Supporting Information for

Stereocontrolled Synthesis of 20,21-Dihydro-*N*-Methylwelwitindolinone B Isothiocyanate

Vikram Bhat, and Viresh H. Rawal*

Searle Chemistry Laboratory, Department of Chemistry, The University of Chicago
5735 South Ellis Avenue, Chicago, IL 60637

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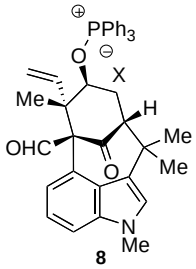
General Information

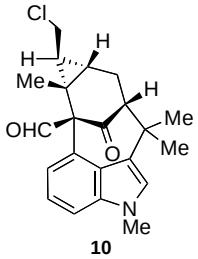
Unless stated otherwise, reactions were performed in flame or oven-dried glassware under an argon or nitrogen atmosphere using dry, deoxygenated solvents (distilled or passed over a column of activated alumina). Commercially available chemicals were used as received, unless otherwise stated. Ambient temperature refers to 22–26 °C. Higher than ambient reaction temperatures were controlled by IKAmag temperature modulators. All reactions were monitored by thin-layer chromatography (TLC) which were performed using pre-coated Whatman K6F silica gel (250 μm, 60 Å) plates and Dynamic Adsorbents silica gel (250 μm, 60 Å) plates with F-254 indicator and visualized by UV fluorescence quenching, ceric ammonium molybdate, anisaldehyde, or potassium permanganate staining. Preparative TLC was performed using Whatman Partisil PK6F pre-coated 60 Å silica gel plates (500 μm) with fluorescent indicator. Zeochem ZEOprep Flash ECO 40–63 Academic Grade silica gel was used for column chromatography. Melting points were measured on a Fisher-Johns melting point apparatus and are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on Bruker DRX-500 and DMX-500 (at 500 MHz and 125 MHz, respectively) and are reported relative to Me_4Si (δ 0.0), unless otherwise stated. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm) (multiplicity, coupling constant (Hz), integration). Infrared spectra were recorded on Nicolet 6700 FT-IR spectrometer and are reported in frequency of absorption (cm^{-1}). High resolution mass spectra (HRMS) were recorded at the Old Dominion University, Norfolk, and Hunter College, New York.

Experimental Procedures and Characterization Data

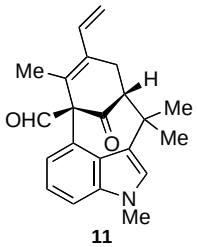


8

Oxo-phosphonium 8. To a solution of alcohol **7** (21.0 mg, 0.06 mmol) in 7.0 mL DCM at ambient temperature was added triphenylphosphine (31.3 mg, 0.12 mmol). Neat hexachloroacetone[1] (36.0 μL, 0.24 mmol) was added to the reaction and the reaction mixture was stirred ambient temperature. TLC after 10 min indicated complete disappearance of starting material and a new spot was observed at the baseline. The reaction was concentrated and the crude reaction mixture was titurated with hexanes and ether to remove most of the non-polar impurities. ^{31}P NMR (202 MHz, $CDCl_3$) δ 63.3. ^{1}H NMR ($CDCl_3$) δ 9.67 (s, 1H), 7.68-7.42 (m),[2] 7.32 (d, *J* = 8.3 Hz, 1H), 7.16 (s, 1H), 7.08 (app t, *J* = 8.3 Hz, 1H), 6.28 (d, *J* = 8.3 Hz, 1H), 5.29 (d, *J* = 17.4 Hz, 1H), 5.16 (d, *J* = 11.4 Hz, 1H), 4.63 (dd, *J* = 17.4, 11.4 Hz, 1H), 4.22-4.17 (m, 1H), 3.98 (s, 3H), 2.78 (d, *J* = 9.9 Hz, 1H), 2.50-2.43 (m, 1H), 2.11 (dd, *J* = 13.7, 6.1 Hz, 1H), 1.72 (s, 3H), 1.31 (s, 3H), 1.05 (s, 3H).



10

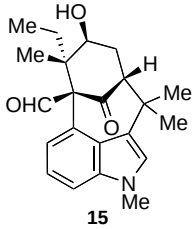


11

Cyclopropane 10 and Diene 11. To a solution of alcohol **7** (20.0 mg, 0.057 mmol) in 4.0 mL DCM at ambient temperature was added tri(2-furyl)phosphine (32.9 mg, 0.142 mmol). Neat hexachloroacetone (40.0 μL, 0.29 mmol) was added to the reaction and the reaction mixture was placed in an oil bath that was preheated to 41 °C. The reaction was followed by TLC and compounds **10**, and **11** were found to have the same R_f. After 2h reaction was cooled to ambient

[1] Distilled before use.
[2] We were unable to completely remove triphenylphosphine oxide from phosphonium **8**, hence the aromatic resonances belonging to the triphenylphosphonium adduct could not be accurately identified or integrated.

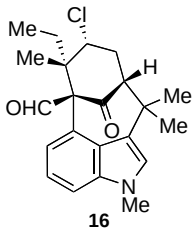
temperature, concentrated, and loaded on a PTLC plate (10 cm x 20 cm) with 10:1 Hex:EtOAc as the eluent. The plate was repeatedly eluted until the two bands separated (5-6 times). Diene **11**: 7.8 mg (41% yield). White solid. R_f = 0.5 (4:1 Hex:EtOAc). Crystals suitable for X-ray diffraction were grown via diffusion crystallization of the white solid from DCM and hexanes. MP 189-191 °C. IR (Neat film, NaCl): 2967, 1739, 1691, 1447, 1417, 1333, 1267, 1232, 1206, 1153 cm^{-1}. ^1H NMR ($CDCl_3$) δ 9.96 (s, 1H), 7.28-7.21 (m, 2H), 6.97 (s, 1H), 6.73 (d, *J* = 7.0 Hz, 1H), 6.65 (dd, *J* = 17.4, 11.0 Hz, 1H), 5.17 (d, *J* = 17.4 Hz, 1H), 5.05 (d, *J* = 11.0 Hz, 1H), 3.76 (s, 3H), 2.88 (dd, *J* = 16.8, 9.3 Hz, 1H), 2.74 (dd, *J* = 9.3, 5.8 Hz, 1H), 2.70-2.64 (m, 1H), 2.00 (d, *J* = 1.2 Hz, 3H), 1.53 (s, 3H), 1.31 (s, 3H). ^{13}C NMR ($CDCl_3$) δ 211.1, 198.7, 138.0, 134.0, 133.6, 132.0, 127.3, 127.0, 124.6, 121.8, 120.9, 118.2, 113.8, 108.9, 71.8, 55.9, 37.7, 33.6, 33.0, 29.1, 28.5, 15.7. HRMS *m/z* calc'd for $C_{22}H_{24}NO_2$ $[M+H]^+$: 334.1802, found 334.18. Cyclopropane **10**: 5.2 mg (25% yield). White solid. R_f = 0.5 (4:1 Hex:EtOAc). Crystals suitable for X-ray diffraction were grown via diffusion crystallization of the white solid from DCM and hexanes. MP 193-195 °C. IR (Neat film, NaCl): 2965, 1724, 1693, 1447, 1419, 1367, 1335, 1243, 1195, 1152, 1053 cm^{-1}. ^1H NMR ($CDCl_3$) δ 9.61 (s, 1H), 7.28-7.23 (m, 2H), 6.98 (dd, *J* = 7.1, 1.8 Hz, 1H), 6.96 (s, 1H), 3.76 (s, 3H), 3.62 (dd, *J* = 11.5, 7.3 Hz, 1H), 3.53 (dd, *J* = 11.5, 8.6 Hz, 1H), 2.64 (dd, *J* = 11.5, 8.7 Hz, 1H), 2.41 (dt, *J* = 15.2, 8.7 Hz, 1H), 1.63-1.56 (m, 1H), 1.41 (s, 3H), 1.39 (s, 3H), 1.35 (s, 3H), 1.25-1.22 (m, 1H), 0.75 (app td, *J* = 8.4, 4.7 Hz, 1H). ^{13}C NMR ($CDCl_3$) δ 211.7, 194.9, 137.1, 127.8, 126.0, 124.5, 121.8, 121.7, 120.4, 109.4, 70.4, 58.0, 45.2, 37.4, 36.8, 33.1, 33.0, 32.1, 30.9, 28.7, 26.0, 18.9. HRMS *m/z* calc'd for $C_{22}H_{25}ClNO_2$ $[M+H]^+$: 370.1568, found 370.1575.



15

Alcohol 15. A solution of alcohol **7** (50.0 mg, 0.14 mmol) in 1.0 mL EtOAc (not anhydrous) was purged with N_2. To this solution, 20.0 mg of Pearlman's catalyst

($Pd(OH)_2$/C, Sigma-Aldrich, Pd content ~20%, H_2O content ≤ 50%) was added and black suspension was purged once again with N_2. H_2 was introduced via balloon and the reaction was vigorously stirred at ambient temperature for 12h. The reaction mixture was filtered through a 1" Celite® plug and concentrated to obtain a white solid (50 mg). ^{1}H NMR of the crude material indicated formation of the desired product (product **15** had same R_f [= 0.12 (3:1 Hex:EtOAc)] as that of the starting material (**7**)) and was found to be pure enough for the next step and was used as such. IR (Neat film, NaCl): 3342, 2966, 1733, 1684, 1449, 1418, 1333, 1231, 1097, 1021 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ 9.92 (s, 1H), 7.31 (d, *J* = 8.3 Hz, 1H), 7.22 (app t, *J* = 8.3 Hz, 1H), 7.01 (s, 1H), 6.64 (d, *J* = 8.3 Hz, 1H), 3.79 (s, 3H), 3.57 (dd, *J* = 11.6, 6.0 Hz, 1H), 2.69 (d, *J* = 9.0 Hz, 1H), 2.19 (dd, *J* = 14.2, 6.0 Hz, 1H), 1.92 (ddd, *J* = 14.1, 11.4, 9.2 Hz, 1H), 1.69 (dq,[3] *J* = 14.9, 7.8 Hz, 1H), 1.57 (s, 3H), 1.26 (s, 3H), 1.23 (m,[4] 1H), 1.12 (s, 3H), 0.75 (t, *J* = 7.8 Hz, 3H). ^{13}C NMR ($CDCl_3$) δ 213.3, 198.0, 137.6, 126.9, 126.3, 122.8, 121.7, 121.4, 121.2, 109.4, 74.9, 71.4, 64.3, 46.9, 35.7, 35.2, 32.9, 31.2, 28.7, 28.1, 13.8, 10.1. HRMS *m/z* calc'd for $C_{22}H_{28}NO_3$ $[M+H]^+$: 354.2064, found 354.2063.

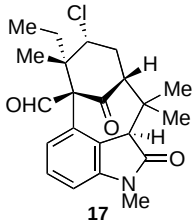


16

Chloride 16. To a solution of alcohol **15** (35.0 mg, 0.099 mmol) in 4.0 mL DCM at ambient temperature was added tri(2-furyl)phosphine (34.5 mg, 0.149 mmol). Neat hexachloroacetone (80.0 μL, 0.594 mmol) was added and the reaction mixture was placed in an oil bath that was preheated to 41 °C. After 1h the reaction was cooled to ambient temperature, concentrated, and loaded on a silica gel column (eluant 8:1 Hex:EtOAc). The material obtained from the column above was further purified via PTLC (plate size 10 cm x 20 cm; eluant 1:1 Hex:DCM) to afford 25.9 mg (70%) of chloride **16** as a white solid. R_f = 0.49 (4:1

[3] The two quartets partially overlap. This trend was observed in the remaining compounds as well.
[4] Presumably a dq but not fully visible due to partial overlap with the singlet at 1.23 ppm.

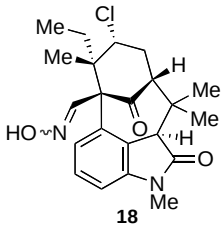
Hex:EtOAc). Crystals suitable for X-ray diffraction were grown via diffusion crystallization of the white solid from DCM and hexanes. MP 152-154 °C. IR (Neat film, NaCl): 2965, 2924, 1729, 1686, 1451, 1417, 1334, 1128, 1076 cm^{-1}. ^1H NMR ($CDCl_3$) δ 9.73 (s, 1H), 7.28 (d, *J* = 7.9 Hz, 1H), 7.20 (app t, *J* = 7.9 Hz, 1H), 7.01 (s, 1H), 6.51 (d, *J* = 7.9 Hz, 1H), 4.02-4.00 (m, 1H), 3.77 (s, 3H), 2.74-2.68 (m, 3H), 1.97 (dq, *J* = 14.1, 7.3 Hz, 1H), 1.77 (dq, *J* = 13.9, 7.3 Hz, 1H), 1.60 (s, 3H), 1.39 (s, 3H), 1.33 (s, 3H), 0.82 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR ($CDCl_3$) δ 212.9, 196.7, 137.2, 127.4, 126.5, 122.34, 122.32, 121.7, 120.6, 109.3, 73.1, 60.5, 59.4, 47.8, 36.5, 35.9, 33.0, 31.7, 29.1, 26.8, 21.8, 7.6. HRMS *m/z* calc'd for $C_{22}H_{27}ClNO_2$ $[M+H]^+$: 372.1725, found 372.1722.



17

Oxindole 17. To a solution of chloride **16** (15.0 mg, 0.04 mmol) in 0.7 mL MeCN and 0.11 mL H_2O was added $NaIO_4$ (18.9 mg, 0.09 mmol) and $RuCl_3$ (8.3 mg, 0.04 mmol). The resulting dark green solution was stirred at ambient temperature for 24h. The reaction mixture was then diluted with EtOAc (2 mL) and filtered over Celite. To the filtrate was added water (2 mL) and the biphasic mixture was extracted with EtOAc (3 x 10 mL). Combined organic layers were dried ($MgSO_4$) and concentrated. The crude material was purified by PTLC (plate size 10 cm x 20 cm; eluant 5:1 Hex:EtOAc) to afford oxindole **17** as a white solid (6.0 mg), 48% yield based on 19% recovered chloride **16** (2.9 mg). R_f = 0.32 (4:1 Hex:EtOAc). IR (Neat film, NaCl): 2959, 2924, 2854, 1711, 1606, 1585, 1460, 1369, 1340, 1262, 1077, 1037 cm^{-1}. ^1H NMR ($CDCl_3$) δ 9.36 (s, 1H), 7.31 (app t, *J* = 8.1 Hz, 1H), 6.82 (d, *J* = 8.1 Hz, 1H), 6.44 (d, *J* = 8.1 Hz, 1H), 4.51 (s, 1H), 4.25 (d, *J* = 7.4 Hz, 1H), 3.21 (s, 3H), 2.87 (ddd, *J* = 16.8, 9.4, 7.4 Hz, 1H), 2.77 (d, *J* = 16.8 Hz, 1H), 2.58 (d, *J* = 9.4 Hz, 1H), 2.00 (dq, *J* = 13.8, 7.0 Hz, 1H), 1.84 (dq, *J* = 13.8, 7.0 Hz, 1H),

1.67 (s, 3H), 1.36 (s, 3H), 0.91 (m,[5] 6H). ^{13}C NMR ($CDCl_3$) δ 210.8, 196.2, 174.7, 145.0, 128.4, 128.3, 127.3, 125.2, 107.7, 72.1, 61.6, 61.4, 54.8, 47.4, 37.6, 31.2, 26.6, 26.2, 26.0, 22.8, 21.2, 7.2. HRMS *m/z* calc'd for $C_{22}H_{26}ClNO_3Na$ $[M+Na]^+$: 410.1493, found 410.1493.

