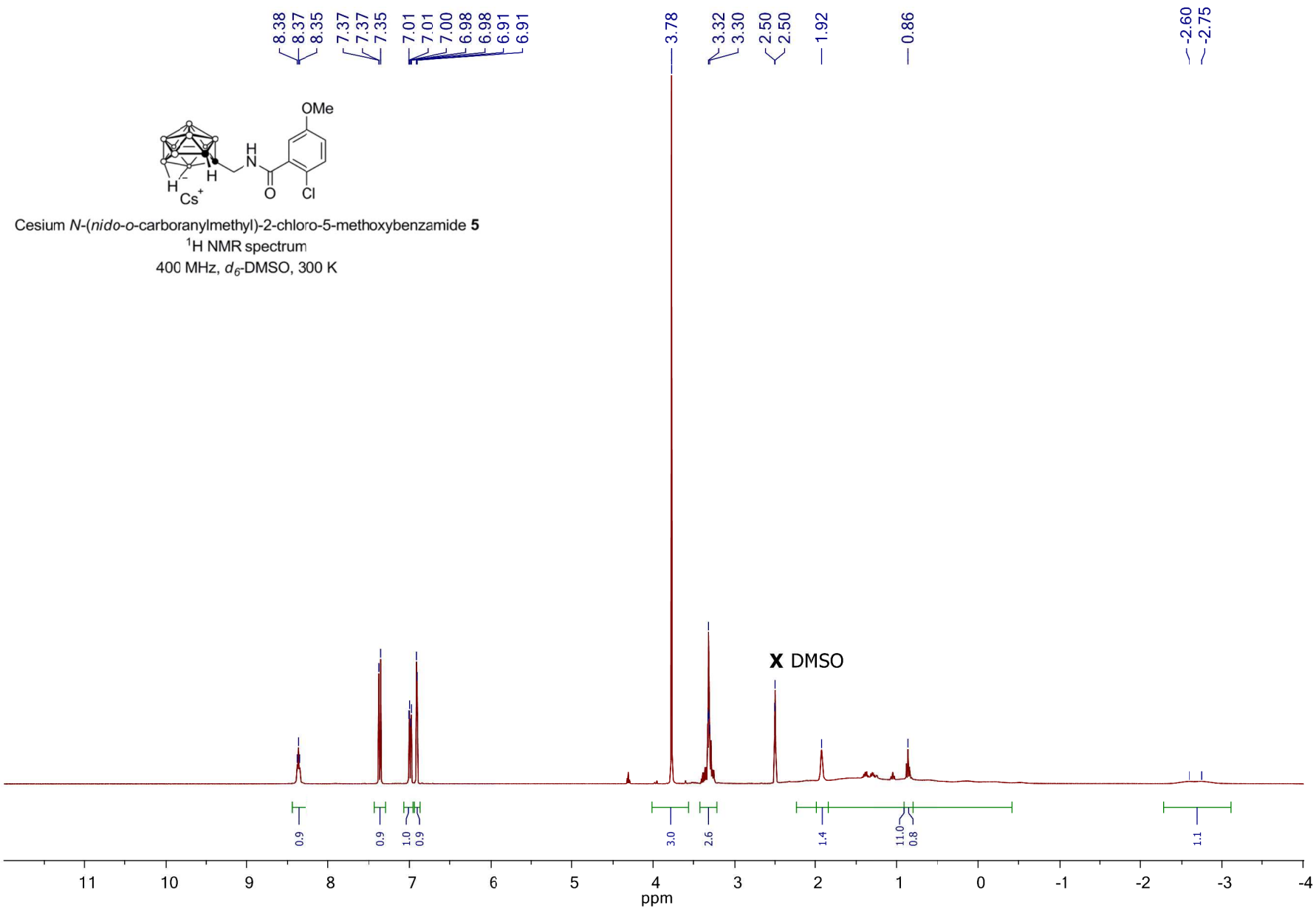
N-(*closo*-*o*-carboranylmethyl)-2-chloro-5-methoxybenzamide **4**
^{1}H NMR spectrum
400 MHz, d_6-DMSO, 300 K



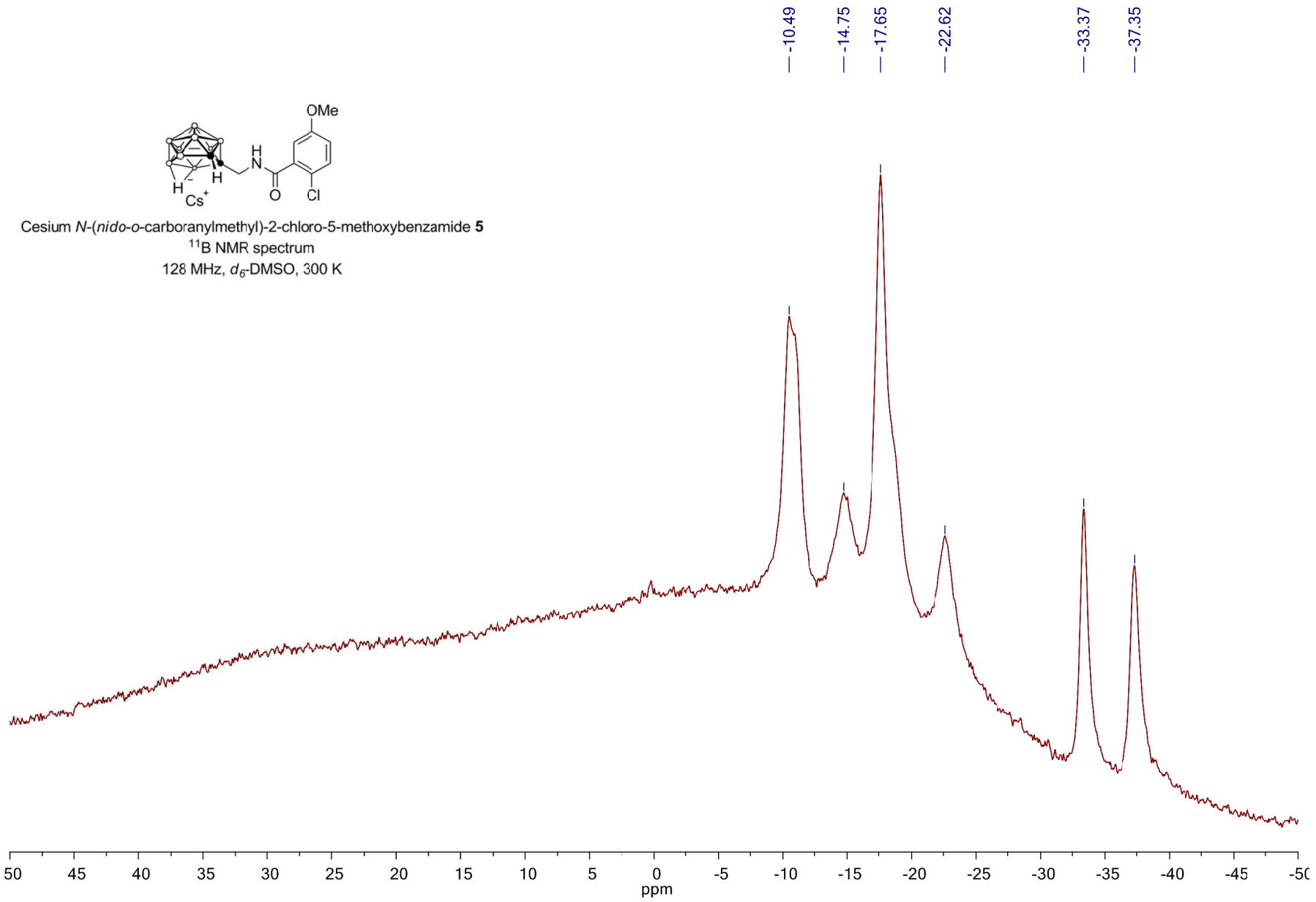
N-(*closo*-*o*-carboranylmethyl)-2-chloro-5-methoxybenzamide **4**
^{11}B NMR spectrum
128 MHz, d_6-DMSO, 300 K



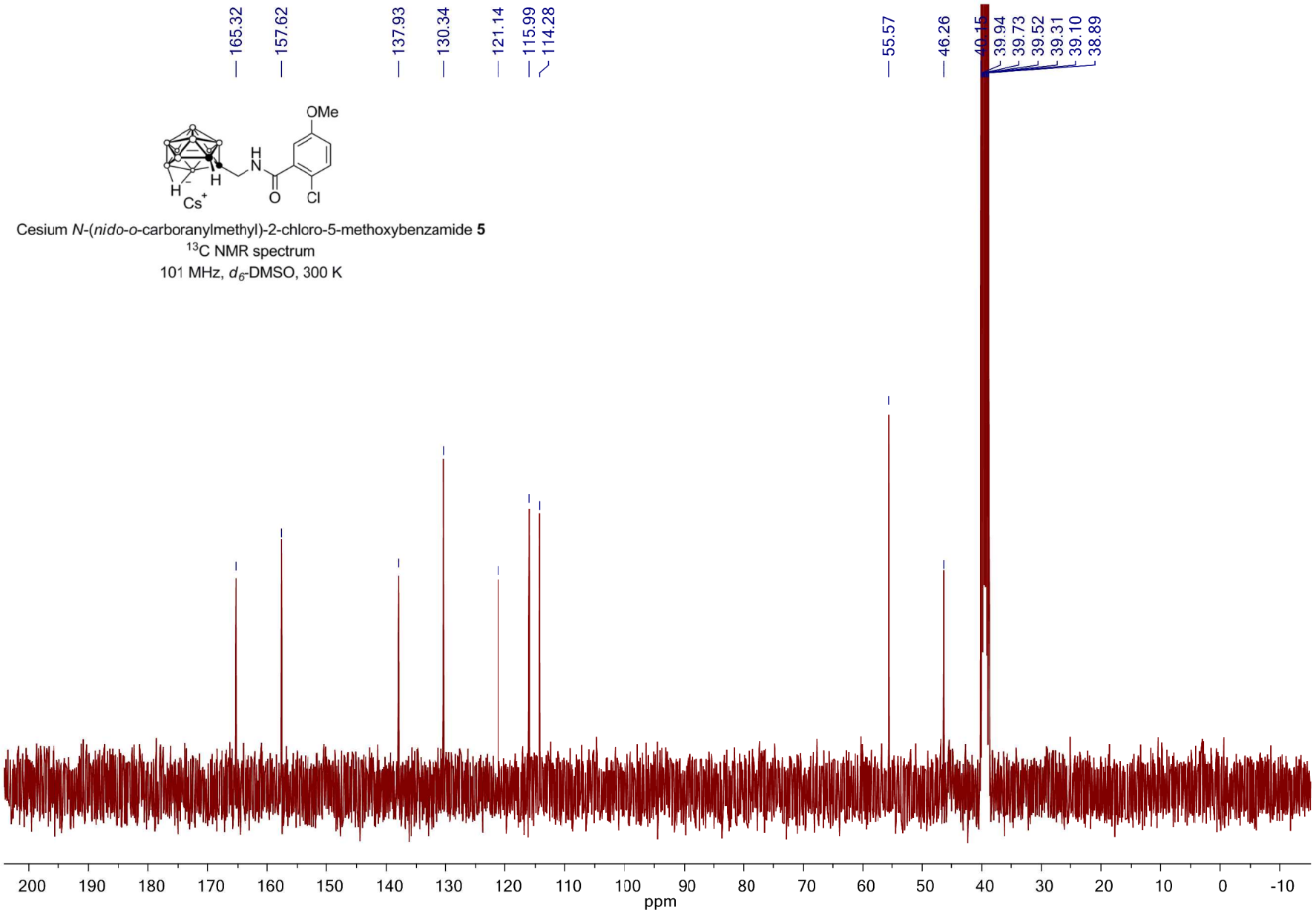
N-(*closo*-*o*-carboranylmethyl)-2-chloro-5-methoxybenzamide **4**
^{13}C NMR spectrum
101 MHz, d_6-DMSO, 300 K



Cesium *N*-(*nido*-*o*-carboranylmethyl)-2-chloro-5-methoxybenzamide **5**
^{1}H NMR spectrum
400 MHz, d_6-DMSO, 300 K



Cesium *N*-(*nido*-*o*-carboranylmethyl)-2-chloro-5-methoxybenzamide **5**
^{11}B NMR spectrum
128 MHz, d_6-DMSO, 300 K



Cesium *N*-(*nido*-*o*-carboranylmethyl)-2-chloro-5-methoxybenzamide **5**

^{13}C NMR spectrum

101 MHz, d_6-DMSO, 300 K

X-Ray Crystal Structure Experimental

Cubanyl benzamide 2

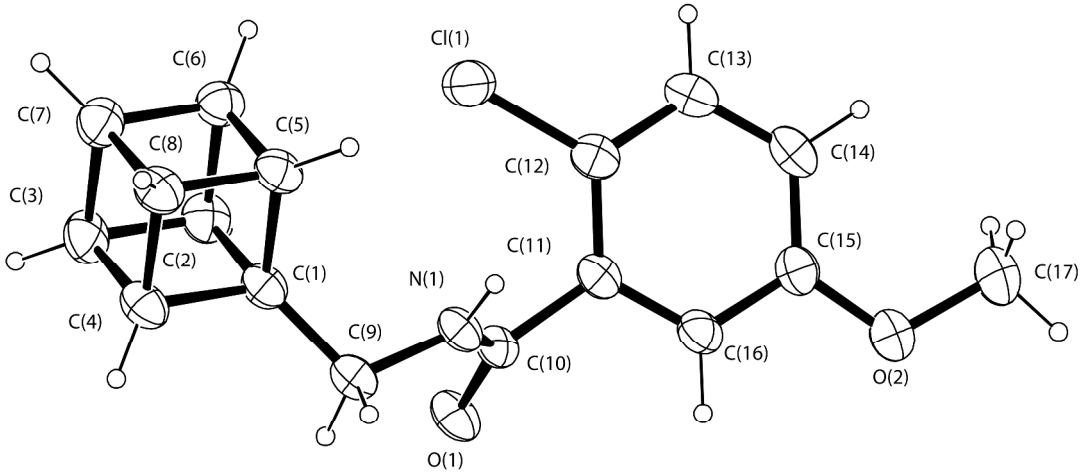


Figure 3. The asymmetric unit for the single crystal structure determination of 2-chloro-*N*-(cubane-1-methyl)-5-methoxybenzamide 2 (CCDC 966907). Anisotropic displacement ellipsoids are given at 50% probability levels for the non-hydrogen atoms.

Experimental Details for 2-chloro-*N*-(cubane-1-methyl)-5-methoxybenzamide (2)

A colorless plate like crystal was attached with Exxon Paratone N, to a short length of fibre supported on a thin piece of copper wire inserted in a copper mounting pin. The crystal was quenched in a cold N_2 gas stream from an Oxford Cryosystems Cryostream A SuperNova Dual equipped with an Atlas detector and employing mirror monochromated Cu(Ka) radiation from a micro-source was used for the data collection. Cell constants were obtained from a least squares refinement against 6986 reflections located between 10 and 148º 2q. Data were collected at 150(2) Kelvin with w scans to 150º 2q. The data processing was undertaken with CrysAlisPro[19] and subsequent computations were carried out with the XP SHELXTL-Plus[20] and WinGX[21] graphical user interfaces. A multi-scan absorption correction was applied to the data.

The structure was solved in the space group *C*12/c1(#15) by direct methods with SIR97[22] and extended and refined with SHELXL-97[23]. The non-hydrogen atoms in the asymmetric unit were modelled with anisotropic displacement parameters and in general a riding atom model was used for the hydrogen atoms. The amide hydrogen site was located in final difference maps and modelled with an isotropic displacement parameter. An ORTEP[24] depiction of the molecule with 50% displacement ellipsoids is provided in **Figure 3**.

Table 2. Results for 2-chloro-*N*-(cubane-1-methyl)-5-methoxybenzamide 2

Formula of the Refinement Model	$C_{17}H_{16}ClNO_2$
Model Molecular Weight	301.76
Crystal System	monoclinic
Space Group	C12/c1(#15)
a	34.4384(13) Å
b	8.3183(3) Å
c	8.3183(3) Å
β	90.322(3)
V	2807.84(17) $Å^3$
D_C	1.428 g cm^{-3}
Z	8
Crystal Size	1.428 x 1.428 x 0.033 mm
Crystal Color	Colorless
Crystal Habit	plate
Temperature	150(2) Kelvin
λ(CuKα)	1.5418 Å
μ(CuKα)	2.439 mm^{-1}
$T_{min,max}$	0.658, 1.00
$2\theta_{max}$	150.00
hkl range	-41 41, -10 10, -12 12
N	12464
N_{ind}	2798(R_{merge} 0.0340)
N_{obs}	2563(I > 2σ(I))
N_{var}	195
Residuals* *R*1(*F*), *wR*2(F^2)	0.0414, 0.1142
GoF(all)	1.230
Residual Extrema	-0.416, 0.732 e^- $Å^{-3}$

*$R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ for $F_o > 2\sigma(F_o)$; $wR2 = (\Sigma w(F_o^2 - F_c^2)^2/\Sigma(wF_c^2)^2)^{½}$ all reflections

$w=1/[\sigma^2 (F_o^2)+(0.05P)^2+0.13P]$ where $P=(F_o^2+2F_c^2)/3$

Table 3. Non-Hydrogen Atom Coordinates, Isotropic Thermal Parameters and Occupancies

Atom	x	y	z	U_{eq}(Å^2)	Occ
Cl(1)	0.086689(13)	0.25771(5)	0.36102(5)	0.03818(16)	1
O(1)	0.15341(4)	0.40480(15)	0.66345(11)	0.0335(3)	1
O(2)	0.24515(4)	-0.00087(18)	0.41467(16)	0.0483(4)	1
N(1)	0.14402(4)	0.53666(17)	0.46216(13)	0.0268(3)	1
C(1)	0.08731(5)	0.7131(2)	0.48394(16)	0.0282(4)	1
C(2)	0.05274(5)	0.6080(2)	0.53709(17)	0.0323(4)	1
C(3)	0.02898(6)	0.7649(2)	0.56874(19)	0.0364(4)	1
C(4)	0.06351(5)	0.8700(2)	0.51549(17)	0.0323(4)	1
C(5)	0.06909(5)	0.7088(2)	0.33632(16)	0.0305(4)	1
C(6)	0.03460(5)	0.6041(2)	0.39020(17)	0.0321(4)	1
C(7)	0.01087(6)	0.7605(2)	0.42204(19)	0.0352(4)	1
C(8)	0.04540(5)	0.8651(2)	0.36814(17)	0.0331(4)	1
C(9)	0.12945(5)	0.6874(2)	0.51902(16)	0.0289(4)	1
C(10)	0.15223(5)	0.4073(2)	0.53729(15)	0.0253(3)	1
C(11)	0.16120(5)	0.25715(19)	0.45734(15)	0.0253(4)	1
C(12)	0.13365(5)	0.1814(2)	0.37378(16)	0.0291(4)	1
C(13)	0.14328(5)	0.0439(2)	0.30161(17)	0.0332(4)	1
C(14)	0.18054(6)	-0.0201(2)	0.31099(17)	0.0334(4)	1
C(15)	0.20785(5)	0.0526(2)	0.39556(17)	0.0322(4)	1
C(16)	0.19789(5)	0.1899(2)	0.46918(16)	0.0299(4)	1
C(17)	0.25820(7)	-0.1295(3)	0.3293(3)	0.0640(7)	1

Table 4. Hydrogen Atom Coordinates, Isotropic Thermal Parameters and Occupancies

Atom	x	y	z	U_{eq} (Å^2)	Occ
H(1N)	0.1449(6)	0.530(3)	0.378(2)	0.031(5)	1
H(2)	0.0553	0.5133	0.5993	0.039	1
H(3)	0.0142	0.7854	0.6546	0.044	1
H(4)	0.0740	0.9681	0.5616	0.039	1
H(5)	0.0838	0.6882	0.2504	0.037	1
H(6)	0.0240	0.5062	0.3440	0.039	1
H(7)	-0.0172	0.7778	0.3995	0.042	1
H(8)	0.0428	0.9596	0.3057	0.040	1
H(9A)	0.1449	0.7781	0.4830	0.035	1
H(9B)	0.1326	0.6858	0.6194	0.035	1
H(13)	0.1244	-0.0069	0.2455	0.040	1
H(14)	0.1873	-0.1130	0.2599	0.040	1
H(16)	0.2165	0.2379	0.5283	0.036	1
H(17A)	0.2422	-0.2250	0.3453	0.096	1
H(17B)	0.2854	-0.1542	0.3508	0.096	1
H(17C)	0.2560	-0.0973	0.2334	0.096	1

Table 5. Anisotropic Thermal Parameters ($Å^2$)

Atom	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Cl(1)	0.0372(3)	0.0359(3)	0.0414(3)	0.00021(16)	-0.00785(19)	-0.00313(17)
O(1)	0.0497(8)	0.0363(7)	0.0144(5)	0.0034(5)	0.0033(5)	0.0004(5)
O(2)	0.0395(8)	0.0488(8)	0.0566(9)	0.0091(6)	-0.0018(6)	-0.0234(7)
N(1)	0.0368(8)	0.0288(7)	0.0147(6)	-0.0006(6)	0.0036(5)	-0.0001(5)
C(1)	0.0357(9)	0.0296(8)	0.0195(7)	-0.0004(7)	0.0013(6)	0.0001(7)
C(2)	0.0364(9)	0.0355(9)	0.0249(8)	-0.0006(7)	0.0025(7)	0.0040(7)
C(3)	0.0371(10)	0.0478(11)	0.0244(8)	0.0047(8)	0.0060(7)	-0.0001(8)
C(4)	0.0419(10)	0.0325(9)	0.0224(8)	0.0020(7)	-0.0007(7)	-0.0020(7)
C(5)	0.0369(9)	0.0345(9)	0.0202(8)	-0.0021(7)	0.0016(6)	-0.0007(7)
C(6)	0.0332(9)	0.0350(9)	0.0281(8)	-0.0025(7)	-0.0002(6)	0.0000(7)
C(7)	0.0338(9)	0.0426(10)	0.0292(9)	0.0032(7)	0.0012(7)	0.0014(7)
C(8)	0.0406(10)	0.0341(9)	0.0247(8)	0.0014(7)	-0.0015(7)	0.0023(7)
C(9)	0.0374(9)	0.0265(8)	0.0229(7)	-0.0014(7)	0.0008(6)	-0.0017(7)
C(10)	0.0283(8)	0.0310(8)	0.0167(7)	-0.0024(6)	0.0042(6)	0.0003(6)
C(11)	0.0334(9)	0.0273(8)	0.0153(7)	-0.0020(6)	0.0048(6)	0.0031(6)
C(12)	0.0357(9)	0.0296(9)	0.0219(7)	-0.0018(7)	-0.0008(6)	0.0030(7)
C(13)	0.0437(10)	0.0293(9)	0.0266(8)	-0.0038(7)	-0.0040(7)	-0.0018(7)
C(14)	0.0465(10)	0.0292(8)	0.0246(8)	-0.0004(7)	0.0052(7)	-0.0039(7)
C(15)	0.0338(9)	0.0346(9)	0.0283(8)	0.0014(7)	0.0048(7)	-0.0018(7)
C(16)	0.0338(9)	0.0335(9)	0.0225(7)	-0.0012(7)	0.0022(6)	-0.0034(7)
C(17)	0.0472(13)	0.0620(15)	0.0828(18)	0.0133(11)	0.0002(12)	-0.0397(14)