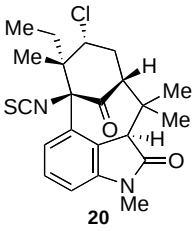


18

Oxime 18. To a solution of oxindole **17** (8.0 mg, 0.0206 mmol) in 1.0 mL MeOH was added hydroxylamine hydrochloride (11.4 mg, 0.165 mmol) and pyridine (33.0 μL, 0.412 mmol) and the reaction mixture was heated to 40 °C in an oil bath. The reaction was followed by TLC. After 1h, 6.0 mg of NH_2OH•HCl was added to speed up the reaction. The reaction was stopped after 6h because of the increasing amount of C3-epimer in the reaction mixture (as judged by TLC). The reaction mixture was cooled to ambient temperature and 1.0 N HCl was added (1.0 mL) and the biphasic mixture was extracted with DCM (3 x 5 mL). Combined organic extracts were dried ($MgSO_4$) and concentrated. The crude material was purified via PTLC (plate size 5 x 10 cm, eluant 2:1 Hex:EtOAc) to afford 4.5 mg of oxime **18** (54%), 1.5 mg of the C3-epimer (not shown) (18%), and 1.2 mg (15%) recovered oxindole **17**. Oxime **18**: white solid. R_f = 0.19 (4:1 Hex:EtOAc). IR (Neat film, NaCl): 3374, 2961, 2924, 2854, 1697, 1608, 1583, 1458, 1366, 1346, 1260, 1034, 921 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ 7.40 (s, 1H), 7.27-7.24 (m,[6] 1H), 7.03 (br s,[7] 1H), 6.82 (d, *J* = 8.0 Hz, 1H), 6.75 (d, *J* = 8.0 Hz, 1H), 4.52 (s, 1H), 4.30 (d, *J* = 7.3 Hz, 1H), 3.19 (s, 3H), 2.89 (ddd, *J* = 17.1, 9.5, 7.3 Hz, 1H), 2.78 (d, *J* = 17.3 Hz, 1H), 2.68 (d, *J* = 9.3 Hz, 1H), 2.31 (dq, *J* = 14.4, 7.4 Hz, 1H), 1.91 (dq, *J* = 14.2, 7.4 Hz, 1H), 1.67 (s, 3H), 1.34 (s, 3H), 0.89 (t, *J* = 7.4 Hz, 3H), 0.87 (s, 3H). ^{13}C NMR ($CDCl_3$) δ 208.0, 174.7, 154.4, 144.1, 130.9, 127.4, 127.3, 126.4, 107.3, 66.6, 61.6, 60.6, 55.1,

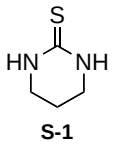
[5] Triplet (3H) and singlet (3H) overlap.
[6] Residual solvent signal overlap.
[7] Variable

47.8, 37.3, 30.7, 26.6, 26.1, 26.0, 22.8, 22.1, 7.3. HRMS *m/z* calc'd for $C_{22}H_{27}ClN_2O_3Na$ $[M+Na]^+$: 425.1602, found 425.1603.



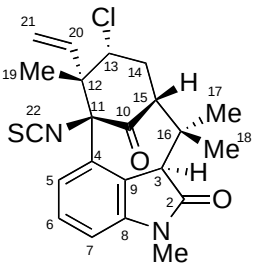
20

20,21-Dihydro *N*-Methylwelwitindolinone B Isothiocyanate (20). To a solution of oxime **18** (2.0 mg, 0.0049 mmol) in 50.0 μL DMF was added *N*-chlorosuccinimide (NCS, 1.3 mg, 0.0099 mmol) and the reaction was stirred at ambient temperature. After 5 h, the reaction was diluted with THF (250.0 μL). Solid propylene thiourea (**S-1**, 1.2 mg, 0.0099 mmol) and triethylamine (10.0 μL) were added and the reaction was stirred at ambient temperature for 10 min. The reaction was diluted with EtOAc (3 mL) and poured over water (1.0 mL) and the aqueous layer was extracted with EtOAc (4 x 4.0 mL). The combined organic layers were dried ($MgSO_4$) and concentrated. The crude material was purified via PTLC (plate size 5 cm x 10 cm; eluant 3:1 Hex:EtOAc) to afford 1.5 mg (72%) of isothiocyanate **20** as a white solid. R_f = 0.38 (3:1 Hex:EtOAc). IR (Neat film, NaCl): 2970, 2925, 2047, 1706, 1606, 1584, 1457, 1365, 1347, 891 cm^{-1}. ^1H NMR ($CDCl_3$) δ 7.35-7.30 (m, 2H), 6.80 (dd, *J* = 7.2, 1.6 Hz, 1H), 4.52 (s, 1H), 4.37 (d, *J* = 5.4 Hz, 1H), 3.19 (s, 3H), 2.91-2.79 (m, 3H), 1.91 (dq, *J* = 13.7, 7.0 Hz, 1H), 1.73-1.65 (m, 1H), 1.68 (s, 3H), 1.20 (s, 3H), 0.92 (t, *J* = 7.5 Hz, 3H), 0.87 (s, 3H). ^{13}C NMR ($CDCl_3$) δ 199.3, 174.4, 143.9, 139.7, 130.0, 128.0, 125.0, 124.1, 108.2, 84.7, 60.0, 59.6, 54.8, 50.3, 38.4, 30.5, 28.0, 26.2, 26.0, 22.7, 22.2, 7.1. HRMS *m/z* calc'd for $C_{22}H_{25}ClN_2O_2SNa$ $[M+Na]^+$: 439.1217, found 439.1218.

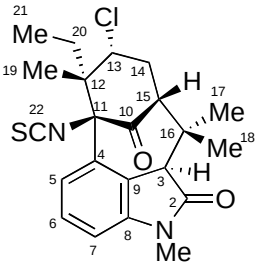


S-1

^{13}C NMR (125 MHz, $CDCl_3$) δ (assignment; *^{1}H-HMBC*)[8]



1b



20

Welwitindolinone B (**1b**)[9]	DihydroWelwitindolinone B (**20**)
198.6 (C10; *15*)	199.3 (C10; *15, 14, 19*)
174.4 (C2; *3, NMe*)	174.4 (C2; *3, NMe*)
144.1 (C8; *3, 6, NMe*)	143.9 (C8; *3, 6, NMe*)
139.6 (C22)	139.7 (C22)
136.4 (C20; *13, 19, 21*)	28.0 (C20; *13, 19, 21*)
131.3 (C4; *3, 6*)	130.0 (C4; *3, 6*)
128.8 (C6)	128.0 (C6)
123.9 (C9; *3, 5, 7*)	125.0 (C9; *3, 5, 7*)
122.6 (C5; *7*)	124.1 (C5; *6, 7*)
118.1 (C21)	7.1 (C21; *20*)
108.3 (C7; *5, NMe*)	108.2 (C7; *5*)
83.5 (C11; *5, 13, 15, 19, 20, 21*)	84.7 (C11; *5, 13, 15, 19*)
62.5 (C13; *14, 15, 19, 20*)	59.6 (C13; *14, 15, 19*)
60.1 (C15; *13, 14, 17, 18*)	60.0 (C15; *3, 13, 14, 17, 18*)
53.2 (C3; *15, 17, 18*)	54.8 (C3; *15, 17, 18*)
52.9 (C12; *13, 14, 19, 20, 21*)	50.3 (C12; *13, 14, 19, 21*)
39.8 (C16; *3, 14, 15, 17, 18*)	38.5 (C16; *3, 14, 15, 17, 18*)
31.5 (C14; *13*)	30.5 (C14; *13, 15*)
26.4 (*N*Me)	26.2 (*N*Me)
25.4 (C17; *3, 15, 18*)	26.0 (C17; *3, 18*)
24.1 (C19; *13, 20*)	22.3 (C19; *13, 20*)
22.5 (C18; *3, 17*)	22.7 (C18; *3, 17*)

[8] Key: 174.4 (C2; *3, NMe*) = Carbon labeled 2 appears at 174.4 ppm and shows HMBC correlations with protons at positions 3 and *N*Me. See page S19 for the HMBC spectra.

[9] K. Stratmann, R. E. Moore, R. Bonjouklian, J. B. Deeter, G. M. L. Patterson, S. Shaffer, C. D. Smith, T. A. Smitka, *J. Am. Chem. Soc.*, 1994, **116**, 9935.

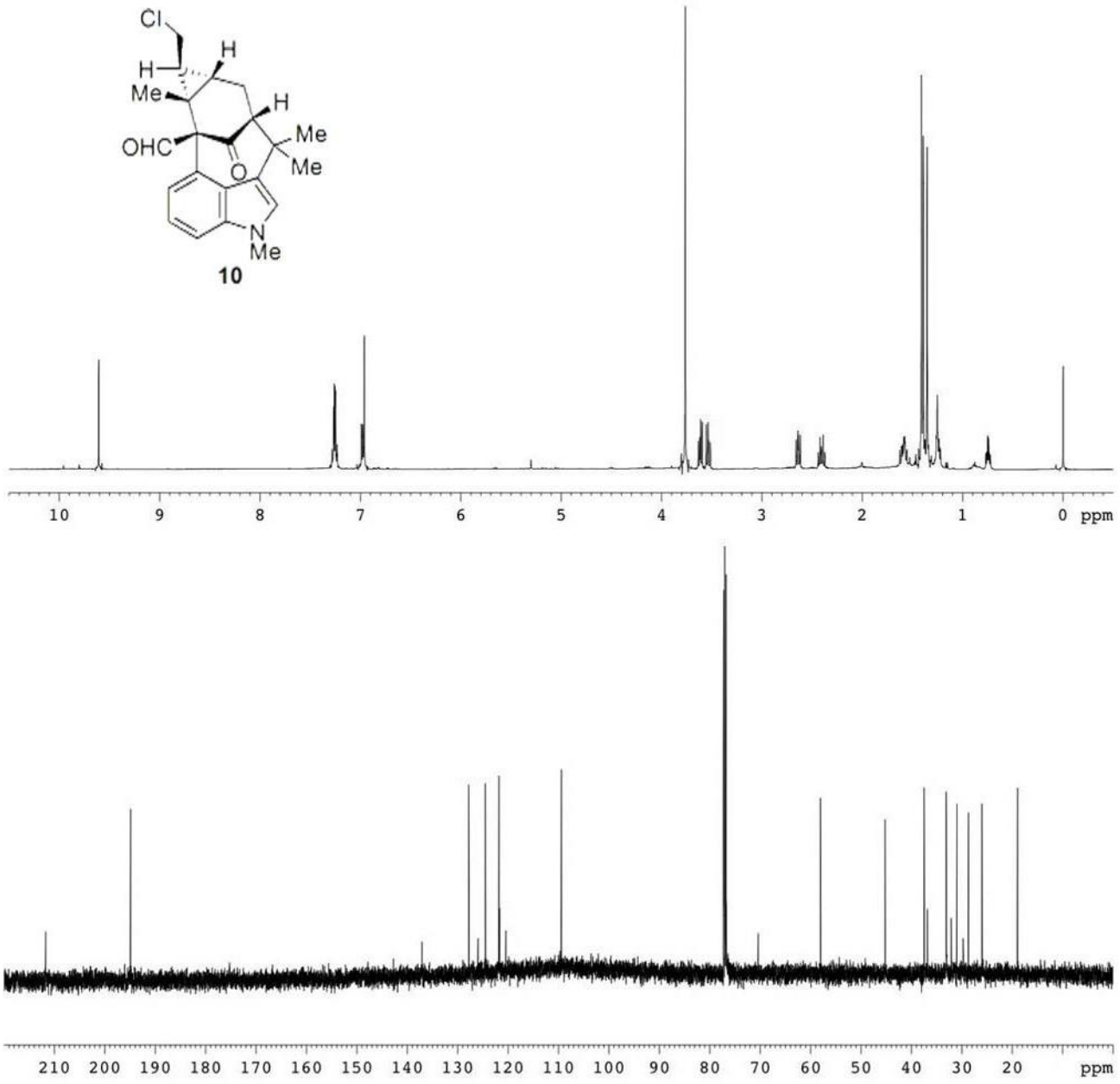


Figure S1. ^{1}H (500 MHz, $CDCl_3$) and ^{13}C (125 MHz, $CDCl_3$) NMR spectra for compound **10**.

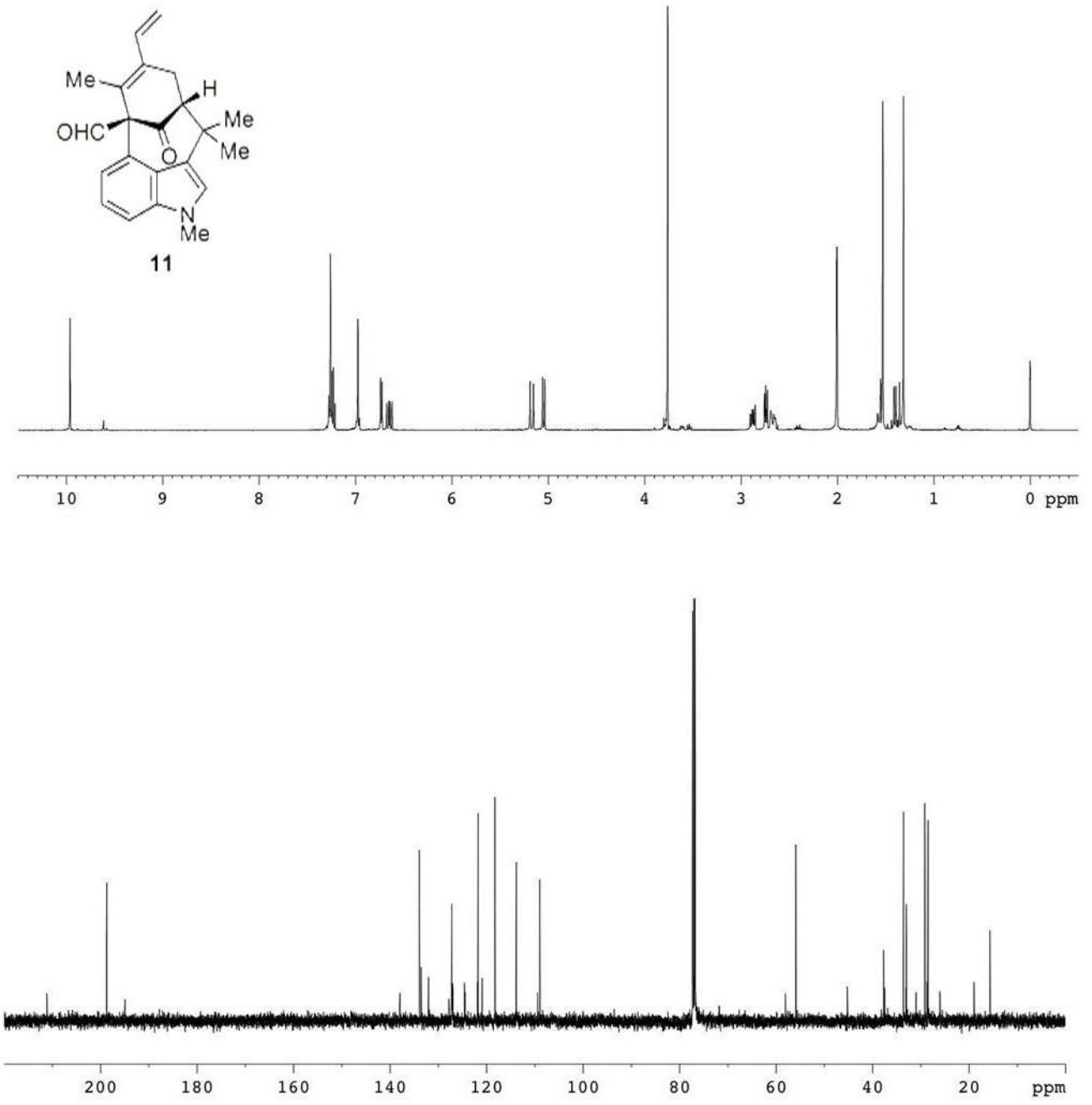


Figure S2. ^{1}H (500 MHz, $CDCl_3$) and ^{13}C (125 MHz, $CDCl_3$) NMR spectra for compound **11**.