Table 6. Non Hydrogen Bond Lengths (Å)

Atom	Atom	Distance	Atom	Atom	Distance
O(1)	N(1)	1.4010(13)	O(2)	C(1)	1.4236(15)
O(21)	N(21)	1.4049(12)	O(22)	C(21)	1.4289(14)
N(1)	C(8)	1.4985(15)	N(1)	C(9)	1.4990(14)
N(1)	C(2)	1.5435(13)	N(21)	C(28)	1.4927(16)
N(21)	C(29)	1.4951(16)	N(21)	C(22)	1.5396(14)
C(1)	C(7)	1.5276(16)	C(1)	C(6)	1.5416(16)
C(1)	C(2)	1.5552(14)	C(2)	C(3)	1.5223(16)
C(3)	C(4)	1.5451(16)	C(4)	C(10)	1.5187(17)
C(4)	C(5)	1.5342(16)	C(5)	C(6)	1.5224(18)
C(10)	C(11)	1.3290(18)	C(10)	C(12)	1.5041(16)
C(21)	C(26)	1.5302(18)	C(21)	C(27)	1.5333(18)
C(21)	C(22)	1.5523(15)	C(22)	C(23)	1.5284(14)
C(23)	C(24)	1.5419(16)	C(24)	C(31)	1.5213(16)
C(24)	C(25)	1.5321(16)	C(25)	C(26)	1.5278(17)
C(31)	C(32)	1.325(2)	C(31)	C(33)	1.5053(19)

Symmetry Operators

(1) x, y, z (2) -x, y+1/2, -z

Table 7. Non Hydrogen Bond Angles (º)

Atom	Atom	Atom	Angle	Atom	Atom	Atom	Angle
O(1)	N(1)	C(8)	108.27(9)	C(5)	C(4)	C(3)	107.77(10)
O(1)	N(1)	C(9)	107.77(9)	C(6)	C(5)	C(4)	111.63(9)
C(8)	N(1)	C(9)	107.93(9)	C(5)	C(6)	C(1)	115.34(10)
O(1)	N(1)	C(2)	110.50(8)	C(11)	C(10)	C(12)	120.10(12)
C(8)	N(1)	C(2)	113.72(9)	C(11)	C(10)	C(4)	124.13(11)
C(9)	N(1)	C(2)	108.47(8)	C(12)	C(10)	C(4)	115.61(11)
O(21)	N(21)	C(28)	107.80(8)	O(22)	C(21)	C(26)	105.56(10)
O(21)	N(21)	C(29)	108.12(9)	O(22)	C(21)	C(27)	109.36(10)
C(28)	N(21)	C(29)	107.50(10)	C(26)	C(21)	C(27)	110.30(11)
O(21)	N(21)	C(22)	110.51(8)	O(22)	C(21)	C(22)	109.04(9)
C(28)	N(21)	C(22)	113.83(9)	C(26)	C(21)	C(22)	106.79(9)
C(29)	N(21)	C(22)	108.89(8)	C(27)	C(21)	C(22)	115.32(10)
O(2)	C(1)	C(7)	108.94(10)	C(23)	C(22)	N(21)	111.13(9)
O(2)	C(1)	C(6)	105.91(9)	C(23)	C(22)	C(21)	112.22(9)
C(7)	C(1)	C(6)	110.75(10)	N(21)	C(22)	C(21)	114.98(8)
O(2)	C(1)	C(2)	109.28(9)	C(22)	C(23)	C(24)	111.04(9)
C(7)	C(1)	C(2)	115.42(10)	C(31)	C(24)	C(25)	115.03(10)
C(6)	C(1)	C(2)	106.09(9)	C(31)	C(24)	C(23)	113.67(9)
C(3)	C(2)	N(1)	111.57(9)	C(25)	C(24)	C(23)	108.10(9)
C(3)	C(2)	C(1)	112.72(8)	C(26)	C(25)	C(24)	111.69(9)
N(1)	C(2)	C(1)	114.64(9)	C(25)	C(26)	C(21)	113.69(11)
C(2)	C(3)	C(4)	110.58(9)	C(32)	C(31)	C(33)	119.99(12)
C(10)	C(4)	C(5)	115.21(10)	C(32)	C(31)	C(24)	124.24(12)
C(10)	C(4)	C(3)	112.73(9)	C(33)	C(31)	C(24)	115.69(11)

Symmetry Operators

(1) x, y, z (2) -x, y+1/2, -z

Table 8. Hydrogen Bond Geometry

Donor	Hydrogen	Acceptor	D-H (Å)	H-A (Å)	D-A (Å)	DHA Angle (º)
O(2)	H(2)	O(1)	0.85(2)	1.78(2)	2.5589(12)	151.2(18)
O(2)	H(2)	N(1)	0.85(2)	2.51(2)	3.0184(13)	118.9(15)
O(22)	H(22)	O(21)	0.91(2)	1.72(2)	2.5675(14)	154.3(18)
O(22)	H(22)	N(21)	0.91(2)	2.45(2)	3.0177(14)	120.6(15)
O(31)	H(31A)	O(21)	0.89(2)	1.94(2)	2.8301(15)	176(2)
O(31)	H(31B)	O(1)	0.87(2)	1.99(2)	2.8517(14)	170.2(19)

Symmetry Operators

(1) x, y, z (2) -x, y+1/2, -z

***closo o*-Carboranyl benzamide 4**

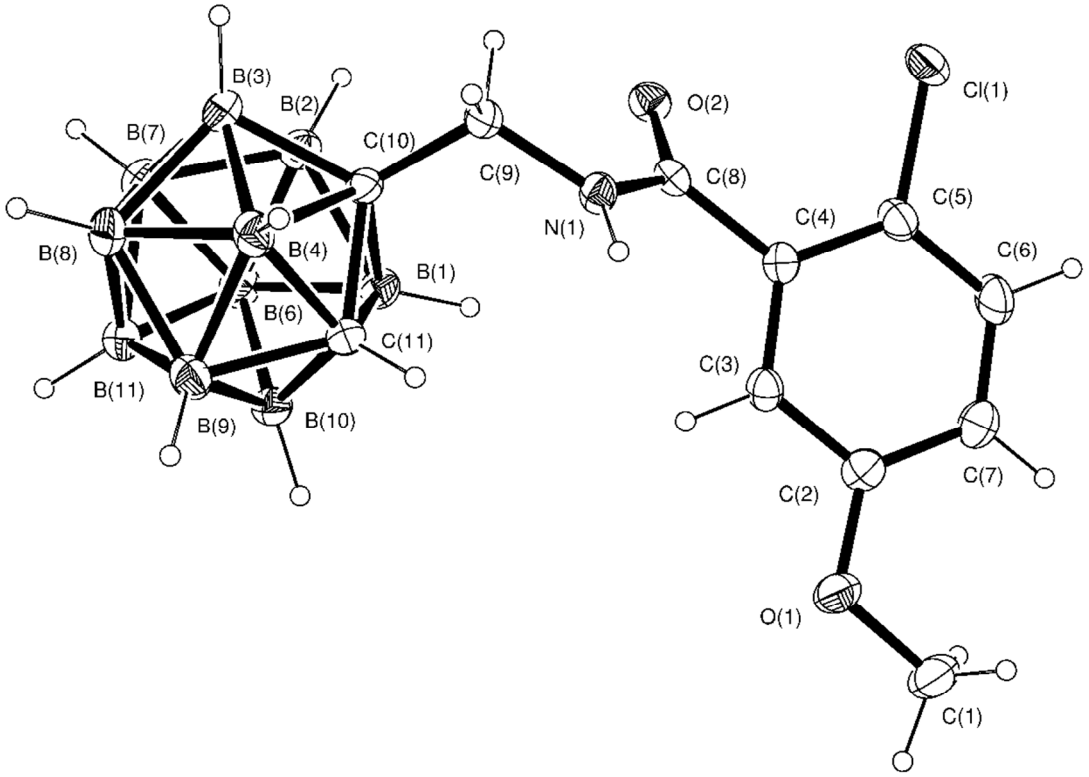


Figure 4. The asymmetric unit for the single crystal structure determination of *N*-(*closo*-1,2-carboranylmethyl)-2-chloro-5-methoxybenzamide 4 (CCDC 966908). Anistropic displacement ellipsoids are given at 50% probability levels for the non-hydrogen atoms.

Experimental Details for *N*-(*closo*-1,2-carboranylmethyl)-2-chloro-5-methoxybenzamide (4)

A colorless plate-like crystal was attached with Exxon Paratone N, to a short length of fibre supported on a thin piece of copper wire inserted in a copper mounting pin. The crystal was quenched in a cold nitrogen gas stream from an Oxford Cryosystems Cryostream. A APEXII-FR591 diffractometer employing graphite monochromated MoKα radiation generated from a rotating anode was used for the data collection. Cell constants were obtained from a least squares refinement against 4031 reflections located between 4.9 and 64.5º 2θ. Data were collected at 150(2) Kelvin with ω+ϕ scans to 55.00º 2θ. The data integration and reduction were undertaken with SAINT and XPREP,[25] and subsequent computations were carried out with the XP SHELXTL-Plus[26] and WinGX[21] graphical user interfaces.

The structure was solved in the space group *P*ccn(#56) by direct methods with SHELXS-97,[23] and extended and refined with SHELXL-97.[23] The non-hydrogen atoms in the asymmetric unit were modelled with anisotropic displacement parameters. Of the 20 hydrogen atoms included in the model the 11 attached to the carborane cluster were located and modelled with isotropic displacement parameters, and a riding atom model was used for the remainder. An ORTEP[27] depiction of the molecule with 50% displacement ellipsoids is provided in **Figure 4**.