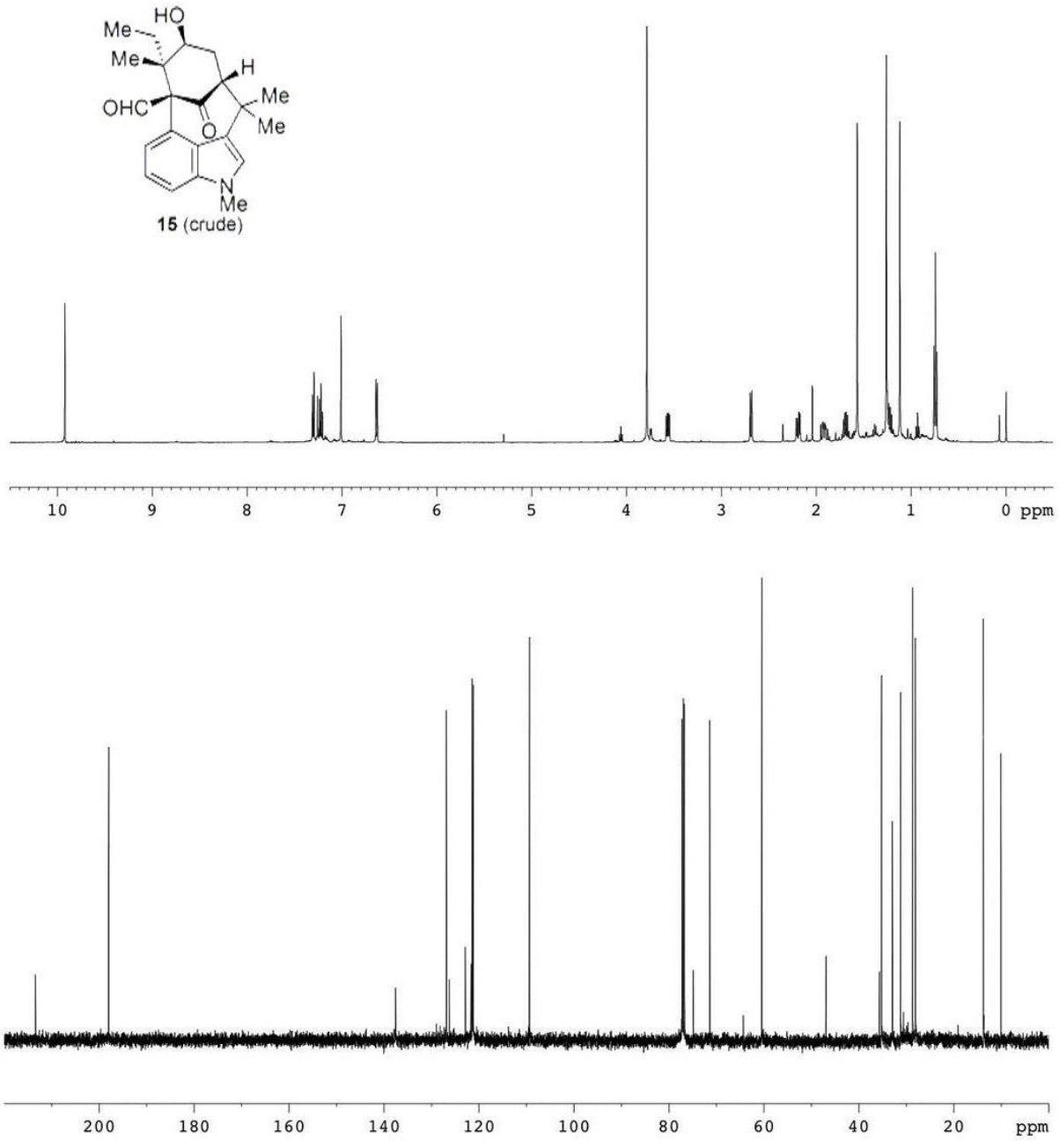


Figure S3. ^{1}H (500 MHz, $CDCl_3$) and ^{13}C (125 MHz, $CDCl_3$) NMR spectra for compound **15**.

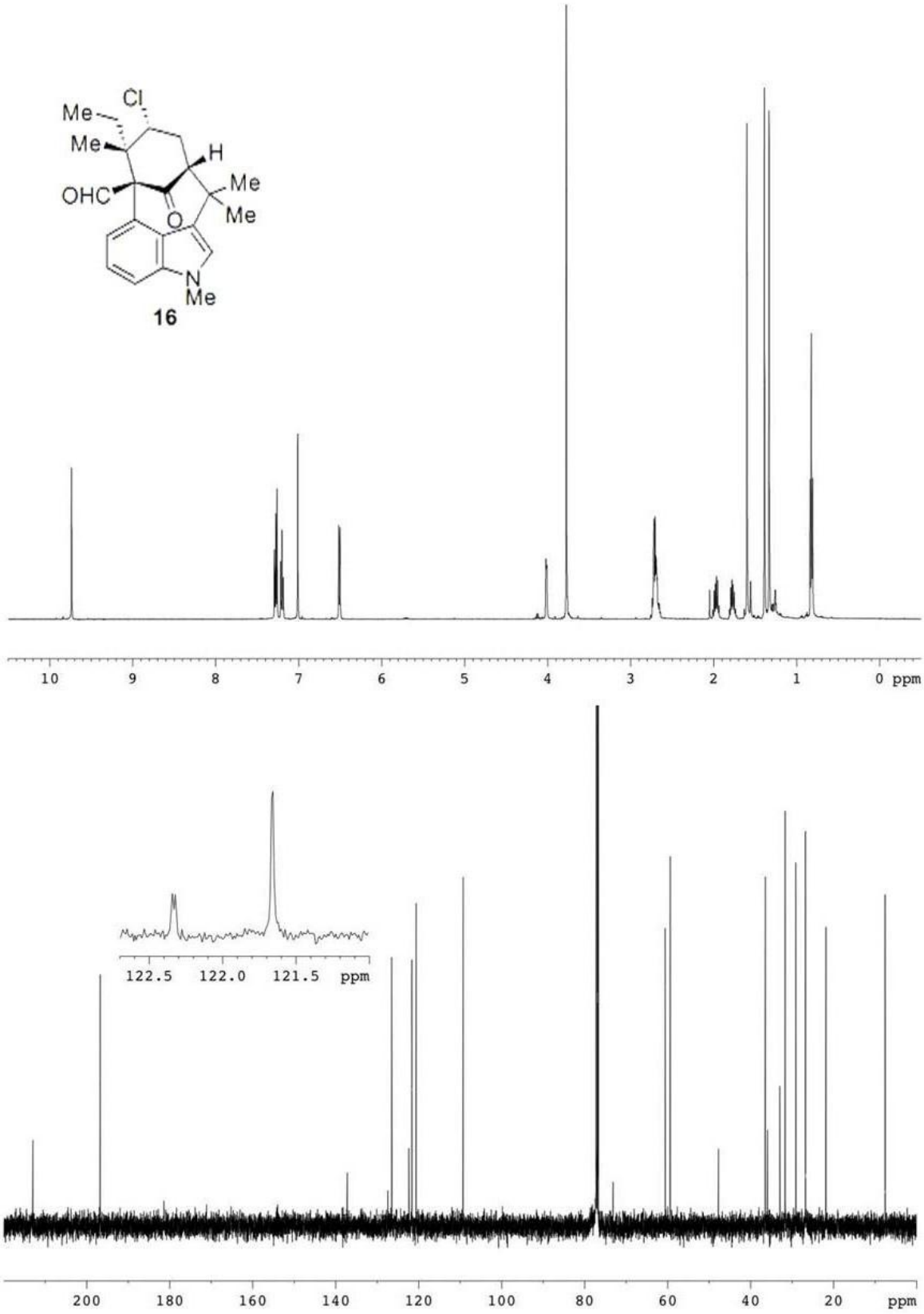


Figure S4. ^{1}H (500 MHz, $CDCl_3$) and ^{13}C (125 MHz, $CDCl_3$) NMR spectra for compound **16**.

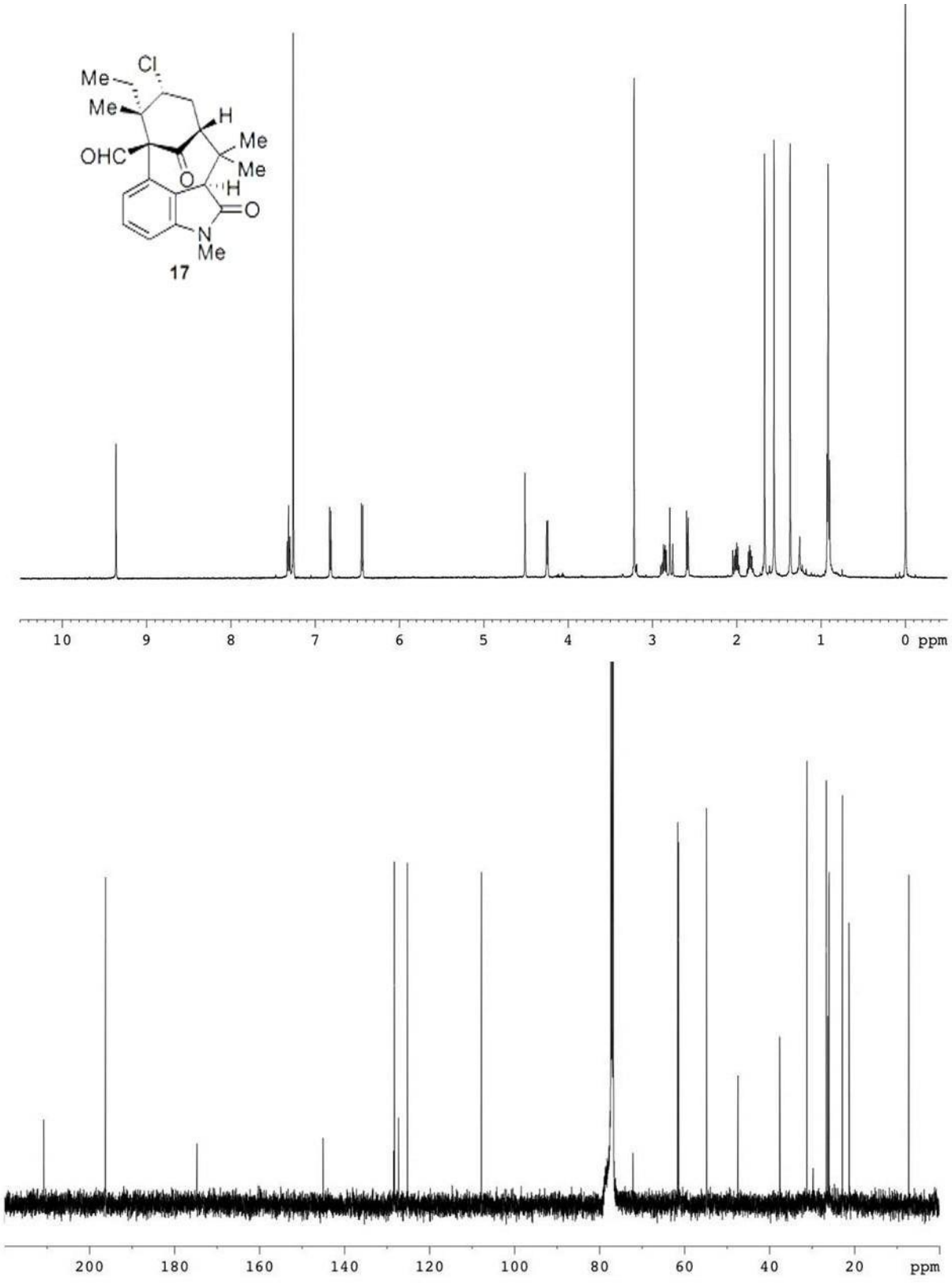


Figure S5. ^{1}H (500 MHz, $CDCl_3$) and ^{13}C (125 MHz, $CDCl_3$) NMR spectra for compound **17**.