Table 9. Results for *N*-(*closo*-1,2-carboranylmethyl)-2-chloro-5-methoxybenzamide 4

Formula of the Refinement Model	$C_{11}H_{20}B_{10}ClNO_2$
Model Molecular Weight	341.83
Crystal System	orthorhombic
Space Group	*P*ccn(#56)
a	16.5319(10) Å
b	21.1019(12) Å
c	10.0474(6) Å
V	3505.1(4) $Å^3$
D_C	1.296 g cm^{-3}
Z	8
Crystal Size	0.312x0.120x0.058 mm
Crystal Color	colorless
Crystal Habit	plate
Temperature	150(2) Kelvin
λ(CuKα)	0.71069 Å
μ(CuKα)	0.221 mm^{-1}
$2\theta_{max}$	55.0º
hkl range	-21 21, -26 27, -13 12
N	56954
N_{ind}	4031(R_{merge} 0.0276)
N_{obs}	3706(I > 2σ(I))
N_{var}	271
Residuals* R1(F), wR2(F^2)	0.0316, 0.0915
GoF(all)	1.067
Residual Extrema	-0.249, 0.354 e^- $Å^{-3}$

*$R1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ for $F_o > 2\sigma(F_o)$; $wR2 = (\Sigma w(F_o^2 - F_c^2)^2/\Sigma(wF_c^2)^2)^{1/2}$ all reflections

$w=1/[\sigma^2 (F_o^2)+(0.05P)^2+0.13P]$ where $P=(F_o^2+2F_c^2)/3$

Table 10. Non-Hydrogen Atom Coordinates, Isotropic Thermal Parameters and Occupancies

Atom	x	y	z	U_{eq}(Å^2)	Occ
Cl(1)	0.239946(18)	0.668008(14)	0.13203(3)	0.03245(10)	1
C(1)	0.08659(8)	0.48517(6)	0.61493(13)	0.0321(3)	1
O(1)	0.07189(6)	0.55079(4)	0.58493(8)	0.0303(2)	1
C(2)	0.11232(7)	0.57539(5)	0.47833(11)	0.0221(2)	1
C(3)	0.09210(6)	0.63769(5)	0.44387(10)	0.0195(2)	1
C(4)	0.12972(6)	0.66775(5)	0.33713(10)	0.0174(2)	1
C(5)	0.18850(6)	0.63419(5)	0.26530(11)	0.0222(2)	1
C(6)	0.20899(7)	0.57299(5)	0.30054(13)	0.0296(3)	1
C(7)	0.17099(8)	0.54301(5)	0.40659(12)	0.0282(3)	1
C(8)	0.10424(6)	0.73363(5)	0.29824(10)	0.0166(2)	1
O(2)	0.08680(4)	0.74730(4)	0.18215(7)	0.02072(17)	1
N(1)	0.09955(5)	0.77588(4)	0.39809(9)	0.01808(19)	1
C(9)	0.07248(6)	0.84047(5)	0.37565(11)	0.0198(2)	1
C(10)	-0.01736(6)	0.85161(5)	0.40851(10)	0.0171(2)	1
C(11)	-0.05581(6)	0.81808(5)	0.54134(10)	0.0189(2)	1
B(1)	-0.08481(7)	0.79003(6)	0.38777(12)	0.0200(2)	1
B(2)	-0.08953(8)	0.85897(6)	0.28739(12)	0.0218(2)	1
B(3)	-0.05851(8)	0.92498(6)	0.38608(13)	0.0231(3)	1
B(4)	-0.03526(8)	0.89746(6)	0.54929(12)	0.0221(2)	1
B(6)	-0.17918(8)	0.82682(6)	0.35869(13)	0.0231(3)	1
B(7)	-0.16336(8)	0.91036(6)	0.35765(13)	0.0247(3)	1
B(8)	-0.12930(8)	0.93429(6)	0.51910(14)	0.0255(3)	1
B(9)	-0.12450(8)	0.86516(6)	0.61999(12)	0.0238(3)	1
B(10)	-0.15459(7)	0.79890(6)	0.52139(13)	0.0221(2)	1
B(11)	-0.20400(8)	0.87324(6)	0.50207(13)	0.0238(3)	1

Table 11. Hydrogen Atom Coordinates, Isotropic Thermal Parameters and Occupancies

Atom	x	y	z	U_{eq}(Å^2)	Occ
H(1A)	0.0751	0.4593	0.5361	0.048	1
H(1B)	0.0514	0.4719	0.6883	0.048	1
H(1C)	0.1433	0.4796	0.6408	0.048	1
H(3A)	0.0523	0.6597	0.4939	0.023	1
H(6A)	0.2495	0.5511	0.2517	0.035	1
H(7A)	0.1850	0.5008	0.4298	0.034	1
H(1D)	0.1132	0.7641	0.4790	0.022	1
H(9A)	0.0819	0.8515	0.2811	0.024	1
H(9B)	0.1057	0.8694	0.4307	0.024	1
H(11A)	-0.0188(9)	0.7912(7)	0.5896(15)	0.031(4)	1
H(1)	-0.0611(9)	0.7445(6)	0.3586(14)	0.027(4)	1
H(2)	-0.0698(9)	0.8569(7)	0.1836(15)	0.032(4)	1
H(3)	-0.0201(9)	0.9611(7)	0.3461(15)	0.032(4)	1
H(4)	0.0174(9)	0.9119(7)	0.6068(13)	0.028(4)	1
H(6)	-0.2246(8)	0.8039(7)	0.2980(14)	0.030(4)	1
H(7)	-0.1999(9)	0.9415(7)	0.2942(15)	0.035(4)	1
H(8)	-0.1407(9)	0.9815(7)	0.5593(15)	0.035(4)	1
H(9)	-0.1269(9)	0.8618(7)	0.7270(15)	0.036(4)	1
H(10)	-0.1745(9)	0.7567(7)	0.5686(15)	0.029(4)	1
H(11)	-0.2658(8)	0.8807(6)	0.5329(14)	0.027(3)	1

Table 12. Anisotropic Thermal Parameters ($Å^2$)

Atom	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Cl(1)	0.02985(17)	0.02660(16)	0.04091(19)	0.00225(11)	0.01958(12)	0.00123(11)
C(1)	0.0432(7)	0.0226(6)	0.0304(6)	0.0032(5)	0.0016(5)	0.0069(5)
O(1)	0.0433(5)	0.0232(4)	0.0245(4)	0.0066(4)	0.0076(4)	0.0055(3)
C(2)	0.0264(5)	0.0210(5)	0.0188(5)	0.0003(4)	-0.0014(4)	-0.0010(4)
C(3)	0.0208(5)	0.0207(5)	0.0170(5)	0.0035(4)	-0.0002(4)	-0.0034(4)
C(4)	0.0174(5)	0.0175(5)	0.0173(5)	0.0008(4)	-0.0019(4)	-0.0040(4)
C(5)	0.0208(5)	0.0205(5)	0.0251(5)	-0.0010(4)	0.0056(4)	-0.0036(4)
C(6)	0.0288(6)	0.0212(5)	0.0387(7)	0.0055(4)	0.0105(5)	-0.0053(5)
C(7)	0.0329(6)	0.0173(5)	0.0343(6)	0.0045(4)	0.0034(5)	-0.0013(4)
C(8)	0.0134(4)	0.0196(5)	0.0167(5)	-0.0008(4)	0.0018(3)	-0.0015(4)
O(2)	0.0227(4)	0.0245(4)	0.0150(3)	0.0001(3)	0.0001(3)	-0.0003(3)
N(1)	0.0211(4)	0.0179(4)	0.0153(4)	0.0028(3)	0.0000(3)	-0.0015(3)
C(9)	0.0205(5)	0.0159(5)	0.0230(5)	0.0001(4)	0.0042(4)	-0.0009(4)
C(10)	0.0211(5)	0.0138(4)	0.0165(5)	-0.0003(4)	0.0020(4)	0.0000(4)
C(11)	0.0202(5)	0.0191(5)	0.0176(5)	0.0014(4)	0.0006(4)	0.0025(4)
B(1)	0.0218(5)	0.0159(5)	0.0223(6)	-0.0015(4)	-0.0002(4)	-0.0017(4)
B(2)	0.0252(6)	0.0221(6)	0.0182(5)	0.0013(5)	-0.0009(4)	0.0020(4)
B(3)	0.0275(6)	0.0145(5)	0.0274(6)	0.0020(5)	0.0039(5)	0.0021(4)
B(4)	0.0244(6)	0.0195(5)	0.0224(6)	0.0001(4)	0.0025(5)	-0.0059(5)
B(6)	0.0210(6)	0.0230(6)	0.0254(6)	0.0006(5)	-0.0025(5)	0.0005(5)
B(7)	0.0255(6)	0.0217(6)	0.0268(6)	0.0055(5)	0.0003(5)	0.0060(5)
B(8)	0.0275(6)	0.0192(6)	0.0298(6)	0.0047(5)	0.0049(5)	-0.0034(5)
B(9)	0.0243(6)	0.0266(6)	0.0204(6)	0.0037(5)	0.0040(5)	-0.0011(5)
B(10)	0.0194(5)	0.0212(6)	0.0259(6)	0.0004(4)	0.0022(5)	0.0047(5)
B(11)	0.0214(6)	0.0240(6)	0.0261(6)	0.0043(5)	0.0022(5)	0.0030(5)

Table 13. Non Hydrogen Bond Lengths (Å)

Atom	Atom	Distance	Atom	Atom	Distance
Cl(1)	C(5)	1.7394(11)	C(1)	O(1)	1.4377(14)
O(1)	C(2)	1.3650(14)	C(2)	C(7)	1.3882(16)
C(2)	C(3)	1.4000(15)	C(3)	C(4)	1.3926(15)
C(4)	C(5)	1.4023(14)	C(4)	C(8)	1.5044(14)
C(5)	C(6)	1.3813(16)	C(6)	C(7)	1.3894(17)
C(8)	O(2)	1.2357(12)	C(8)	N(1)	1.3443(13)
N(1)	C(9)	1.4522(13)	C(9)	C(10)	1.5394(14)
C(10)	C(11)	1.6389(14)	C(10)	B(3)	1.7059(15)
C(10)	B(2)	1.7113(16)	C(10)	B(1)	1.7251(16)
C(10)	B(4)	1.7391(15)	C(11)	B(10)	1.6943(16)
C(11)	B(9)	1.7031(16)	C(11)	B(4)	1.7110(16)
C(11)	B(1)	1.7208(16)	B(1)	B(6)	1.7669(18)
B(1)	B(2)	1.7719(17)	B(1)	B(10)	1.7799(17)
B(2)	B(7)	1.7788(18)	B(2)	B(6)	1.7804(18)
B(2)	B(3)	1.7850(18)	B(3)	B(4)	1.7816(18)
B(3)	B(7)	1.7837(19)	B(3)	B(8)	1.7874(18)
B(4)	B(8)	1.7643(18)	B(4)	B(9)	1.7736(18)
B(6)	B(7)	1.7822(18)	B(6)	B(10)	1.7845(18)
B(6)	B(11)	1.7898(18)	B(7)	B(11)	1.7805(18)
B(7)	B(8)	1.7899(19)	B(8)	B(9)	1.7782(19)
B(8)	B(11)	1.7928(19)	B(9)	B(11)	1.7777(19)
B(9)	B(10)	1.7842(18)	B(10)	B(11)	1.7792(17)

Symmetry Operators

(1) x, y, z	(2) -x+1/2, -y+1/2, z	(3) -x, y+1/2, -z+1/2	(4) x+1/2, -y, -z+1/2
(5) -x, -y, -z	(6) x-1/2, y-1/2, -z	(7) x, -y-1/2, z-1/2	(8) -x-1/2, y, z-1/2

Table 14. Non Hydrogen Bond Angles (°)