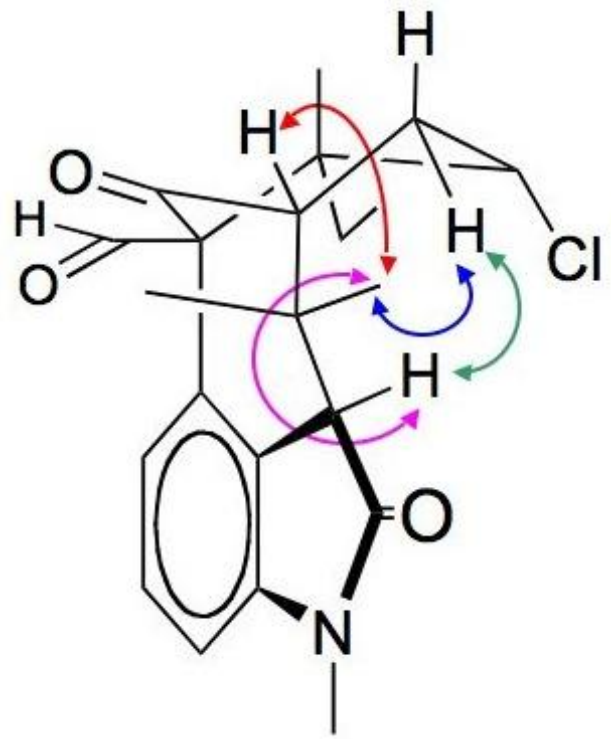
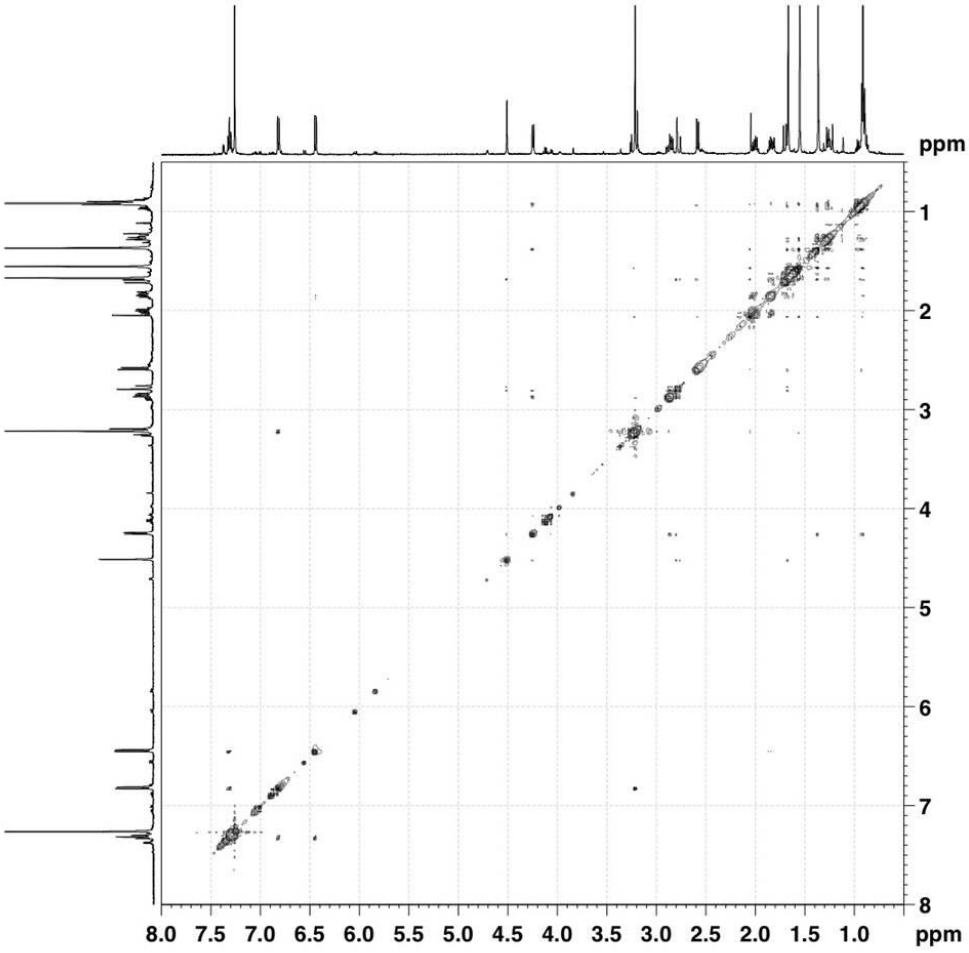


Figure S6a. Key NOE correlations observed in oxindole **17**.

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TD                      2048
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DS                        16
SWH                4770.992 Hz
FIDRES             2.329586 Hz
AQ                 0.2146804 sec
RG                     724.1
DW                   104.800 usec
DE                     7.50 usec
TE                    295.0 K
d0                 0.00009799 sec
D1                 2.39000010 sec
D8                 1.13999999 sec
D16                0.00020000 sec
IN0                0.00020960 sec
ST1CNT                     0
TAU                0.56479996 sec

======== CHANNEL f1 ========
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P1                      5.35 usec
p2                     10.70 usec
PL1                     0.00 dB
SFO1            499.8726208 MHz

====== GRADIENT CHANNEL =====
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GPNAM2               SINE.100
GPZ1                    40.00 %
GPZ2                   -40.00 %
P16                   5000.00 usec
ND0                        1
TD                       256
SFO1                499.8726 MHz
FIDRES             18.636688 Hz
SW                      9.544 ppm
FnMODE           States-TPPI
SI                      1024
SF                499.8700185 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.00
SI                      1024
MC2              States-TPPI
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SSB                        2
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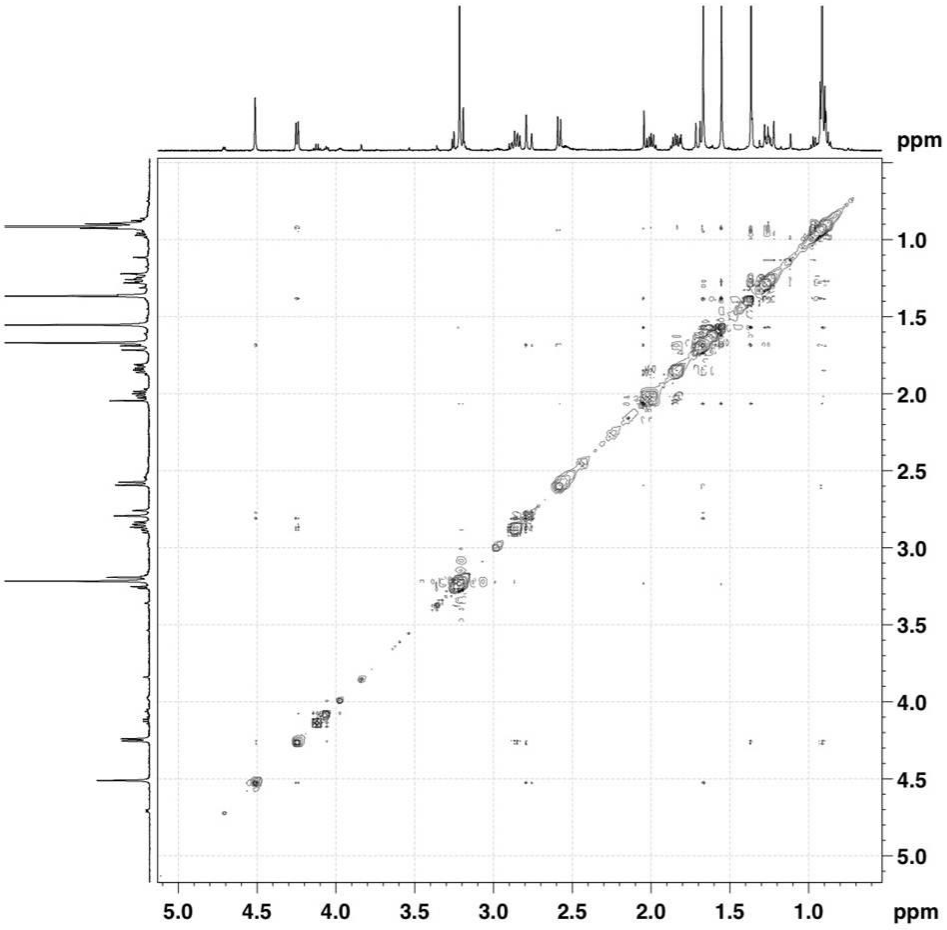
GB                         0
```

Figure S6b. NOESY for compound **17**.

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PROCNO                 1
Date_           20110429
Time               15.19
INSTRUM            spect
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PULPROG        noesygpph
TD                  2048
SOLVENT             CDCl3
NS                     8
DS                    16
SWH             4770.992 Hz
FIDRES          2.329586 Hz
AQ              0.2146804 sec
RG                 724.1
DW                104.800 usec
DE                  7.50 usec
TE                295.0 K
d0              0.00009799 sec
D1              2.39000010 sec
D8              1.13999999 sec
D16             0.00020000 sec
IN0             0.00020960 sec
ST1CNT                 0
TAU             0.56479996 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  5.35 usec
p2                 10.70 usec
PL1                 0.00 dB
SFO1         499.8726208 MHz

====== GRADIENT CHANNEL =====
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GPNAM2           SINE.100
GPZ1                40.00 %
GPZ2               -40.00 %
P16              5000.00 usec
ND0                    1
TD                   256
SFO1            499.8726 MHz
FIDRES         18.636688 Hz
SW                 9.544 ppm
FnMODE       States-TPPI
SI                  1024
SF           499.8700185 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.00
SI                  1024
MC2          States-TPPI
SF           499.8700095 MHz
WDW                QSINE
SSB                    2
LB                  0.00 Hz
GB                     0
```

Figure S6c. NOESY for compound **17**.

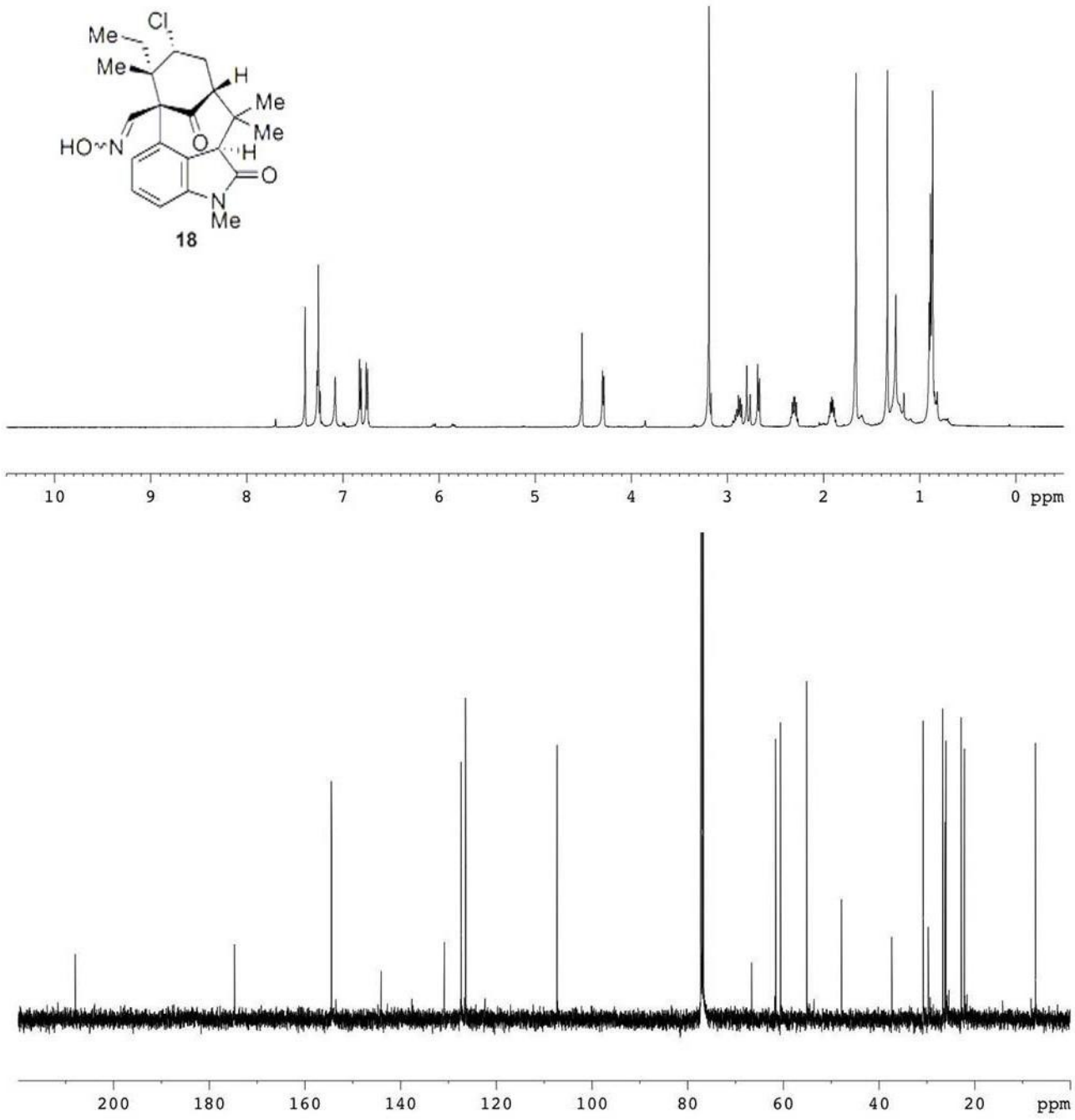


Figure S7. ^{1}H (500 MHz, $CDCl_3$) and ^{13}C (125 MHz, $CDCl_3$) NMR spectra for compound **18**.

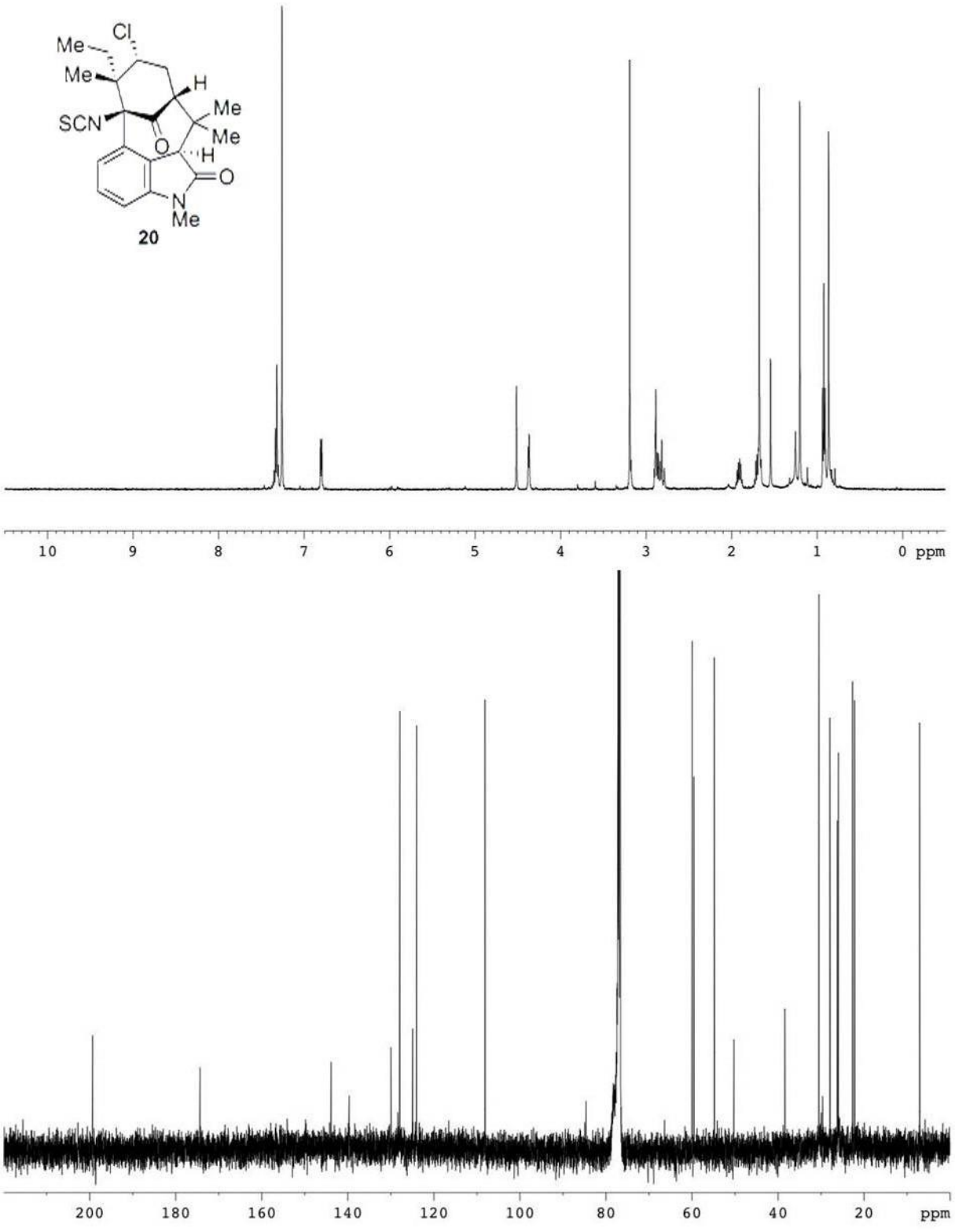


Figure S8. ^{1}H (500 MHz, $CDCl_3$) and ^{13}C (125 MHz, $CDCl_3$) NMR spectra for compound **20**.

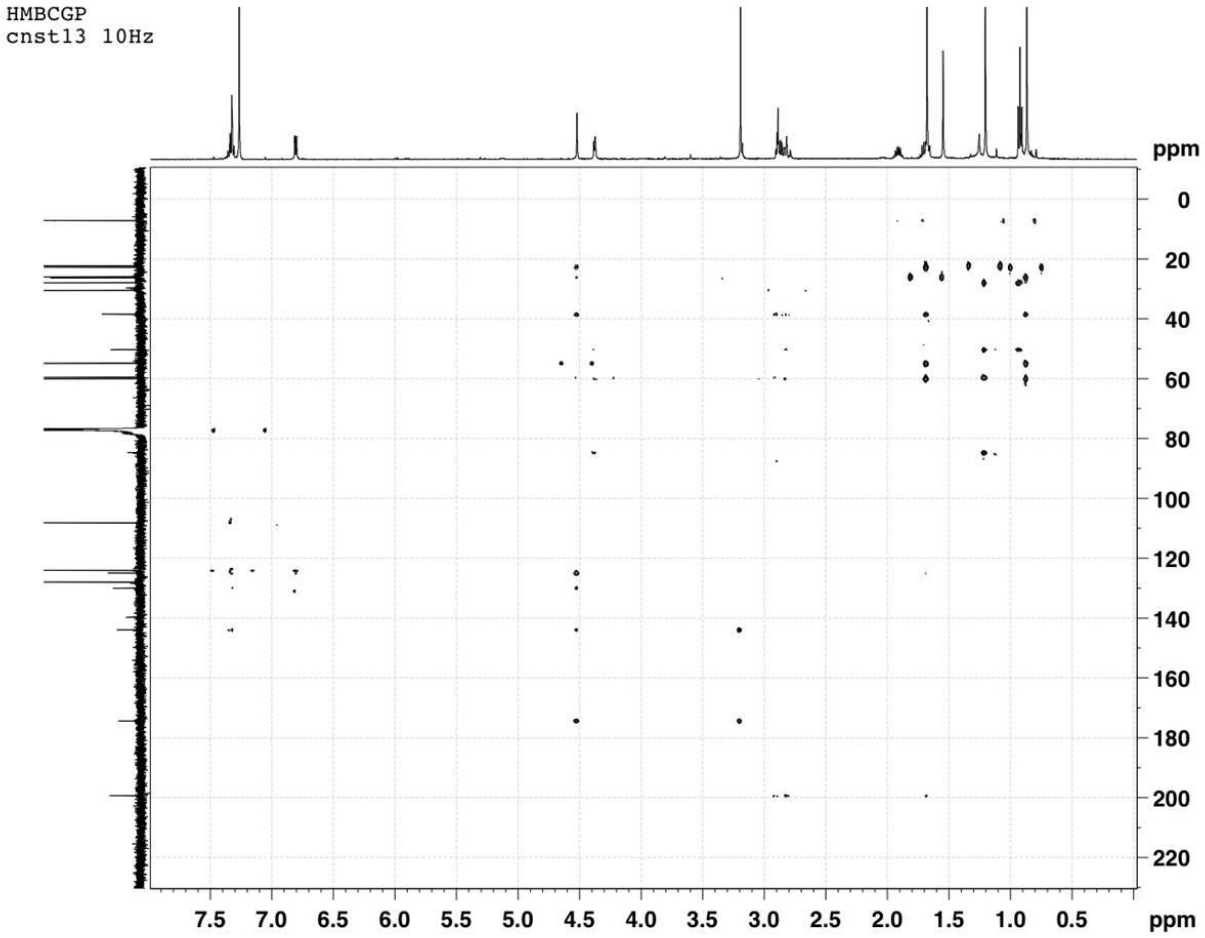


Figure S9. HMBC for compound **20**.

Crystallographic Experimental Section

Cyclopropane 10 (CCDC 829859)

Data Collection

An irregular broken fragment (0.40 x 0.16 x 0.16 mm) was selected under a stereo-microscope while immersed in Fluorolube oil to avoid possible reaction with air. The crystal was removed from the oil using a tapered glass fiber that also served to hold the crystal for data collection. The crystal was mounted and centered on a Bruker SMART APEX system at 100 K. Rotation and still images showed the diffractions to be sharp. Frames separated in reciprocal space were obtained and provided an orientation matrix and initial cell parameters. Final cell parameters were obtained from the full data set.

A "full sphere" data set was obtained which samples approximately all of reciprocal space to a resolution of 0.84 Å using 0.3^{o} steps in ω using 10 second integration times for each frame. Data collection was made at 100 K. Integration of intensities and refinement of cell parameters were done using SAINT [1]. Absorption corrections were applied using SADABS [1] based on redundant diffractions.

Structure solution and refinement

The space group was determined as $P2_1/c$ based on systematic absences and intensity statistics. Direct methods were used to locate the Cl atom and most C atoms from the E-map. Repeated difference Fourier maps allowed recognition of all expected C, N O and Cl atoms. Following anisotropic refinement of all non-H atoms, ideal H atom positions were calculated. Final refinement was anisotropic for all non-H atoms and isotropic-riding for H atoms. The R value at this point was 0.12 and twinning was suspected. Using Cell-Now, two crystal orientations were derived and following integration using SAINT, an HKLF5 file was used to reduce the R to the present value of 0.06. No anomalous bond lengths or thermal parameters were noted. All ORTEP diagrams have been drawn with 50% probability ellipsoids.

Equations of interest:

$R_{int} = \Sigma \mid F_o^2 - \langle F_o^2 \rangle \mid / \Sigma \mid F_o^2 \mid$ $R1 = \Sigma \mid \mid F_o \mid - \mid F_c \mid \mid / \Sigma \mid F_o \mid$

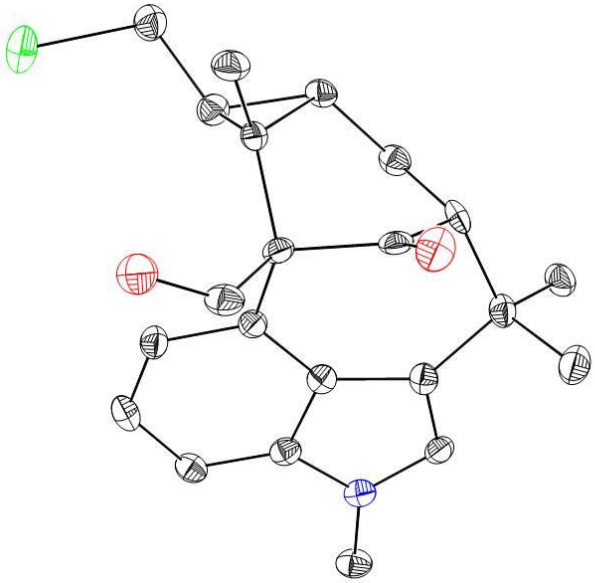
$wR2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2)^2]]^{1/2}$ $GooF = S = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)^{1/2}$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$; n = number of independent reflections;

q, a, b, P as defined in [1] p = number of parameters refined.

References

[1] All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).



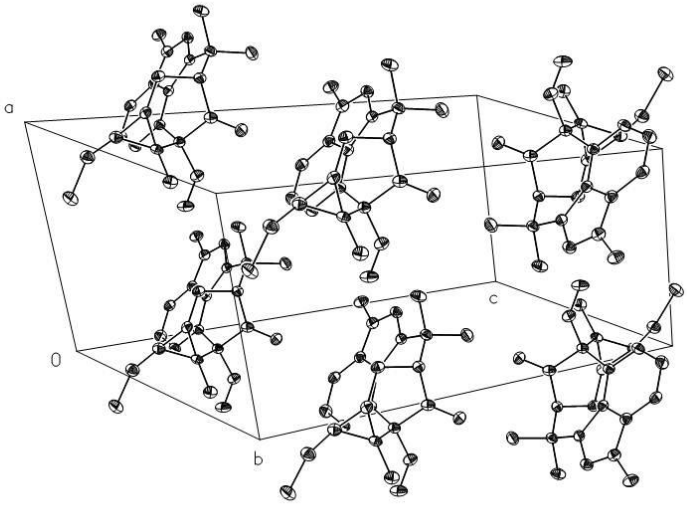


Table 1. Crystal and structure refinement for Bhat04.

Identification Code	Bhat04	
Empirical formula	$C_{22}H_{24}ClNO_2$	
Formula weight	369.87	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space Group	$P2_1/c$	
Unit cell dimensions	a = 8.941(4) Å	α = 90.0 °
	b = 9.465(4) Å	β = 110.502(14) °
	c = 22.998(8) Å	γ = 90.0 °
Volume	1823.0(13) Å^3	
Z	8	
Density (calculated)	1.348 Mg/m^3	
Absorption coefficient	0.226 mm^{-1}	
F(000)	784	
Crystal size, color, habit	0.40 x 0.16 x 0.16mm, clear, fragment	
Theta range for data collection	1.89 – 25.02 °	
Index ranges	$-10 \le h \le 10$, $-11 \le k \le 0$, $-27 \le l \le 9$	
Reflections collected	3,587	
Independent reflections	3,699 (R_{int} = 0.0619)	
Reflections with I > 4σ(F_o)	3,577	
Absorption correction	SADABS based on redundant diffractions	
Max. and min. transmission	1.0, 0.632	
Refinement method	Full-matrix least squares on F^2	
Weighting scheme	$w = q\,[\sigma^2 (F_o^2) + (aP)^2 + bP]^{-1}$ where:	
	$P = (F_o^2 + 2F_c^2)/3$, a = 0.1021, b = 2.346, q =1	
Data / restraints / parameters	3699 / 0 / 240	
Goodness-of-fit on F^2	1.093	
Final R indices [I > 2 sigma(I)]	R1 = 0.0627, wR2 = 0.1734	
R indices (all data)	R1 = 0.0651, wR2 = 0.1760	
Largest diff. peak and hole	0.357, -0.338 eÅ^{-3}	

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [Å^2 x 10^3] for Bhat04. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
C(1)	11792(4)	11520(4)	9945(2)	24(1)	
C(2)	12247(4)	9007(4)	9720(2)	19(1)	
C(3)	11501(4)	7948(4)	9321(2)	18(1)	
C(4)	9978(4)	8512(3)	8939(2)	17(1)	
C(5)	9941(4)	9948(4)	9124(2)	18(1)	
C(6)	8700(4)	10875(3)	8827(2)	19(1)	
C(7)	7449(4)	10367(4)	8331(2)	23(1)	
C(8)	7399(4)	8950(4)	8154(2)	20(1)	
C(9)	8610(4)	7995(4)	8451(2)	16(1)	
C(10)	8269(4)	6428(3)	8287(2)	16(1)	
C(11)	6756(4)	6157(4)	8437(2)	20(1)	
C(12)	9578(4)	5425(4)	8695(2)	17(1)	
C(13)	11276(4)	5696(3)	8706(2)	17(1)	
C(14)	12230(4)	6503(4)	9305(2)	19(1)	
C(15)	12298(5)	5655(4)	9883(2)	25(1)	
C(16)	13956(4)	6691(4)	9316(2)	24(1)	
C(17)	11230(4)	6418(4)	8102(2)	20(1)	
C(18)	9811(4)	5933(4)	7554(2)	17(1)	
C(19)	8183(4)	5921(4)	7634(2)	17(1)	
C(20)	7123(4)	4659(4)	7378(2)	22(1)	
C(21)	8510(4)	6960(4)	7197(2)	21(1)	
C(22)	7783(4)	6789(4)	6511(2)	25(1)	
Cl(1)	5781(1)	7514(1)	6227(1)	33(1)	
N(1)	11331(4)	10192(3)	9616(1)	19(1)	
O(1)	9247(3)	4417(3)	8945(1)	25(1)	
O(2)	5428(3)	6129(3)	8071(1)	27(1)	

Table 3. Bond lengths [Å] and angles [°] for Bhat04.

C(1)-N(1)	1.450(5)	C(10)-C(19)	1.553(4)
C(2)-N(1)	1.360(5)	C(11)-O(2)	1.191(4)
C(2)-C(3)	1.363(5)	C(12)-O(1)	1.203(4)
C(3)-C(4)	1.440(5)	C(12)-C(13)	1.531(5)
C(3)-C(14)	1.521(5)	C(13)-C(17)	1.537(5)
C(4)-C(9)	1.425(5)	C(13)-C(14)	1.545(5)
C(4)-C(5)	1.429(5)	C(14)-C(15)	1.535(5)
C(5)-N(1)	1.377(5)	C(14)-C(16)	1.545(5)
C(5)-C(6)	1.391(5)	C(17)-C(18)	1.513(5)
C(6)-C(7)	1.375(5)	C(18)-C(21)	1.517(5)
C(7)-C(8)	1.397(5)	C(18)-C(19)	1.530(4)
C(8)-C(9)	1.392(5)	C(19)-C(21)	1.506(5)
C(9)-C(10)	1.535(5)	C(19)-C(20)	1.510(5)
C(10)-C(11)	1.530(5)	C(21)-C(22)	1.489(5)
C(10)-C(12)	1.543(5)	C(22)-Cl(1)	1.812(4)