Atom	**Atom**	**Atom**	**Angle**	**Atom**	**Atom**	**Atom**	**Angle**
C(2)	O(1)	C(1)	116.62(9)	B(2)	B(3)	B(8)	108.25(9)
O(1)	C(2)	C(7)	124.22(10)	C(11)	B(4)	C(10)	56.71(6)
O(1)	C(2)	C(3)	115.74(10)	C(11)	B(4)	B(8)	104.37(9)
C(7)	C(2)	C(3)	120.04(10)	C(10)	B(4)	B(8)	104.79(9)
C(4)	C(3)	C(2)	120.77(10)	C(11)	B(4)	B(9)	58.48(7)
C(3)	C(4)	C(5)	118.41(9)	C(10)	B(4)	B(9)	104.68(8)
C(3)	C(4)	C(8)	119.73(9)	B(8)	B(4)	B(9)	60.35(7)
C(5)	C(4)	C(8)	121.80(9)	C(11)	B(4)	B(3)	103.49(8)
C(6)	C(5)	C(4)	120.67(10)	C(10)	B(4)	B(3)	57.95(6)
C(6)	C(5)	Cl(1)	117.45(8)	B(8)	B(4)	B(3)	60.54(7)
C(4)	C(5)	Cl(1)	121.86(8)	B(9)	B(4)	B(3)	108.33(9)
C(5)	C(6)	C(7)	120.75(10)	B(1)	B(6)	B(2)	59.93(7)
C(2)	C(7)	C(6)	119.35(10)	B(1)	B(6)	B(7)	107.82(9)
O(2)	C(8)	N(1)	122.43(10)	B(2)	B(6)	B(7)	59.91(7)
O(2)	C(8)	C(4)	121.75(9)	B(1)	B(6)	B(10)	60.16(7)
N(1)	C(8)	C(4)	115.79(9)	B(2)	B(6)	B(10)	107.74(9)
C(8)	N(1)	C(9)	121.62(9)	B(7)	B(6)	B(10)	107.36(9)
N(1)	C(9)	C(10)	114.04(8)	B(1)	B(6)	B(11)	108.05(9)
C(9)	C(10)	C(11)	118.87(8)	B(2)	B(6)	B(11)	107.82(9)
C(9)	C(10)	B(3)	119.67(8)	B(7)	B(6)	B(11)	59.80(7)

C(11)	C(10)	B(3)	110.16(8)	B(10)	B(6)	B(11)	59.71(7)
C(9)	C(10)	B(2)	122.27(9)	B(2)	B(7)	B(11)	108.31(9)
C(11)	C(10)	B(2)	110.35(8)	B(2)	B(7)	B(6)	60.00(7)
B(3)	C(10)	B(2)	62.98(7)	B(11)	B(7)	B(6)	60.32(7)
C(9)	C(10)	B(1)	118.86(8)	B(2)	B(7)	B(3)	60.14(7)
C(11)	C(10)	B(1)	61.47(6)	B(11)	B(7)	B(3)	108.19(9)
B(3)	C(10)	B(1)	114.20(8)	B(6)	B(7)	B(3)	108.22(9)
B(2)	C(10)	B(1)	62.08(7)	B(2)	B(7)	B(8)	108.41(9)
C(9)	C(10)	B(4)	115.06(8)	B(11)	B(7)	B(8)	60.28(7)
C(11)	C(10)	B(4)	60.78(6)	B(6)	B(7)	B(8)	108.66(9)
B(3)	C(10)	B(4)	62.27(7)	B(3)	B(7)	B(8)	60.02(7)
B(2)	C(10)	B(4)	114.16(8)	B(4)	B(8)	B(9)	60.09(7)
B(1)	C(10)	B(4)	114.04(8)	B(4)	B(8)	B(3)	60.21(7)
C(10)	C(11)	B(10)	112.37(8)	B(9)	B(8)	B(3)	107.87(8)
C(10)	C(11)	B(9)	112.62(8)	B(4)	B(8)	B(7)	107.98(9)
B(10)	C(11)	B(9)	63.36(7)	B(9)	B(8)	B(7)	107.41(9)
C(10)	C(11)	B(4)	62.51(6)	B(3)	B(8)	B(7)	59.82(7)
B(10)	C(11)	B(4)	115.52(8)	B(4)	B(8)	B(11)	107.87(9)
B(9)	C(11)	B(4)	62.60(7)	B(9)	B(8)	B(11)	59.71(7)
C(10)	C(11)	B(1)	61.73(6)	B(3)	B(8)	B(11)	107.49(9)
B(10)	C(11)	B(1)	62.82(7)	B(7)	B(8)	B(11)	59.60(7)
B(9)	C(11)	B(1)	115.53(9)	C(11)	B(9)	B(4)	58.92(7)
B(4)	C(11)	B(1)	115.72(8)	C(11)	B(9)	B(11)	103.87(9)
C(11)	B(1)	C(10)	56.80(6)	B(4)	B(9)	B(11)	108.13(9)
C(11)	B(1)	B(6)	104.07(8)	C(11)	B(9)	B(8)	104.11(8)
C(10)	B(1)	B(6)	105.05(8)	B(4)	B(9)	B(8)	59.57(7)
C(11)	B(1)	B(2)	103.89(8)	B(11)	B(9)	B(8)	60.55(7)
C(10)	B(1)	B(2)	58.58(6)	C(11)	B(9)	B(10)	58.08(7)
B(6)	B(1)	B(2)	60.41(7)	B(4)	B(9)	B(10)	108.10(8)
C(11)	B(1)	B(10)	57.86(7)	B(11)	B(9)	B(10)	59.93(7)
C(10)	B(1)	B(10)	104.39(8)	B(8)	B(9)	B(10)	108.29(9)
B(6)	B(1)	B(10)	60.41(7)	C(11)	B(10)	B(11)	104.17(8)
B(2)	B(1)	B(10)	108.32(8)	C(11)	B(10)	B(1)	59.32(6)
C(10)	B(2)	B(1)	59.35(6)	B(11)	B(10)	B(1)	107.94(9)
C(10)	B(2)	B(7)	104.57(8)	C(11)	B(10)	B(9)	58.56(7)
B(1)	B(2)	B(7)	107.75(9)	B(11)	B(10)	B(9)	59.85(7)
C(10)	B(2)	B(6)	105.05(8)	B(1)	B(10)	B(9)	108.70(8)
B(1)	B(2)	B(6)	59.65(7)	C(11)	B(10)	B(6)	104.42(8)
B(7)	B(2)	B(6)	60.09(7)	B(11)	B(10)	B(6)	60.30(7)
C(10)	B(2)	B(3)	58.36(7)	B(1)	B(10)	B(6)	59.43(7)
B(1)	B(2)	B(3)	108.17(9)	B(9)	B(10)	B(6)	108.27(9)
B(7)	B(2)	B(3)	60.07(7)	B(9)	B(11)	B(10)	60.22(7)
B(6)	B(2)	B(3)	108.24(9)	B(9)	B(11)	B(7)	107.85(9)
C(10)	B(3)	B(4)	59.78(6)	B(10)	B(11)	B(7)	107.67(9)
C(10)	B(3)	B(7)	104.58(8)	B(9)	B(11)	B(6)	108.33(9)
B(4)	B(3)	B(7)	107.50(9)	B(10)	B(11)	B(6)	60.00(7)
C(10)	B(3)	B(2)	58.66(6)	B(7)	B(11)	B(6)	59.89(7)
B(4)	B(3)	B(2)	108.60(8)	B(9)	B(11)	B(8)	59.74(7)
B(7)	B(3)	B(2)	59.80(7)	B(10)	B(11)	B(8)	107.87(9)
C(10)	B(3)	B(8)	105.20(8)	B(7)	B(11)	B(8)	60.12(7)
B(4)	B(3)	B(8)	59.25(7)	B(6)	B(11)	B(8)	108.19(9)
B(7)	B(3)	B(8)	60.16(8)				

Symmetry Operators

(1) x, y, z	(2) -x+1/2, -y+1/2, z	(3) -x, y+1/2, -z+1/2
(4) x+1/2, -y, -z+1/2	(5) -x, -y, -z	(6) x-1/2, y-1/2, -z
(7) x, -y-1/2, z-1/2	(8) -x-1/2, y, z-1/2	

Table 15. Torsion Angles (°)

Atom	Atom	Atom	Atom	Angle
C(1)	O(1)	C(2)	C(7)	-6.08(17)
C(1)	O(1)	C(2)	C(3)	174.80(10)
O(1)	C(2)	C(3)	C(4)	179.74(10)
C(7)	C(2)	C(3)	C(4)	0.58(16)
C(2)	C(3)	C(4)	C(5)	-0.20(15)
C(2)	C(3)	C(4)	C(8)	177.22(9)
C(3)	C(4)	C(5)	C(6)	-0.57(16)
C(8)	C(4)	C(5)	C(6)	-177.94(10)
C(3)	C(4)	C(5)	Cl(1)	-179.01(8)
C(8)	C(4)	C(5)	Cl(1)	3.62(15)
C(4)	C(5)	C(6)	C(7)	0.98(18)
Cl(1)	C(5)	C(6)	C(7)	179.48(10)
O(1)	C(2)	C(7)	C(6)	-179.27(11)
C(3)	C(2)	C(7)	C(6)	-0.18(18)
C(5)	C(6)	C(7)	C(2)	-0.59(19)
C(3)	C(4)	C(8)	O(2)	-129.35(11)
C(5)	C(4)	C(8)	O(2)	47.99(15)
C(3)	C(4)	C(8)	N(1)	48.87(13)
C(5)	C(4)	C(8)	N(1)	-133.79(10)
O(2)	C(8)	N(1)	C(9)	0.81(15)
C(4)	C(8)	N(1)	C(9)	-177.39(9)
C(8)	N(1)	C(9)	C(10)	97.64(11)
N(1)	C(9)	C(10)	C(11)	40.42(12)
N(1)	C(9)	C(10)	B(3)	-179.48(9)
N(1)	C(9)	C(10)	B(2)	-104.46(11)
N(1)	C(9)	C(10)	B(1)	-30.98(13)
N(1)	C(9)	C(10)	B(4)	109.47(10)
C(9)	C(10)	C(11)	B(10)	-147.65(9)
B(3)	C(10)	C(11)	B(10)	68.76(11)
B(2)	C(10)	C(11)	B(10)	1.10(11)
B(1)	C(10)	C(11)	B(10)	-38.54(8)
B(4)	C(10)	C(11)	B(10)	108.11(9)
C(9)	C(10)	C(11)	B(9)	143.06(9)
B(3)	C(10)	C(11)	B(9)	-0.52(12)
B(2)	C(10)	C(11)	B(9)	-68.19(11)
B(1)	C(10)	C(11)	B(9)	-107.82(10)
B(4)	C(10)	C(11)	B(9)	38.83(9)
C(9)	C(10)	C(11)	B(4)	104.23(10)
B(3)	C(10)	C(11)	B(4)	-39.35(8)
B(2)	C(10)	C(11)	B(4)	-107.02(9)
B(1)	C(10)	C(11)	B(4)	-146.65(9)
C(9)	C(10)	C(11)	B(1)	-109.12(10)
B(3)	C(10)	C(11)	B(1)	107.30(9)
B(2)	C(10)	C(11)	B(1)	39.63(8)
B(4)	C(10)	C(11)	B(1)	146.65(9)
B(10)	C(11)	B(1)	C(10)	139.64(8)
B(9)	C(11)	B(1)	C(10)	103.13(9)
B(4)	C(11)	B(1)	C(10)	32.77(8)
C(10)	C(11)	B(1)	B(6)	-98.97(9)
B(10)	C(11)	B(1)	B(6)	40.66(8)
B(9)	C(11)	B(1)	B(6)	4.16(12)
B(4)	C(11)	B(1)	B(6)	-66.20(11)
C(10)	C(11)	B(1)	B(2)	-36.51(7)
B(10)	C(11)	B(1)	B(2)	103.12(9)
B(9)	C(11)	B(1)	B(2)	66.62(11)
B(4)	C(11)	B(1)	B(2)	-3.74(11)
B(10)	B(6)	B(7)	B(11)	-37.48(8)
B(1)	B(6)	B(7)	B(3)	0.08(12)
B(2)	B(6)	B(7)	B(3)	-37.31(8)
B(10)	B(6)	B(7)	B(3)	63.52(11)
B(11)	B(6)	B(7)	B(3)	101.00(10)
B(1)	B(6)	B(7)	B(8)	-63.56(11)
B(2)	B(6)	B(7)	B(8)	-100.94(10)
B(10)	B(6)	B(7)	B(8)	-0.12(12)
B(11)	B(6)	B(7)	B(8)	37.36(8)
C(10)	B(3)	B(7)	B(2)	-39.34(8)
B(4)	B(3)	B(7)	B(2)	-101.74(9)
B(8)	B(3)	B(7)	B(2)	-138.72(9)
C(10)	B(3)	B(7)	B(11)	61.76(11)
B(4)	B(3)	B(7)	B(11)	-0.64(11)
B(2)	B(3)	B(7)	B(11)	101.10(9)
B(8)	B(3)	B(7)	B(11)	-37.62(8)
C(10)	B(3)	B(7)	B(6)	-2.10(11)
B(4)	B(3)	B(7)	B(6)	-64.49(11)
B(2)	B(3)	B(7)	B(6)	37.25(8)
B(8)	B(3)	B(7)	B(6)	-101.48(10)
C(10)	B(3)	B(7)	B(8)	99.38(9)
B(4)	B(3)	B(7)	B(8)	36.98(8)
B(2)	B(3)	B(7)	B(8)	138.72(9)
C(11)	B(4)	B(8)	B(9)	39.93(8)
C(10)	B(4)	B(8)	B(9)	98.71(9)
B(3)	B(4)	B(8)	B(9)	137.50(9)
C(11)	B(4)	B(8)	B(3)	-97.57(9)
C(10)	B(4)	B(8)	B(3)	-38.79(8)
B(9)	B(4)	B(8)	B(3)	-137.50(9)
C(11)	B(4)	B(8)	B(7)	-60.22(11)
C(10)	B(4)	B(8)	B(7)	-1.43(11)
B(9)	B(4)	B(8)	B(7)	-100.14(10)
B(3)	B(4)	B(8)	B(7)	37.35(8)
C(11)	B(4)	B(8)	B(11)	2.76(11)
C(10)	B(4)	B(8)	B(11)	61.54(10)
B(9)	B(4)	B(8)	B(11)	-37.17(8)
B(3)	B(4)	B(8)	B(11)	100.33(9)
C(10)	B(3)	B(8)	B(4)	39.78(8)
B(7)	B(3)	B(8)	B(4)	138.12(9)
B(2)	B(3)	B(8)	B(4)	101.23(9)
C(10)	B(3)	B(8)	B(9)	1.81(12)
B(4)	B(3)	B(8)	B(9)	-37.97(9)
B(7)	B(3)	B(8)	B(9)	100.14(10)
B(2)	B(3)	B(8)	B(9)	63.25(11)
C(10)	B(3)	B(8)	B(7)	-98.34(9)
B(4)	B(3)	B(8)	B(7)	-138.12(9)
B(2)	B(3)	B(8)	B(7)	-36.89(8)
C(10)	B(3)	B(8)	B(11)	-61.19(11)
B(4)	B(3)	B(8)	B(11)	-100.97(9)
B(7)	B(3)	B(8)	B(11)	37.15(8)
B(2)	B(3)	B(8)	B(11)	0.26(12)
B(2)	B(7)	B(8)	B(4)	-0.45(12)
B(11)	B(7)	B(8)	B(4)	100.58(9)
B(6)	B(7)	B(8)	B(4)	63.20(11)
B(3)	B(7)	B(8)	B(4)	-37.53(8)
B(2)	B(7)	B(8)	B(9)	-63.85(11)