N(1)-C(2)-C(3)	111.5(3)	C(12)-C(13)-C(17)	110.3(3)
C(2)-C(3)-C(4)	105.9(3)	C(12)-C(13)-C(14)	109.5(3)
C(2)-C(3)-C(14)	124.3(3)	C(17)-C(13)-C(14)	114.7(3)
C(4)-C(3)-C(14)	129.8(3)	C(3)-C(14)-C(15)	109.7(3)
C(9)-C(4)-C(5)	117.2(3)	C(3)-C(14)-C(13)	111.0(3)
C(9)-C(4)-C(3)	136.1(3)	C(15)-C(14)-C(13)	110.8(3)
C(5)-C(4)-C(3)	106.6(3)	C(3)-C(14)-C(16)	109.3(3)
N(1)-C(5)-C(6)	129.3(3)	C(15)-C(14)-C(16)	108.1(3)
N(1)-C(5)-C(4)	107.2(3)	C(13)-C(14)-C(16)	107.9(3)
C(6)-C(5)-C(4)	123.5(3)	C(18)-C(17)-C(13)	111.5(3)
C(7)-C(6)-C(5)	117.6(3)	C(17)-C(18)-C(21)	121.1(3)
C(6)-C(7)-C(8)	120.7(3)	C(17)-C(18)-C(19)	117.3(3)
C(9)-C(8)-C(7)	122.7(3)	C(21)-C(18)-C(19)	59.3(2)
C(8)-C(9)-C(4)	118.0(3)	C(21)-C(19)-C(20)	119.9(3)
C(8)-C(9)-C(10)	116.9(3)	C(21)-C(19)-C(18)	60.0(2)
C(4)-C(9)-C(10)	124.5(3)	C(20)-C(19)-C(18)	116.9(3)
C(11)-C(10)-C(9)	102.8(3)	C(21)-C(19)-C(10)	119.2(3)
C(11)-C(10)-C(12)	107.6(3)	C(20)-C(19)-C(10)	115.7(3)
C(9)-C(10)-C(12)	113.7(3)	C(18)-C(19)-C(10)	112.8(3)
C(11)-C(10)-C(19)	114.7(3)	C(22)-C(21)-C(19)	121.4(3)
C(9)-C(10)-C(19)	118.4(3)	C(22)-C(21)-C(18)	118.1(3)
C(12)-C(10)-C(19)	99.6(3)	C(19)-C(21)-C(18)	60.8(2)
O(2)-C(11)-C(10)	125.9(3)	C(21)-C(22)-Cl(1)	110.5(3)
O(1)-C(12)-C(13)	122.2(3)	C(2)-N(1)-C(5)	108.7(3)
O(1)-C(12)-C(10)	121.1(3)	C(2)-N(1)-C(1)	125.5(3)
C(13)-C(12)-C(10)	116.4(3)	C(5)-N(1)-C(1)	125.7(3)

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for Bhat04. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+ ... + 2hka^*b^*U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	24(2)	23(2)	25(2)	-8(2)	10(2)	-7(2)
C(2)	17(2)	24(2)	14(2)	-1(1)	5(1)	0(1)
C(3)	15(2)	20(2)	19(2)	3(1)	7(1)	0(1)
C(4)	16(2)	17(2)	17(2)	1(1)	7(1)	-1(1)
C(5)	18(2)	21(2)	17(2)	0(1)	8(1)	-3(1)
C(6)	20(2)	14(2)	24(2)	-2(1)	10(1)	-2(1)
C(7)	19(2)	22(2)	29(2)	3(2)	10(2)	6(1)
C(8)	15(2)	25(2)	19(2)	1(1)	4(1)	2(2)
C(9)	14(2)	20(2)	15(2)	-1(1)	6(1)	0(1)
C(10)	16(2)	19(2)	12(2)	-4(1)	4(1)	-2(1)
C(11)	22(2)	18(2)	21(2)	-6(1)	9(2)	-4(1)
C(12)	18(2)	19(2)	14(2)	-8(1)	5(1)	-3(1)
C(13)	13(2)	14(2)	26(2)	1(1)	7(1)	5(1)
C(14)	14(2)	19(2)	21(2)	1(1)	3(1)	2(1)
C(15)	26(2)	22(2)	22(2)	3(2)	2(2)	2(2)
C(16)	16(2)	26(2)	27(2)	-5(2)	5(1)	3(2)
C(17)	17(2)	20(2)	23(2)	-2(1)	8(1)	-1(1)
C(18)	17(2)	21(2)	17(2)	0(1)	11(1)	1(1)
C(19)	12(2)	20(2)	17(2)	-3(1)	4(1)	2(1)
C(20)	23(2)	19(2)	23(2)	-7(1)	7(1)	-1(2)
C(21)	20(2)	22(2)	20(2)	-2(1)	6(1)	-1(1)
C(22)	21(2)	30(2)	24(2)	0(2)	7(2)	2(2)
Cl(1)	23(1)	40(1)	29(1)	7(1)	1(1)	4(1)
N(1)	20(1)	21(1)	18(1)	-3(1)	8(1)	-3(1)
O(1)	26(1)	18(1)	27(1)	2(1)	5(1)	-5(1)
O(2)	16(1)	38(2)	29(1)	-8(1)	8(1)	-5(1)

Table 5. Hydrogen coordinates [x 10^4] and isotropic displacement parameters [Å^2 x 10^3] for Bhat04.

	x	y	z	U(eq)
H(1A)	12407	12081	9749	35
H(1B)	12447	11333	10379	35
H(1C)	10832	12044	9929	35
H(2)	13277	8926	10030	22
H(6)	8718	11826	8961	23
H(7)	6609	10985	8106	28
H(8)	6501	8625	7818	24
H(11)	6889	5998	8860	24
H(13)	11800	4755	8725	21
H(15A)	12994	6140	10256	38
H(15B)	12723	4709	9861	38
H(15C)	11221	5573	9899	38
H(16A)	13955	7326	8979	36
H(16B)	14389	5770	9260	36
H(16C)	14620	7096	9715	36
H(17A)	11174	7455	8148	23
H(17B)	12226	6200	8024	23
H(18)	10017	5136	7307	20
H(20A)	6002	4956	7232	33
H(20B)	7303	3946	7705	33
H(20C)	7376	4256	7031	33
H(21)	8676	7959	7348	25
H(22A)	7746	5774	6404	30
H(22B)	8450	7279	6309	30

Table 6. Torsion angles [°] for Bhat04.

N(1)-C(2)-C(3)-C(4)	-0.6(4)	C(2)-C(3)-C(14)-C(16)	-46.9(4)
N(1)-C(2)-C(3)-C(14)	178.2(3)	C(4)-C(3)-C(14)-C(16)	131.6(4)
C(2)-C(3)-C(4)-C(9)	-178.0(4)	C(12)-C(13)-C(14)-C(3)	-63.0(4)
C(14)-C(3)-C(4)-C(9)	3.2(7)	C(17)-C(13)-C(14)-C(3)	61.5(4)
C(2)-C(3)-C(4)-C(5)	2.5(4)	C(12)-C(13)-C(14)-C(15)	59.1(4)
C(14)-C(3)-C(4)-C(5)	-176.2(3)	C(17)-C(13)-C(14)-C(15)	-176.3(3)
C(9)-C(4)-C(5)-N(1)	176.9(3)	C(12)-C(13)-C(14)-C(16)	177.2(3)
C(3)-C(4)-C(5)-N(1)	-3.5(4)	C(17)-C(13)-C(14)-C(16)	-58.2(4)
C(9)-C(4)-C(5)-C(6)	-4.5(5)	C(12)-C(13)-C(17)-C(18)	-33.2(4)
C(3)-C(4)-C(5)-C(6)	175.1(3)	C(14)-C(13)-C(17)-C(18)	-157.3(3)
N(1)-C(5)-C(6)-C(7)	178.5(3)	C(13)-C(17)-C(18)-C(21)	116.7(3)
C(4)-C(5)-C(6)-C(7)	0.3(5)	C(13)-C(17)-C(18)-C(19)	47.8(4)
C(5)-C(6)-C(7)-C(8)	3.0(5)	C(17)-C(18)-C(19)-C(21)	111.7(4)
C(6)-C(7)-C(8)-C(9)	-1.9(6)	C(17)-C(18)-C(19)-C(20)	-137.7(3)
C(7)-C(8)-C(9)-C(4)	-2.5(5)	C(21)-C(18)-C(19)-C(20)	110.6(3)
C(7)-C(8)-C(9)-C(10)	169.5(3)	C(17)-C(18)-C(19)-C(10)	0.0(4)
C(5)-C(4)-C(9)-C(8)	5.4(5)	C(21)-C(18)-C(19)-C(10)	-111.6(3)
C(3)-C(4)-C(9)-C(8)	-174.0(4)	C(11)-C(10)-C(19)-C(21)	124.5(3)
C(5)-C(4)-C(9)-C(10)	-165.9(3)	C(9)-C(10)-C(19)-C(21)	2.8(4)
C(3)-C(4)-C(9)-C(10)	14.7(6)	C(12)-C(10)-C(19)-C(21)	-120.9(3)
C(8)-C(9)-C(10)-C(11)	-56.6(4)	C(11)-C(10)-C(19)-C(20)	-29.9(4)
C(4)-C(9)-C(10)-C(11)	114.8(4)	C(9)-C(10)-C(19)-C(20)	-151.7(3)
C(8)-C(9)-C(10)-C(12)	-172.6(3)	C(12)-C(10)-C(19)-C(20)	84.6(3)
C(4)-C(9)-C(10)-C(12)	-1.2(5)	C(11)-C(10)-C(19)-C(18)	-168.3(3)
C(8)-C(9)-C(10)-C(19)	71.0(4)	C(9)-C(10)-C(19)-C(18)	70.0(4)
C(4)-C(9)-C(10)-C(19)	-117.6(3)	C(12)-C(10)-C(19)-C(18)	-53.7(3)
C(9)-C(10)-C(11)-O(2)	99.4(4)	C(20)-C(19)-C(21)-C(22)	1.2(5)
C(12)-C(10)-C(11)-O(2)	-140.3(4)	C(18)-C(19)-C(21)-C(22)	106.9(4)
C(19)-C(10)-C(11)-O(2)	-30.5(5)	C(10)-C(19)-C(21)-C(22)	-152.2(3)
C(11)-C(10)-C(12)-O(1)	16.7(4)	C(20)-C(19)-C(21)-C(18)	-105.7(3)
C(9)-C(10)-C(12)-O(1)	129.8(3)	C(10)-C(19)-C(21)-C(18)	100.9(3)
C(19)-C(10)-C(12)-O(1)	-103.3(3)	C(17)-C(18)-C(21)-C(22)	142.5(3)
C(11)-C(10)-C(12)-C(13)	-169.4(3)	C(19)-C(18)-C(21)-C(22)	-112.2(4)
C(9)-C(10)-C(12)-C(13)	-56.3(4)	C(17)-C(18)-C(21)-C(19)	-105.3(3)
C(19)-C(10)-C(12)-C(13)	70.6(3)	C(19)-C(21)-C(22)-Cl(1)	83.3(4)
O(1)-C(12)-C(13)-C(17)	147.2(3)	C(18)-C(21)-C(22)-Cl(1)	154.5(3)
C(10)-C(12)-C(13)-C(17)	-26.6(4)	C(3)-C(2)-N(1)-C(5)	-1.6(4)
O(1)-C(12)-C(13)-C(14)	-85.7(4)	C(3)-C(2)-N(1)-C(1)	-177.9(3)
C(10)-C(12)-C(13)-C(14)	100.5(3)	C(6)-C(5)-N(1)-C(2)	-175.3(4)
C(2)-C(3)-C(14)-C(15)	71.4(4)	C(4)-C(5)-N(1)-C(2)	3.2(4)
C(4)-C(3)-C(14)-C(15)	-110.1(4)	C(6)-C(5)-N(1)-C(1)	1.0(6)
C(2)-C(3)-C(14)-C(13)	-165.9(3)	C(4)-C(5)-N(1)-C(1)	179.4(3)
C(4)-C(3)-C(14)-C(13)	12.7(5)		

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Data Collection

An irregular broken fragment (0.28 x 0.24 x 0.24 mm) was selected under a stereo-microscope while immersed in Fluorolube oil to avoid possible reaction with air. The crystal was removed from the oil using a tapered glass fiber that also served to hold the crystal for data collection. The crystal was mounted and centered on a Bruker SMART APEX system at 100 K. Rotation and still images showed the diffractions to be rather diffuse. Frames separated in reciprocal space were obtained and provided an orientation matrix and initial cell parameters. Final cell parameters were obtained from the full data set.

A "full sphere" data set was obtained which samples approximately all of reciprocal space to a resolution of 0.84 Å using 0.3° steps in ω using 10 second integration times for each frame. Data collection was made at 100 K. Integration of intensities and refinement of cell parameters were done using SAINT [1]. Absorption corrections were applied using SADABS [1] based on redundant diffractions. The integration of intensities maintained a large box of enclosure due to the large and poorly shaped diffractions.

Structure solution and refinement

The space group was determined as P1(bar) based on systematic absences and intensity statistics. Direct methods were used to locate most C atoms from the E-map. Repeated difference Fourier maps allowed recognition of all expected C, N and O atoms. Following anisotropic refinement of all non-H atoms, ideal H-atom positions were calculated. Final refinement was anisotropic for all non-H atoms, and isotropic-riding for H atoms. The relatively high R of 9.5% is most certainly due to the broad and irregularly shaped diffractions which prevented a good integration. No anomalous bond lengths or thermal parameters were noted. All ORTEP diagrams have been drawn with 50% probability ellipsoids.

Equations of interest:

$R_{int} = \Sigma \mid F_o^2 - \langle F_o^2 \rangle \mid / \Sigma \mid F_o^2 \mid$ $\qquad$ $R1 = \Sigma \mid \mid F_o \mid - \mid F_c \mid \mid / \Sigma \mid F_o \mid$

$wR2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2)^2]]^{1/2}$ $GooF = S = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$; n = number of independent reflections;

q, a, b, P as defined in [1] p = number of parameters refined.

References

[1] All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).

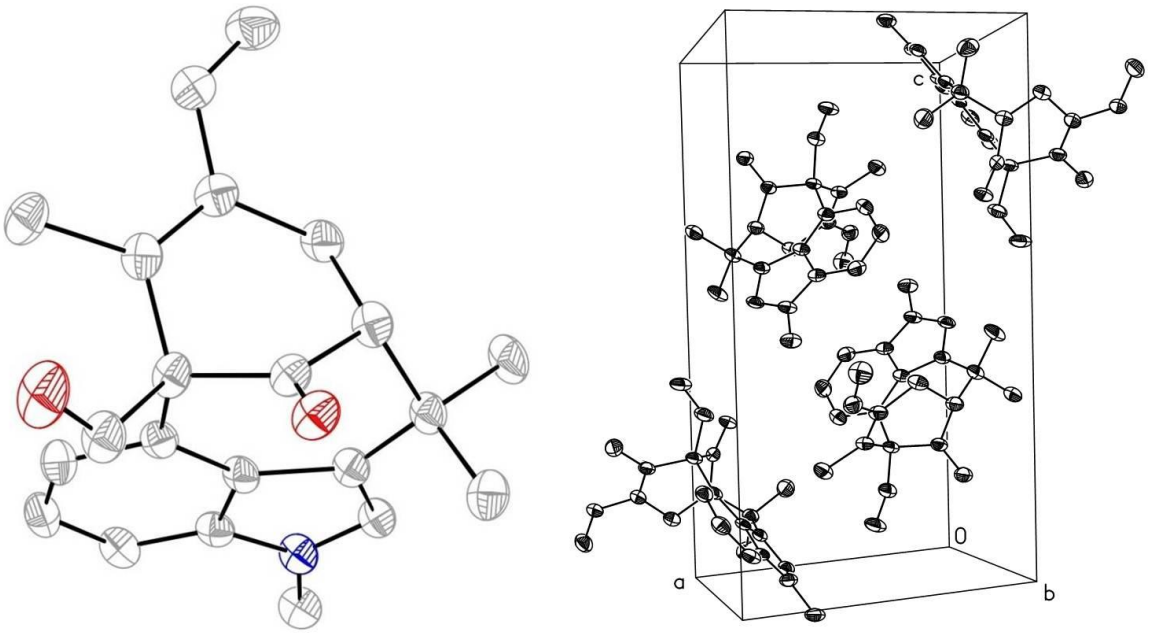


Table 1. Crystal and structure refinement for Bhat03.