C(10)	C(11)	B(1)	B(10)	-139.64(8)
B(9)	C(11)	B(1)	B(10)	-36.50(9)
B(4)	C(11)	B(1)	B(10)	-106.86(10)
C(9)	C(10)	B(1)	C(11)	109.15(10)
B(3)	C(10)	B(1)	C(11)	-100.71(9)
B(2)	C(10)	B(1)	C(11)	-137.40(8)
B(4)	C(10)	B(1)	C(11)	-31.69(8)
C(9)	C(10)	B(1)	B(6)	-153.68(9)
C(11)	C(10)	B(1)	B(6)	97.17(9)
B(3)	C(10)	B(1)	B(6)	-3.54(11)
B(2)	C(10)	B(1)	B(6)	-40.23(8)
B(4)	C(10)	B(1)	B(6)	65.48(11)
C(9)	C(10)	B(1)	B(2)	-113.45(10)
C(11)	C(10)	B(1)	B(2)	137.40(8)
B(3)	C(10)	B(1)	B(2)	36.69(9)
B(4)	C(10)	B(1)	B(2)	105.71(9)
C(9)	C(10)	B(1)	B(10)	143.63(9)
C(11)	C(10)	B(1)	B(10)	34.48(7)
B(3)	C(10)	B(1)	B(10)	-66.23(10)
B(2)	C(10)	B(1)	B(10)	-102.92(9)
B(4)	C(10)	B(1)	B(10)	2.79(11)
C(9)	C(10)	B(2)	B(1)	108.14(10)
C(11)	C(10)	B(2)	B(1)	-39.36(8)
B(3)	C(10)	B(2)	B(1)	-142.28(9)
B(4)	C(10)	B(2)	B(1)	-105.52(9)
C(9)	C(10)	B(2)	B(7)	-149.62(9)
C(11)	C(10)	B(2)	B(7)	62.88(10)
B(3)	C(10)	B(2)	B(7)	-40.03(8)
B(1)	C(10)	B(2)	B(7)	102.25(9)
B(4)	C(10)	B(2)	B(7)	-3.27(11)
C(9)	C(10)	B(2)	B(6)	148.00(9)
C(11)	C(10)	B(2)	B(6)	0.50(11)
B(3)	C(10)	B(2)	B(6)	-102.42(9)
B(1)	C(10)	B(2)	B(6)	39.86(8)
B(4)	C(10)	B(2)	B(6)	-65.65(10)
C(9)	C(10)	B(2)	B(3)	-109.58(10)
C(11)	C(10)	B(2)	B(3)	102.92(9)
B(1)	C(10)	B(2)	B(3)	142.28(9)
B(4)	C(10)	B(2)	B(3)	36.76(8)
C(11)	B(1)	B(2)	C(10)	35.69(7)
B(6)	B(1)	B(2)	C(10)	134.17(9)
B(10)	B(1)	B(2)	C(10)	96.00(9)
C(11)	B(1)	B(2)	B(7)	-61.04(10)
C(10)	B(1)	B(2)	B(7)	-96.74(9)
B(6)	B(1)	B(2)	B(7)	37.44(8)
B(10)	B(1)	B(2)	B(7)	-0.74(11)
C(11)	B(1)	B(2)	B(6)	-98.48(9)
C(10)	B(1)	B(2)	B(6)	-134.17(9)
B(10)	B(1)	B(2)	B(6)	-38.18(8)
C(11)	B(1)	B(2)	B(3)	2.45(11)
C(10)	B(1)	B(2)	B(3)	-33.25(8)
B(6)	B(1)	B(2)	B(3)	100.93(9)
B(10)	B(1)	B(2)	B(3)	62.75(11)
C(9)	C(10)	B(3)	B(4)	-104.56(10)
C(11)	C(10)	B(3)	B(4)	38.69(8)
B(2)	C(10)	B(3)	B(4)	141.91(9)
B(1)	C(10)	B(3)	B(4)	105.56(9)
C(9)	C(10)	B(3)	B(7)	153.44(9)
C(11)	C(10)	B(3)	B(7)	-63.31(10)
B(11)	B(7)	B(8)	B(9)	37.17(8)
B(6)	B(7)	B(8)	B(9)	-0.20(12)
B(3)	B(7)	B(8)	B(9)	-100.93(9)
B(2)	B(7)	B(8)	B(3)	37.08(8)
B(11)	B(7)	B(8)	B(3)	138.11(9)
B(6)	B(7)	B(8)	B(3)	100.73(9)
B(2)	B(7)	B(8)	B(11)	-101.03(9)
B(6)	B(7)	B(8)	B(11)	-37.38(8)
B(3)	B(7)	B(8)	B(11)	-138.11(9)
C(10)	C(11)	B(9)	B(4)	-38.79(8)
B(10)	C(11)	B(9)	B(4)	-143.41(9)
B(1)	C(11)	B(9)	B(4)	-107.11(9)
C(10)	C(11)	B(9)	B(11)	64.23(11)
B(10)	C(11)	B(9)	B(11)	-40.38(8)
B(4)	C(11)	B(9)	B(11)	103.03(9)
B(1)	C(11)	B(9)	B(11)	-4.08(12)
C(10)	C(11)	B(9)	B(8)	1.62(12)
B(10)	C(11)	B(9)	B(8)	-102.99(9)
B(4)	C(11)	B(9)	B(8)	40.42(8)
B(1)	C(11)	B(9)	B(8)	-66.69(11)
C(10)	C(11)	B(9)	B(10)	104.61(9)
B(4)	C(11)	B(9)	B(10)	143.41(9)
B(1)	C(11)	B(9)	B(10)	36.30(9)
C(10)	B(4)	B(9)	C(11)	34.29(7)
B(8)	B(4)	B(9)	C(11)	133.17(9)
B(3)	B(4)	B(9)	C(11)	94.88(9)
C(11)	B(4)	B(9)	B(11)	-95.57(9)
C(10)	B(4)	B(9)	B(11)	-61.28(11)
B(8)	B(4)	B(9)	B(11)	37.60(8)
B(3)	B(4)	B(9)	B(11)	-0.69(11)
C(11)	B(4)	B(9)	B(8)	-133.17(9)
C(10)	B(4)	B(9)	B(8)	-98.88(9)
B(3)	B(4)	B(9)	B(8)	-38.29(8)
C(11)	B(4)	B(9)	B(10)	-32.16(8)
C(10)	B(4)	B(9)	B(10)	2.13(11)
B(8)	B(4)	B(9)	B(10)	101.01(10)
B(3)	B(4)	B(9)	B(10)	62.72(11)
B(4)	B(8)	B(9)	C(11)	-40.09(8)
B(3)	B(8)	B(9)	C(11)	-2.06(12)
B(7)	B(8)	B(9)	C(11)	61.03(11)
B(11)	B(8)	B(9)	C(11)	98.16(9)
B(3)	B(8)	B(9)	B(4)	38.03(8)
B(7)	B(8)	B(9)	B(4)	101.12(9)
B(11)	B(8)	B(9)	B(4)	138.25(9)
B(4)	B(8)	B(9)	B(11)	-138.25(9)
B(3)	B(8)	B(9)	B(11)	-100.22(10)
B(7)	B(8)	B(9)	B(11)	-37.12(8)
B(4)	B(8)	B(9)	B(10)	-100.68(9)
B(3)	B(8)	B(9)	B(10)	-62.65(11)
B(7)	B(8)	B(9)	B(10)	0.45(12)
B(11)	B(8)	B(9)	B(10)	37.57(8)
C(10)	C(11)	B(10)	B(11)	-64.60(10)
B(9)	C(11)	B(10)	B(11)	40.40(8)
B(4)	C(11)	B(10)	B(11)	4.50(12)
B(1)	C(11)	B(10)	B(11)	-102.69(9)
C(10)	C(11)	B(10)	B(1)	38.09(8)
B(9)	C(11)	B(10)	B(1)	143.09(9)
B(4)	C(11)	B(10)	B(1)	107.19(9)
C(10)	C(11)	B(10)	B(9)	-105.01(9)

B(2)	C(10)	B(3)	B(7)	39.90(8)
B(1)	C(10)	B(3)	B(7)	3.56(11)
B(4)	C(10)	B(3)	B(7)	-102.00(9)
C(9)	C(10)	B(3)	B(2)	113.53(10)
C(11)	C(10)	B(3)	B(2)	-103.22(9)
B(1)	C(10)	B(3)	B(2)	-36.35(9)
B(4)	C(10)	B(3)	B(2)	-141.91(9)
C(9)	C(10)	B(3)	B(8)	-144.08(9)
C(11)	C(10)	B(3)	B(8)	-0.83(11)
B(2)	C(10)	B(3)	B(8)	102.38(10)
B(1)	C(10)	B(3)	B(8)	66.04(11)
B(4)	C(10)	B(3)	B(8)	-39.53(8)
B(1)	B(2)	B(3)	C(10)	33.64(8)
B(7)	B(2)	B(3)	C(10)	134.08(9)
B(6)	B(2)	B(3)	C(10)	96.79(9)
C(10)	B(2)	B(3)	B(4)	-34.23(8)
B(1)	B(2)	B(3)	B(4)	-0.59(12)
B(7)	B(2)	B(3)	B(4)	99.85(10)
B(6)	B(2)	B(3)	B(4)	62.56(11)
C(10)	B(2)	B(3)	B(7)	-134.08(9)
B(1)	B(2)	B(3)	B(7)	-100.44(9)
B(6)	B(2)	B(3)	B(7)	-37.29(8)
C(10)	B(2)	B(3)	B(8)	-97.03(9)
B(1)	B(2)	B(3)	B(8)	-63.39(11)
B(7)	B(2)	B(3)	B(8)	37.05(8)
B(6)	B(2)	B(3)	B(8)	-0.24(11)
B(10)	C(11)	B(4)	C(10)	-103.13(9)
B(9)	C(11)	B(4)	C(10)	-139.31(9)
B(1)	C(11)	B(4)	C(10)	-32.51(8)
C(10)	C(11)	B(4)	B(8)	98.45(9)
B(10)	C(11)	B(4)	B(8)	-4.68(12)
B(9)	C(11)	B(4)	B(8)	-40.86(8)
B(1)	C(11)	B(4)	B(8)	65.94(11)
C(10)	C(11)	B(4)	B(9)	139.31(9)
B(10)	C(11)	B(4)	B(9)	36.19(9)
B(1)	C(11)	B(4)	B(9)	106.80(10)
C(10)	C(11)	B(4)	B(3)	35.88(8)
B(10)	C(11)	B(4)	B(3)	-67.25(11)
B(9)	C(11)	B(4)	B(3)	-103.43(9)
B(1)	C(11)	B(4)	B(3)	3.37(11)
C(9)	C(10)	B(4)	C(11)	-110.44(9)
B(3)	C(10)	B(4)	C(11)	137.75(9)
B(2)	C(10)	B(4)	C(11)	100.71(9)
B(1)	C(10)	B(4)	C(11)	31.93(8)
C(9)	C(10)	B(4)	B(8)	151.87(9)
C(11)	C(10)	B(4)	B(8)	-97.69(9)
B(3)	C(10)	B(4)	B(8)	40.05(8)
B(2)	C(10)	B(4)	B(8)	3.02(11)
B(1)	C(10)	B(4)	B(8)	-65.76(11)
C(9)	C(10)	B(4)	B(9)	-145.51(9)
C(11)	C(10)	B(4)	B(9)	-35.07(8)
B(3)	C(10)	B(4)	B(9)	102.68(9)
B(2)	C(10)	B(4)	B(9)	65.64(11)
B(1)	C(10)	B(4)	B(9)	-3.14(11)
C(9)	C(10)	B(4)	B(3)	111.81(10)
C(11)	C(10)	B(4)	B(3)	-137.75(9)
B(2)	C(10)	B(4)	B(3)	-37.04(9)
B(1)	C(10)	B(4)	B(3)	-105.82(9)
C(10)	B(3)	B(4)	C(11)	-35.31(7)
B(4)	C(11)	B(10)	B(9)	-35.90(9)
B(1)	C(11)	B(10)	B(9)	-143.09(9)
C(10)	C(11)	B(10)	B(6)	-2.17(11)
B(9)	C(11)	B(10)	B(6)	102.84(9)
B(4)	C(11)	B(10)	B(6)	66.93(11)
B(1)	C(11)	B(10)	B(6)	-40.25(8)
C(10)	B(1)	B(10)	C(11)	-34.02(7)
B(6)	B(1)	B(10)	C(11)	-133.38(9)
B(2)	B(1)	B(10)	C(11)	-95.20(9)
C(11)	B(1)	B(10)	B(11)	96.15(9)
C(10)	B(1)	B(10)	B(11)	62.13(10)
B(6)	B(1)	B(10)	B(11)	-37.23(8)
B(2)	B(1)	B(10)	B(11)	0.94(11)
C(11)	B(1)	B(10)	B(9)	32.75(8)
C(10)	B(1)	B(10)	B(9)	-1.27(11)
B(6)	B(1)	B(10)	B(9)	-100.63(9)
B(2)	B(1)	B(10)	B(9)	-62.46(11)
C(11)	B(1)	B(10)	B(6)	133.38(9)
C(10)	B(1)	B(10)	B(6)	99.36(9)
B(2)	B(1)	B(10)	B(6)	38.18(8)
B(4)	B(9)	B(10)	C(11)	32.49(8)
B(11)	B(9)	B(10)	C(11)	133.38(9)
B(8)	B(9)	B(10)	C(11)	95.54(9)
C(11)	B(9)	B(10)	B(11)	-133.38(9)
B(4)	B(9)	B(10)	B(11)	-100.89(10)
B(8)	B(9)	B(10)	B(11)	-37.85(8)
C(11)	B(9)	B(10)	B(1)	-33.04(8)
B(4)	B(9)	B(10)	B(1)	-0.55(12)
B(11)	B(9)	B(10)	B(1)	100.34(9)
B(8)	B(9)	B(10)	B(1)	62.49(11)
C(11)	B(9)	B(10)	B(6)	-96.06(9)
B(4)	B(9)	B(10)	B(6)	-63.57(11)
B(11)	B(9)	B(10)	B(6)	37.32(8)
B(8)	B(9)	B(10)	B(6)	-0.53(12)
B(1)	B(6)	B(10)	C(11)	40.20(8)
B(2)	B(6)	B(10)	C(11)	2.39(11)
B(7)	B(6)	B(10)	C(11)	-60.77(11)
B(11)	B(6)	B(10)	C(11)	-98.29(9)
B(1)	B(6)	B(10)	B(11)	138.49(9)
B(2)	B(6)	B(10)	B(11)	100.68(9)
B(7)	B(6)	B(10)	B(11)	37.52(8)
B(2)	B(6)	B(10)	B(1)	-37.81(8)
B(7)	B(6)	B(10)	B(1)	-100.97(10)
B(11)	B(6)	B(10)	B(1)	-138.49(9)
B(1)	B(6)	B(10)	B(9)	101.37(9)
B(2)	B(6)	B(10)	B(9)	63.56(11)
B(7)	B(6)	B(10)	B(9)	0.40(12)
B(11)	B(6)	B(10)	B(9)	-37.12(8)
C(11)	B(9)	B(11)	B(10)	39.45(8)
B(4)	B(9)	B(11)	B(10)	100.85(9)
B(8)	B(9)	B(11)	B(10)	138.01(9)
C(11)	B(9)	B(11)	B(7)	-61.10(11)
B(4)	B(9)	B(11)	B(7)	0.30(12)
B(8)	B(9)	B(11)	B(7)	37.46(8)
B(10)	B(9)	B(11)	B(7)	-100.55(9)
C(11)	B(9)	B(11)	B(6)	2.24(11)
B(4)	B(9)	B(11)	B(6)	63.64(11)
B(8)	B(9)	B(11)	B(6)	100.81(9)
B(10)	B(9)	B(11)	B(6)	-37.20(8)