Identification Code	Bhat03	
Empirical formula	$C_{44}H_{46}N_2O_4$	
Formula weight	666.83	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space Group	P1(bar)	
Unit cell dimensions	a = 9.579(5) Å	α = 84.784(9) °
	b = 10.734(5) Å	β = 89.891(9) °
	c = 16.874(8) Å	γ = 75.579(8) °
Volume	1673.0(14) Å^3	
Z	2	
Density (calculated)	1.324 Mg/m^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	712	
Crystal size, color, habit	0.28 x 0.24 x 0.24mm, clear, fragment	
Theta range for data collection	1.21 – 25.11 °	
Index ranges	$-11 \le h \le 11$, $-12 \le k \le 12$, $-20 \le l \le 20$	
Reflections collected	16,310	
Independent reflections	5, 935 (R_{int} = 0.0593)	
Reflections with I > 4σ(F_o)	4,025	
Absorption correction	SADABS based on redundant diffractions	
Max. and min. transmission	1.0, 0.388	
Refinement method	Full-matrix least squares on F^2	
Weighting scheme	$w = q\,[\sigma^2(F_o^2) + (aP)^2 + bP]^{-1}$ where:	
	$P = (F_o^2 + 2F_c^2)/3$, a = 0.1679, b = 0.0, q =1	
Data / restraints / parameters	5935 / 0 / 459	
Goodness-of-fit on F^2	0.992	
Final R indices [I > 2 sigma(I)]	R1 = 0.0938, wR2 = 0.2320	
R indices (all data)	R1 = 0.1194, wR2 = 0.2498	
Largest diff. peak and hole	0.872, -0.363 eÅ^{-3}	

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [Å^2 x 10^3] for Bhat03. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
C(1)	2571(4)	3823(4)	5443(2)	32(1)	
C(2)	3728(4)	4690(3)	4286(2)	24(1)	
C(3)	4899(4)	3660(3)	4215(2)	29(1)	
C(4)	5804(4)	3761(4)	3603(2)	32(1)	
C(5)	5540(4)	4859(3)	3072(2)	30(1)	
C(6)	4387(4)	5907(3)	3142(2)	24(1)	
C(7)	3449(4)	5840(3)	3772(2)	23(1)	
C(8)	2183(4)	6677(3)	4066(2)	24(1)	
C(9)	1789(4)	5999(3)	4702(2)	26(1)	
C(10)	1401(4)	8028(3)	3773(2)	26(1)	
C(11)	43(4)	8023(4)	3314(2)	31(1)	
C(12)	949(4)	8838(4)	4471(2)	35(1)	
C(13)	2386(4)	8685(3)	3252(2)	27(1)	
C(14)	2764(4)	7997(3)	2521(2)	23(1)	
C(15)	4220(4)	7046(3)	2516(2)	25(1)	
C(16)	4236(4)	6524(4)	1703(2)	31(1)	
C(17)	5334(4)	7791(3)	2659(2)	26(1)	
C(18)	6710(4)	7473(4)	2226(2)	35(1)	
C(19)	5061(4)	8645(3)	3207(2)	27(1)	
C(20)	6094(4)	9353(4)	3407(2)	30(1)	
C(21)	6009(5)	10140(4)	3960(3)	43(1)	
C(22)	3707(4)	8820(4)	3688(2)	29(1)	
C(23)	6826(4)	6099(4)	-455(2)	32(1)	
C(24)	8482(4)	5288(3)	709(2)	24(1)	
C(25)	9027(4)	6350(3)	772(2)	29(1)	
C(26)	9995(4)	6269(4)	1374(2)	30(1)	
C(27)	10368(4)	5173(3)	1909(2)	29(1)	
C(28)	9854(4)	4102(3)	1849(2)	25(1)	
C(29)	8885(3)	4133(3)	1216(2)	23(1)	
C(30)	8143(3)	3260(3)	921(2)	24(1)	
C(31)	7341(4)	3912(3)	294(2)	24(1)	
C(32)	8210(4)	1880(3)	1212(2)	26(1)	
C(33)	8259(4)	1079(4)	499(2)	37(1)	
C(34)	6866(4)	1799(4)	1677(2)	36(1)	
C(35)	9569(4)	1286(3)	1725(2)	25(1)	
C(36)	9516(3)	1951(3)	2470(2)	24(1)	
C(37)	10347(4)	2975(3)	2477(2)	26(1)	
C(38)	10032(4)	3479(4)	3289(2)	32(1)	
C(39)	11914(4)	2279(3)	2341(2)	26(1)	
C(40)	13059(4)	2643(4)	2793(2)	35(1)	
C(41)	12163(4)	1458(3)	1776(2)	26(1)	
C(42)	13626(4)	829(3)	1575(2)	28(1)	
C(43)	14006(4)	223(4)	939(2)	37(1)	
C(44)	10964(4)	1263(3)	1280(2)	26(1)	
N(1)	2684(3)	4813(3)	4838(2)	26(1)	
N(2)	7520(3)	5122(3)	158(2)	27(1)	
O(1)	1974(3)	8205(2)	1942(1)	29(1)	
O(2)	4790(3)	6852(3)	1118(2)	43(1)	
O(3)	8900(3)	1646(2)	3052(1)	31(1)	
O(4)	10755(3)	3124(3)	3875(2)	48(1)	

Table 3. Bond lengths [Å] and angles [°] for Bhat03.

C(1)-N(1)	1.430(4)	C(23)-N(2)	1.439(4)
C(2)-N(1)	1.355(4)	C(24)-N(2)	1.363(4)
C(2)-C(3)	1.379(5)	C(24)-C(25)	1.379(5)
C(2)-C(7)	1.410(5)	C(24)-C(29)	1.410(5)
C(3)-C(4)	1.361(5)	C(25)-C(26)	1.359(5)
C(4)-C(5)	1.384(5)	C(26)-C(27)	1.387(5)
C(5)-C(6)	1.380(5)	C(27)-C(28)	1.369(5)
C(6)-C(7)	1.400(5)	C(28)-C(29)	1.410(5)
C(6)-C(15)	1.518(5)	C(28)-C(37)	1.514(5)
C(7)-C(8)	1.434(5)	C(29)-C(30)	1.431(5)
C(8)-C(9)	1.348(5)	C(30)-C(31)	1.342(5)
C(8)-C(10)	1.497(5)	C(30)-C(32)	1.503(5)
C(9)-N(1)	1.346(5)	C(31)-N(2)	1.351(4)
C(10)-C(11)	1.517(5)	C(32)-C(34)	1.522(5)
C(10)-C(12)	1.525(5)	C(32)-C(35)	1.529(5)
C(10)-C(13)	1.542(5)	C(32)-C(33)	1.535(5)
C(13)-C(14)	1.493(5)	C(35)-C(36)	1.497(5)
C(13)-C(22)	1.509(5)	C(35)-C(44)	1.528(5)
C(14)-O(1)	1.207(4)	C(36)-O(3)	1.206(4)
C(14)-C(15)	1.510(5)	C(36)-C(37)	1.511(5)
C(15)-C(17)	1.516(5)	C(37)-C(38)	1.521(5)
C(15)-C(16)	1.526(5)	C(37)-C(39)	1.528(5)
C(16)-O(2)	1.187(4)	C(38)-O(4)	1.186(4)
C(17)-C(19)	1.341(5)	C(39)-C(41)	1.339(5)
C(17)-C(18)	1.485(5)	C(39)-C(40)	1.486(5)
C(19)-C(20)	1.446(5)	C(41)-C(42)	1.450(5)
C(19)-C(22)	1.510(5)	C(41)-C(44)	1.489(5)
C(20)-C(21)	1.303(5)	C(42)-C(43)	1.310(5)

N(1)-C(2)-C(3)	128.3(3)	C(13)-C(14)-C(15)	117.3(3)
N(1)-C(2)-C(7)	108.2(3)	C(14)-C(15)-C(17)	106.6(3)
C(3)-C(2)-C(7)	123.4(3)	C(14)-C(15)-C(6)	113.2(3)
C(4)-C(3)-C(2)	117.5(3)	C(17)-C(15)-C(6)	110.2(3)
C(3)-C(4)-C(5)	120.7(3)	C(14)-C(15)-C(16)	103.3(3)
C(6)-C(5)-C(4)	122.5(3)	C(17)-C(15)-C(16)	116.2(3)
C(5)-C(6)-C(7)	118.2(3)	C(6)-C(15)-C(16)	107.3(3)
C(5)-C(6)-C(15)	117.1(3)	O(2)-C(16)-C(15)	127.3(4)
C(7)-C(6)-C(15)	124.8(3)	C(19)-C(17)-C(18)	123.4(3)
C(6)-C(7)-C(2)	117.6(3)	C(19)-C(17)-C(15)	118.1(3)
C(6)-C(7)-C(8)	136.4(3)	C(18)-C(17)-C(15)	118.3(3)
C(2)-C(7)-C(8)	106.0(3)	C(17)-C(19)-C(20)	121.8(3)
C(9)-C(8)-C(7)	105.7(3)	C(17)-C(19)-C(22)	120.7(3)
C(9)-C(8)-C(10)	124.1(3)	C(20)-C(19)-C(22)	117.4(3)
C(7)-C(8)-C(10)	130.2(3)	C(21)-C(20)-C(19)	128.0(4)
N(1)-C(9)-C(8)	111.8(3)	C(13)-C(22)-C(19)	115.5(3)
C(8)-C(10)-C(11)	110.1(3)	N(2)-C(24)-C(25)	128.4(3)
C(8)-C(10)-C(12)	110.5(3)	N(2)-C(24)-C(29)	107.3(3)
C(11)-C(10)-C(12)	107.6(3)	C(25)-C(24)-C(29)	124.2(3)
C(8)-C(10)-C(13)	110.6(3)	C(26)-C(25)-C(24)	117.0(3)
C(11)-C(10)-C(13)	110.8(3)	C(25)-C(26)-C(27)	120.5(3)
C(12)-C(10)-C(13)	107.1(3)	C(28)-C(27)-C(26)	123.3(3)
C(14)-C(13)-C(22)	112.0(3)	C(27)-C(28)-C(29)	117.8(3)
C(14)-C(13)-C(10)	109.6(3)	C(27)-C(28)-C(37)	117.7(3)
C(22)-C(13)-C(10)	113.8(3)	C(29)-C(28)-C(37)	124.5(3)
O(1)-C(14)-C(13)	122.5(3)	C(28)-C(29)-C(24)	117.0(3)
O(1)-C(14)-C(15)	120.2(3)	C(28)-C(29)-C(30)	136.5(3)

C(24)-C(29)-C(30)	106.5(3)	C(28)-C(37)-C(38)	108.0(3)
C(31)-C(30)-C(29)	106.1(3)	C(36)-C(37)-C(39)	105.3(3)
C(31)-C(30)-C(32)	124.4(3)	C(28)-C(37)-C(39)	109.8(3)
C(29)-C(30)-C(32)	129.5(3)	C(38)-C(37)-C(39)	115.9(3)
C(30)-C(31)-N(2)	111.4(3)	O(4)-C(38)-C(37)	126.1(4)
C(30)-C(32)-C(34)	110.3(3)	C(41)-C(39)-C(40)	124.5(3)
C(30)-C(32)-C(35)	110.8(3)	C(41)-C(39)-C(37)	117.4(3)
C(34)-C(32)-C(35)	110.6(3)	C(40)-C(39)-C(37)	117.9(3)
C(30)-C(32)-C(33)	109.7(3)	C(39)-C(41)-C(42)	120.6(3)
C(34)-C(32)-C(33)	107.5(3)	C(39)-C(41)-C(44)	121.3(3)
C(35)-C(32)-C(33)	107.8(3)	C(42)-C(41)-C(44)	117.8(3)
C(36)-C(35)-C(44)	111.6(3)	C(43)-C(42)-C(41)	125.7(4)
C(36)-C(35)-C(32)	110.3(3)	C(41)-C(44)-C(35)	114.6(3)
C(44)-C(35)-C(32)	113.4(3)	C(9)-N(1)-C(2)	108.2(3)
O(3)-C(36)-C(35)	122.0(3)	C(9)-N(1)-C(1)	127.1(3)
O(3)-C(36)-C(37)	121.3(3)	C(2)-N(1)-C(1)	124.6(3)
C(35)-C(36)-C(37)	116.6(3)	C(31)-N(2)-C(24)	108.6(3)
C(36)-C(37)-C(28)	114.5(3)	C(31)-N(2)-C(23)	127.0(3)
C(36)-C(37)-C(38)	103.4(3)	C(24)-N(2)-C(23)	124.4(3)

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for Bhat03. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+ ... + 2hka^*b^*U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	43(2)	34(2)	21(2)	2(2)	4(2)	-11(2)
C(2)	31(2)	27(2)	16(2)	-5(1)	0(1)	-10(2)
C(3)	36(2)	26(2)	22(2)	-1(2)	-3(2)	-5(2)
C(4)	31(2)	31(2)	32(2)	-10(2)	2(2)	-1(2)
C(5)	30(2)	33(2)	25(2)	-7(2)	7(2)	-5(2)
C(6)	28(2)	27(2)	18(2)	-7(1)	3(1)	-9(1)
C(7)	29(2)	26(2)	16(2)	-7(1)	2(1)	-8(1)
C(8)	27(2)	30(2)	16(2)	-7(1)	2(1)	-7(1)
C(9)	31(2)	34(2)	15(2)	-10(2)	6(1)	-9(2)
C(10)	29(2)	28(2)	21(2)	-6(2)	7(2)	-6(1)
C(11)	31(2)	38(2)	23(2)	-2(2)	9(2)	-8(2)
C(12)	44(2)	33(2)	28(2)	-12(2)	17(2)	-5(2)
C(13)	31(2)	30(2)	18(2)	-2(2)	3(2)	-5(2)
C(14)	26(2)	28(2)	16(2)	1(1)	4(1)	-9(1)
C(15)	26(2)	36(2)	13(2)	-3(1)	4(1)	-8(2)
C(16)	32(2)	41(2)	20(2)	-7(2)	4(2)	-8(2)
C(17)	30(2)	32(2)	15(2)	0(2)	3(1)	-6(2)
C(18)	31(2)	48(2)	26(2)	-3(2)	8(2)	-12(2)
C(19)	32(2)	32(2)	18(2)	1(2)	2(2)	-10(2)
C(20)	31(2)	32(2)	27(2)	0(2)	-1(2)	-8(2)
C(21)	46(2)	45(3)	43(3)	-9(2)	0(2)	-21(2)
C(22)	37(2)	32(2)	21(2)	-8(2)	7(2)	-12(2)
C(23)	44(2)	33(2)	17(2)	1(2)	4(2)	-4(2)
C(24)	28(2)	31(2)	13(2)	-6(1)	6(1)	-3(2)
C(25)	35(2)	29(2)	22(2)	-2(2)	12(2)	-7(2)
C(26)	34(2)	30(2)	31(2)	-11(2)	9(2)	-12(2)
C(27)	28(2)	35(2)	24(2)	-11(2)	5(2)	-8(2)
C(28)	26(2)	31(2)	17(2)	-6(1)	8(1)	-7(2)