B(7)	B(3)	B(4)	C(11)	61.69(10)
B(2)	B(3)	B(4)	C(11)	-1.54(11)
B(8)	B(3)	B(4)	C(11)	99.08(9)
B(7)	B(3)	B(4)	C(10)	97.00(9)
B(2)	B(3)	B(4)	C(10)	33.77(8)
B(8)	B(3)	B(4)	C(10)	134.39(9)
C(10)	B(3)	B(4)	B(8)	-134.39(9)
B(7)	B(3)	B(4)	B(8)	-37.39(8)
B(2)	B(3)	B(4)	B(8)	-100.61(10)
C(10)	B(3)	B(4)	B(9)	-96.18(9)
B(7)	B(3)	B(4)	B(9)	0.82(11)
B(2)	B(3)	B(4)	B(9)	-62.40(11)
B(8)	B(3)	B(4)	B(9)	38.21(8)
C(11)	B(1)	B(6)	B(2)	98.19(9)
C(10)	B(1)	B(6)	B(2)	39.33(8)
B(10)	B(1)	B(6)	B(2)	137.57(9)
C(11)	B(1)	B(6)	B(7)	60.81(10)
C(10)	B(1)	B(6)	B(7)	1.96(11)
B(2)	B(1)	B(6)	B(7)	-37.38(8)
B(10)	B(1)	B(6)	B(7)	100.19(10)
C(11)	B(1)	B(6)	B(10)	-39.38(8)
C(10)	B(1)	B(6)	B(10)	-98.24(9)
B(2)	B(1)	B(6)	B(10)	-137.57(9)
C(11)	B(1)	B(6)	B(11)	-2.38(11)
C(10)	B(1)	B(6)	B(11)	-61.23(10)
B(2)	B(1)	B(6)	B(11)	-100.57(9)
B(10)	B(1)	B(6)	B(11)	37.00(8)
C(10)	B(2)	B(6)	B(1)	-39.71(8)
B(7)	B(2)	B(6)	B(1)	-138.09(9)
B(3)	B(2)	B(6)	B(1)	-100.81(9)
C(10)	B(2)	B(6)	B(7)	98.38(9)
B(1)	B(2)	B(6)	B(7)	138.09(9)
B(3)	B(2)	B(6)	B(7)	37.28(8)
C(10)	B(2)	B(6)	B(10)	-1.80(11)
B(1)	B(2)	B(6)	B(10)	37.91(8)
B(7)	B(2)	B(6)	B(10)	-100.18(9)
B(3)	B(2)	B(6)	B(10)	-62.90(11)
C(10)	B(2)	B(6)	B(11)	61.23(10)
B(1)	B(2)	B(6)	B(11)	100.95(9)
B(7)	B(2)	B(6)	B(11)	-37.15(8)
B(3)	B(2)	B(6)	B(11)	0.13(11)
C(10)	B(2)	B(7)	B(11)	-61.72(11)
B(1)	B(2)	B(7)	B(11)	0.25(12)
B(6)	B(2)	B(7)	B(11)	37.50(9)
B(3)	B(2)	B(7)	B(11)	-100.91(10)
C(10)	B(2)	B(7)	B(6)	-99.21(9)
B(1)	B(2)	B(7)	B(6)	-37.24(8)
B(3)	B(2)	B(7)	B(6)	-138.41(9)
C(10)	B(2)	B(7)	B(3)	39.19(8)
B(1)	B(2)	B(7)	B(3)	101.16(9)
B(6)	B(2)	B(7)	B(3)	138.41(9)
C(10)	B(2)	B(7)	B(8)	2.16(11)
B(1)	B(2)	B(7)	B(8)	64.13(11)
B(6)	B(2)	B(7)	B(8)	101.38(10)
B(3)	B(2)	B(7)	B(8)	-37.03(8)
B(1)	B(6)	B(7)	B(2)	37.39(8)
B(10)	B(6)	B(7)	B(2)	100.82(9)
B(11)	B(6)	B(7)	B(2)	138.30(9)
B(1)	B(6)	B(7)	B(11)	-100.92(9)
C(11)	B(9)	B(11)	B(8)	-98.56(9)
B(4)	B(9)	B(11)	B(8)	-37.17(8)
B(10)	B(9)	B(11)	B(8)	-138.01(9)
C(11)	B(10)	B(11)	B(9)	-39.76(8)
B(1)	B(10)	B(11)	B(9)	-101.63(9)
B(6)	B(10)	B(11)	B(9)	-138.49(9)
C(11)	B(10)	B(11)	B(7)	61.09(11)
B(1)	B(10)	B(11)	B(7)	-0.78(12)
B(9)	B(10)	B(11)	B(7)	100.85(10)
B(6)	B(10)	B(11)	B(7)	-37.64(9)
C(11)	B(10)	B(11)	B(6)	98.73(9)
B(1)	B(10)	B(11)	B(6)	36.85(8)
B(9)	B(10)	B(11)	B(6)	138.49(9)
C(11)	B(10)	B(11)	B(8)	-2.38(11)
B(1)	B(10)	B(11)	B(8)	-64.25(11)
B(9)	B(10)	B(11)	B(8)	37.38(8)
B(6)	B(10)	B(11)	B(8)	-101.11(10)
B(2)	B(7)	B(11)	B(9)	63.90(11)
B(6)	B(7)	B(11)	B(9)	101.25(9)
B(3)	B(7)	B(11)	B(9)	0.21(12)
B(8)	B(7)	B(11)	B(9)	-37.29(8)
B(2)	B(7)	B(11)	B(10)	0.33(12)
B(6)	B(7)	B(11)	B(10)	37.69(9)
B(3)	B(7)	B(11)	B(10)	-63.35(11)
B(8)	B(7)	B(11)	B(10)	-100.86(10)
B(2)	B(7)	B(11)	B(6)	-37.36(8)
B(3)	B(7)	B(11)	B(6)	-101.04(9)
B(8)	B(7)	B(11)	B(6)	-138.55(9)
B(2)	B(7)	B(11)	B(8)	101.19(10)
B(6)	B(7)	B(11)	B(8)	138.55(9)
B(3)	B(7)	B(11)	B(8)	37.51(8)
B(1)	B(6)	B(11)	B(9)	0.10(12)
B(2)	B(6)	B(11)	B(9)	-63.24(11)
B(7)	B(6)	B(11)	B(9)	-100.43(10)
B(10)	B(6)	B(11)	B(9)	37.30(8)
B(1)	B(6)	B(11)	B(10)	-37.20(8)
B(2)	B(6)	B(11)	B(10)	-100.53(9)
B(7)	B(6)	B(11)	B(10)	-137.73(9)
B(1)	B(6)	B(11)	B(7)	100.53(10)
B(2)	B(6)	B(11)	B(7)	37.20(8)
B(10)	B(6)	B(11)	B(7)	137.73(9)
B(1)	B(6)	B(11)	B(8)	63.36(11)
B(2)	B(6)	B(11)	B(8)	0.02(11)
B(7)	B(6)	B(11)	B(8)	-37.17(9)
B(10)	B(6)	B(11)	B(8)	100.56(9)
B(4)	B(8)	B(11)	B(9)	37.33(8)
B(3)	B(8)	B(11)	B(9)	100.86(9)
B(7)	B(8)	B(11)	B(9)	138.11(9)
B(4)	B(8)	B(11)	B(10)	-0.26(12)
B(9)	B(8)	B(11)	B(10)	-37.59(8)
B(3)	B(8)	B(11)	B(10)	63.27(11)
B(7)	B(8)	B(11)	B(10)	100.52(10)
B(4)	B(8)	B(11)	B(7)	-100.78(9)
B(9)	B(8)	B(11)	B(7)	-138.11(9)
B(3)	B(8)	B(11)	B(7)	-37.24(8)
B(4)	B(8)	B(11)	B(6)	-63.70(11)
B(9)	B(8)	B(11)	B(6)	-101.04(9)
B(3)	B(8)	B(11)	B(6)	-0.17(12)
B(7)	B(8)	B(11)	B(6)	37.07(8)

B(2)	B(6)	B(7)	B(11)	-138.30(9)

Symmetry Operators

(1) x, y, z	(2) -x+1/2, -y+1/2, z	(3) -x, y+1/2, -z+1/2
(4) x+1/2, -y, -z+1/2	(5) -x, -y, -z	(6) x-1/2, y-1/2, -z
(7) x, -y-1/2, z-1/2	(8) -x-1/2, y, z-1/2	

Table 16. Hydrogen Bond Geometry

Donor	Hydrogen	Acceptor	D-H (Å)	H-A (Å)	D-A (Å)	DHA Angle (º)
N(1)	H(1D)	O(2) 7_576	0.88	2.10	2.9033(11)	151.3

Symmetry Operators

(1) x, y, z	(2) -x+1/2, -y+1/2, z	(3) -x, y+1/2, -z+1/2
(4) x+1/2, -y, -z+1/2	(5) -x, -y, -z	(6) x-1/2, y-1/2, -z
(7) x, -y-1/2, z-1/2	(8) -x-1/2, y, z-1/2	

Table 17. Hydrogen Bond Lengths (Å)

Atom	Atom	Distance	Atom	Atom	Distance
C(1)	H(1A)	0.9800	C(1)	H(1B)	0.9800
C(1)	H(1C)	0.9800	C(3)	H(3A)	0.9500
C(6)	H(6A)	0.9500	C(7)	H(7A)	0.9500
N(1)	H(1D)	0.8800	C(9)	H(9A)	0.9900
C(9)	H(9B)	0.9900	C(11)	H(11A)	0.965(15)
B(1)	H(1)	1.079(14)	B(2)	H(2)	1.094(15)
B(3)	H(3)	1.071(15)	B(4)	H(4)	1.088(14)
B(6)	H(6)	1.081(14)	B(7)	H(7)	1.096(15)
B(8)	H(8)	1.091(15)	B(9)	H(9)	1.078(15)
B(10)	H(10)	1.061(14)	B(11)	H(11)	1.080(14)

Symmetry Operators

(1) x, y, z	(2) -x+1/2, -y+1/2, z	(3) -x, y+1/2, -z+1/2	(4) x+1/2, -y, -z+1/2
(5) -x, -y, -z	(6) x-1/2, y-1/2, -z	(7) x, -y-1/2, z-1/2	(8) -x-1/2, y, z-1/2

Table 18. Hydrogen Bond Angles (º)

Atom	Atom	Atom	Angle	Atom	Atom	Atom	Angle
O(1)	C(1)	H(1A)	109.5	B(2)	B(3)	H(3)	121.2(8)
O(1)	C(1)	H(1B)	109.5	B(8)	B(3)	H(3)	126.2(8)
H(1A)	C(1)	H(1B)	109.5	C(11)	B(4)	H(4)	117.2(8)
O(1)	C(1)	H(1C)	109.5	C(10)	B(4)	H(4)	116.8(7)
H(1A)	C(1)	H(1C)	109.5	B(8)	B(4)	H(4)	132.3(8)
H(1B)	C(1)	H(1C)	109.5	B(9)	B(4)	H(4)	124.1(7)
C(4)	C(3)	H(3A)	119.6	B(3)	B(4)	H(4)	124.8(7)
C(2)	C(3)	H(3A)	119.6	B(1)	B(6)	H(6)	120.7(7)
C(5)	C(6)	H(6A)	119.6	B(2)	B(6)	H(6)	121.5(8)
C(7)	C(6)	H(6A)	119.6	B(7)	B(6)	H(6)	122.7(7)
C(2)	C(7)	H(7A)	120.3	B(10)	B(6)	H(6)	121.8(8)
C(6)	C(7)	H(7A)	120.3	B(11)	B(6)	H(6)	122.6(8)
C(8)	N(1)	H(1D)	119.2	B(2)	B(7)	H(7)	120.8(8)
C(9)	N(1)	H(1D)	119.2	B(11)	B(7)	H(7)	122.0(8)
N(1)	C(9)	H(9A)	108.7	B(6)	B(7)	H(7)	121.0(8)
C(10)	C(9)	H(9A)	108.7	B(3)	B(7)	H(7)	121.7(8)
N(1)	C(9)	H(9B)	108.7	B(8)	B(7)	H(7)	122.1(8)
C(10)	C(9)	H(9B)	108.7	B(4)	B(8)	H(8)	119.4(8)
H(9A)	C(9)	H(9B)	107.6	B(9)	B(8)	H(8)	123.1(8)
C(10)	C(11)	H(11A)	114.7(9)	B(3)	B(8)	H(8)	119.4(8)
B(10)	C(11)	H(11A)	122.0(9)	B(7)	B(8)	H(8)	122.6(8)
B(9)	C(11)	H(11A)	122.2(9)	B(11)	B(8)	H(8)	124.9(8)
B(4)	C(11)	H(11A)	115.3(9)	C(11)	B(9)	H(9)	116.7(8)
B(1)	C(11)	H(11A)	115.1(9)	B(4)	B(9)	H(9)	117.1(8)
C(11)	B(1)	H(1)	116.7(7)	B(11)	B(9)	H(9)	130.0(8)
C(10)	B(1)	H(1)	118.0(8)	B(8)	B(9)	H(9)	128.4(8)
B(6)	B(1)	H(1)	131.9(8)	B(10)	B(9)	H(9)	119.4(8)
B(2)	B(1)	H(1)	126.4(7)	C(11)	B(10)	H(10)	116.5(8)
B(10)	B(1)	H(1)	122.3(7)	B(11)	B(10)	H(10)	130.3(8)
C(10)	B(2)	H(2)	117.8(8)	B(1)	B(10)	H(10)	116.7(8)
B(1)	B(2)	H(2)	119.8(8)	B(9)	B(10)	H(10)	119.7(8)
B(7)	B(2)	H(2)	127.4(8)	B(6)	B(10)	H(10)	128.0(8)
B(6)	B(2)	H(2)	128.1(8)	B(9)	B(11)	H(11)	121.5(8)
B(3)	B(2)	H(2)	118.3(8)	B(10)	B(11)	H(11)	122.2(7)
C(10)	B(3)	H(3)	117.4(8)	B(7)	B(11)	H(11)	121.8(7)
B(4)	B(3)	H(3)	116.8(8)	B(6)	B(11)	H(11)	121.9(8)
B(7)	B(3)	H(3)	129.7(8)	B(8)	B(11)	H(11)	121.3(7)

Symmetry Operators

(1) x, y, z	(2) -x+1/2, -y+1/2, z	(3) -x, y+1/2, -z+1/2	(4) x+1/2, -y, -z+1/2
(5) -x, -y, -z	(6) x-1/2, y-1/2, -z	(7) x, -y-1/2, z-1/2	(8) -x-1/2, y, z-1/2

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