C(29)	25(2)	29(2)	14(2)	-5(1)	8(1)	-5(1)
C(30)	23(2)	33(2)	16(2)	-6(2)	6(1)	-6(1)
C(31)	26(2)	31(2)	16(2)	-8(1)	5(1)	-8(1)
C(32)	28(2)	32(2)	21(2)	-8(2)	3(1)	-10(2)
C(33)	45(2)	37(2)	32(2)	-11(2)	-7(2)	-11(2)
C(34)	32(2)	42(2)	35(2)	4(2)	1(2)	-12(2)
C(35)	29(2)	27(2)	21(2)	-2(2)	3(2)	-8(2)
C(36)	21(2)	29(2)	20(2)	-2(2)	0(1)	-2(1)
C(37)	28(2)	36(2)	14(2)	-5(2)	4(1)	-10(2)
C(38)	36(2)	42(2)	20(2)	-8(2)	9(2)	-12(2)
C(39)	28(2)	34(2)	16(2)	-2(2)	4(1)	-10(2)
C(40)	30(2)	46(2)	29(2)	-10(2)	-1(2)	-8(2)
C(41)	26(2)	31(2)	21(2)	-1(2)	5(2)	-7(2)
C(42)	28(2)	35(2)	20(2)	1(2)	5(2)	-6(2)
C(43)	33(2)	43(2)	35(2)	-13(2)	8(2)	-7(2)
C(44)	31(2)	28(2)	21(2)	-6(2)	5(2)	-7(2)
N(1)	36(2)	29(2)	14(1)	-1(1)	6(1)	-8(1)
N(2)	34(2)	28(2)	16(2)	0(1)	1(1)	-3(1)
O(1)	29(1)	39(2)	20(1)	-2(1)	2(1)	-11(1)
O(2)	42(2)	69(2)	19(2)	-10(1)	10(1)	-15(1)
O(3)	32(1)	42(2)	18(1)	1(1)	9(1)	-10(1)
O(4)	46(2)	82(2)	16(1)	-9(1)	4(1)	-17(2)

Table 5. Hydrogen coordinates [x 10^4] and isotropic displacement parameters [Å^2 x 10^3] for Bhat03.

	x	y	z	U(eq)
H(1A)	1736	4142	5771	49
H(1B)	3449	3584	5778	49
H(1C)	2451	3064	5195	49
H(3)	5068	2907	4579	34
H(4)	6625	3071	3540	39
H(5)	6178	4892	2643	35
H(9)	978	6319	5017	31
H(11A)	299	7498	2864	47
H(11B)	-450	8909	3114	47
H(11C)	-598	7657	3666	47
H(12A)	225	8512	4779	53
H(12B)	540	9741	4269	53
H(12C)	1793	8780	4813	53
H(13)	1809	9579	3074	32
H(16)	3747	5863	1661	37
H(18A)	6598	7989	1710	52
H(18B)	6965	6553	2143	52
H(18C)	7476	7668	2540	52
H(20)	6938	9229	3097	36
H(21A)	5191	10303	4289	51
H(21B)	6765	10549	4033	51
H(22A)	3897	8176	4159	35
H(22B)	3493	9688	3884	35
H(23A)	6306	6872	-212	48

H(23B)	6144	5772	-759	48
H(23C)	7554	6319	-813	48
H(25)	8740	7104	411	35
H(26)	10419	6969	1430	37
H(27)	11012	5164	2338	34
H(31)	6725	3567	-16	28
H(33A)	7384	1425	169	56
H(33B)	8317	178	691	56
H(33C)	9106	1124	182	56
H(34A)	6758	2363	2110	55
H(34B)	6958	906	1899	55
H(34C)	6018	2076	1320	55
H(35)	9565	368	1888	30
H(38)	9175	4142	3330	39
H(40A)	13235	2122	3307	52
H(40B)	12748	3561	2881	52
H(40C)	13948	2488	2489	52
H(42)	14373	859	1936	34
H(43A)	13293	169	561	44
H(43B)	14994	-163	855	44
H(44A)	11288	422	1053	32
H(44B)	10758	1946	830	32

Table 6. Torsion angles [°] for Bhat03.

N(1)-C(2)-C(3)-C(4)	179.7(3)	C(10)-C(13)-C(14)-C(15)	-99.7(3)
C(7)-C(2)-C(3)-C(4)	-1.6(5)	O(1)-C(14)-C(15)-C(17)	120.2(3)
C(2)-C(3)-C(4)-C(5)	-0.5(5)	C(13)-C(14)-C(15)-C(17)	-57.7(4)
C(3)-C(4)-C(5)-C(6)	1.6(6)	O(1)-C(14)-C(15)-C(6)	-118.6(3)
C(4)-C(5)-C(6)-C(7)	-0.6(5)	C(13)-C(14)-C(15)-C(6)	63.6(4)
C(4)-C(5)-C(6)-C(15)	-179.7(3)	O(1)-C(14)-C(15)-C(16)	-2.8(4)
C(5)-C(6)-C(7)-C(2)	-1.4(5)	C(13)-C(14)-C(15)-C(16)	179.3(3)
C(15)-C(6)-C(7)-C(2)	177.6(3)	C(5)-C(6)-C(15)-C(14)	166.3(3)
C(5)-C(6)-C(7)-C(8)	178.7(4)	C(7)-C(6)-C(15)-C(14)	-12.8(5)
C(15)-C(6)-C(7)-C(8)	-2.3(6)	C(5)-C(6)-C(15)-C(17)	-74.5(4)
N(1)-C(2)-C(7)-C(6)	-178.5(3)	C(7)-C(6)-C(15)-C(17)	106.4(4)
C(3)-C(2)-C(7)-C(6)	2.6(5)	C(5)-C(6)-C(15)-C(16)	52.9(4)
N(1)-C(2)-C(7)-C(8)	1.4(4)	C(7)-C(6)-C(15)-C(16)	-126.1(3)
C(3)-C(2)-C(7)-C(8)	-177.5(3)	C(14)-C(15)-C(16)-O(2)	98.9(4)
C(6)-C(7)-C(8)-C(9)	179.1(4)	C(17)-C(15)-C(16)-O(2)	-17.4(6)
C(2)-C(7)-C(8)-C(9)	-0.8(4)	C(6)-C(15)-C(16)-O(2)	-141.2(4)
C(6)-C(7)-C(8)-C(10)	-1.4(7)	C(14)-C(15)-C(17)-C(19)	41.6(4)
C(2)-C(7)-C(8)-C(10)	178.7(3)	C(6)-C(15)-C(17)-C(19)	-81.6(4)
C(7)-C(8)-C(9)-N(1)	-0.1(4)	C(16)-C(15)-C(17)-C(19)	156.1(3)
C(10)-C(8)-C(9)-N(1)	-179.6(3)	C(14)-C(15)-C(17)-C(18)	-142.6(3)
C(9)-C(8)-C(10)-C(11)	-77.5(4)	C(6)-C(15)-C(17)-C(18)	94.3(4)
C(7)-C(8)-C(10)-C(11)	103.0(4)	C(16)-C(15)-C(17)-C(18)	-28.1(5)
C(9)-C(8)-C(10)-C(12)	41.2(5)	C(18)-C(17)-C(19)-C(20)	1.5(6)
C(7)-C(8)-C(10)-C(12)	-138.2(4)	C(15)-C(17)-C(19)-C(20)	177.1(3)
C(9)-C(8)-C(10)-C(13)	159.7(3)	C(18)-C(17)-C(19)-C(22)	-173.4(3)
C(7)-C(8)-C(10)-C(13)	-19.8(5)	C(15)-C(17)-C(19)-C(22)	2.2(5)
C(8)-C(10)-C(13)-C(14)	64.3(4)	C(17)-C(19)-C(20)-C(21)	-175.1(4)
C(11)-C(10)-C(13)-C(14)	-58.2(4)	C(22)-C(19)-C(20)-C(21)	0.0(6)
C(12)-C(10)-C(13)-C(14)	-175.3(3)	C(14)-C(13)-C(22)-C(19)	18.5(4)
C(8)-C(10)-C(13)-C(22)	-62.0(4)	C(10)-C(13)-C(22)-C(19)	143.5(3)
C(11)-C(10)-C(13)-C(22)	175.6(3)	C(17)-C(19)-C(22)-C(13)	-35.2(5)
C(12)-C(10)-C(13)-C(22)	58.4(4)	C(20)-C(19)-C(22)-C(13)	149.6(3)
C(22)-C(13)-C(14)-O(1)	-150.2(3)	N(2)-C(24)-C(25)-C(26)	-179.2(3)
C(10)-C(13)-C(14)-O(1)	82.5(4)	C(29)-C(24)-C(25)-C(26)	-1.3(5)
C(22)-C(13)-C(14)-C(15)	27.6(4)	C(24)-C(25)-C(26)-C(27)	-1.8(5)

C(25)-C(26)-C(27)-C(28)	2.9(5)	C(32)-C(35)-C(36)-C(37)	-99.0(3)
C(26)-C(27)-C(28)-C(29)	-0.8(5)	O(3)-C(36)-C(37)-C(28)	-122.5(3)
C(26)-C(27)-C(28)-C(37)	-179.8(3)	C(35)-C(36)-C(37)-C(28)	60.9(4)
C(27)-C(28)-C(29)-C(24)	-2.2(4)	O(3)-C(36)-C(37)-C(38)	-5.3(4)
C(37)-C(28)-C(29)-C(24)	176.7(3)	C(35)-C(36)-C(37)-C(38)	178.1(3)
C(27)-C(28)-C(29)-C(30)	177.7(4)	O(3)-C(36)-C(37)-C(39)	116.7(3)
C(37)-C(28)-C(29)-C(30)	-3.3(6)	C(35)-C(36)-C(37)-C(39)	-59.9(4)
N(2)-C(24)-C(29)-C(28)	-178.4(3)	C(27)-C(28)-C(37)-C(36)	168.3(3)
C(25)-C(24)-C(29)-C(28)	3.3(5)	C(29)-C(28)-C(37)-C(36)	-10.7(5)
N(2)-C(24)-C(29)-C(30)	1.7(4)	C(27)-C(28)-C(37)-C(38)	53.7(4)
C(25)-C(24)-C(29)-C(30)	-176.6(3)	C(29)-C(28)-C(37)-C(38)	-125.2(3)
C(28)-C(29)-C(30)-C(31)	178.6(4)	C(27)-C(28)-C(37)-C(39)	-73.5(4)
C(24)-C(29)-C(30)-C(31)	-1.4(4)	C(29)-C(28)-C(37)-C(39)	107.6(4)
C(28)-C(29)-C(30)-C(32)	-2.4(6)	C(36)-C(37)-C(38)-O(4)	94.5(4)
C(24)-C(29)-C(30)-C(32)	177.6(3)	C(28)-C(37)-C(38)-O(4)	-143.8(4)
C(29)-C(30)-C(31)-N(2)	0.7(4)	C(39)-C(37)-C(38)-O(4)	-20.2(6)
C(32)-C(30)-C(31)-N(2)	-178.4(3)	C(36)-C(37)-C(39)-C(41)	44.1(4)
C(31)-C(30)-C(32)-C(34)	-77.2(4)	C(28)-C(37)-C(39)-C(41)	-79.7(4)
C(29)-C(30)-C(32)-C(34)	103.9(4)	C(38)-C(37)-C(39)-C(41)	157.7(3)
C(31)-C(30)-C(32)-C(35)	160.0(3)	C(36)-C(37)-C(39)-C(40)	-141.1(3)
C(29)-C(30)-C(32)-C(35)	-18.9(5)	C(28)-C(37)-C(39)-C(40)	95.1(4)
C(31)-C(30)-C(32)-C(33)	41.0(4)	C(38)-C(37)-C(39)-C(40)	-27.5(5)
C(29)-C(30)-C(32)-C(33)	-137.9(4)	C(40)-C(39)-C(41)-C(42)	1.3(6)
C(30)-C(32)-C(35)-C(36)	65.2(4)	C(37)-C(39)-C(41)-C(42)	175.8(3)
C(34)-C(32)-C(35)-C(36)	-57.4(4)	C(40)-C(39)-C(41)-C(44)	-172.4(3)
C(33)-C(32)-C(35)-C(36)	-174.7(3)	C(37)-C(39)-C(41)-C(44)	2.1(5)
C(30)-C(32)-C(35)-C(44)	-60.7(4)	C(39)-C(41)-C(42)-C(43)	-165.5(4)
C(34)-C(32)-C(35)-C(44)	176.6(3)	C(44)-C(41)-C(42)-C(43)	8.5(6)
C(33)-C(32)-C(35)-C(44)	59.3(4)	C(39)-C(41)-C(44)-C(35)	-37.3(5)
C(44)-C(35)-C(36)-O(3)	-148.6(3)	C(42)-C(41)-C(44)-C(35)	148.8(3)
C(32)-C(35)-C(36)-O(3)	84.4(4)	C(36)-C(35)-C(44)-C(41)	20.2(4)
C(44)-C(35)-C(36)-C(37)	28.0(4)	C(32)-C(35)-C(44)-C(41)	145.5(3)
C(8)-C(9)-N(1)-C(2)	1.0(4)	C(30)-C(31)-N(2)-C(24)	0.4(4)
C(8)-C(9)-N(1)-C(1)	-178.0(3)	C(30)-C(31)-N(2)-C(23)	-179.3(3)
C(3)-C(2)-N(1)-C(9)	177.4(3)	C(25)-C(24)-N(2)-C(31)	176.9(3)
C(7)-C(2)-N(1)-C(9)	-1.5(4)	C(29)-C(24)-N(2)-C(31)	-1.3(4)
C(3)-C(2)-N(1)-C(1)	-3.6(5)	C(25)-C(24)-N(2)-C(23)	-3.4(6)
C(7)-C(2)-N(1)-C(1)	177.5(3)	C(29)-C(24)-N(2)-C(23)	178.4(3)

Chloride 16 (CCDC 829858)

Data Collection

An irregular elongate broken fragment (0.48 x 0.12 x 0.12 mm) was selected under a stereo-microscope while immersed in Fluorolube oil to avoid possible reaction with air. The crystal was removed from the oil using a tapered glass fiber that also served to hold the crystal for data collection. The crystal was mounted and centered on a Bruker SMART APEX system at 100 K. Rotation and still images showed the diffractions to be sharp. Frames separated in reciprocal space were obtained and provided an orientation matrix and initial cell parameters. Final cell parameters were obtained from the full data set. The intensity was very low and seldom were diffractions observed beyond 2-theta of 25 degrees.

A "full sphere" data set was obtained which samples approximately all of reciprocal space to a resolution of 0.90 Å using 0.3^{o} steps in ω using 20 second integration times for each frame. Data collection was made at 100 K. Integration of intensities and refinement of cell parameters were done using SAINT [1]. Absorption corrections were applied using SADABS [1] based on redundant diffractions.

Structure solution and refinement

The space group was determined as $P2_1$ based on systematic absences and intensity statistics. Direct methods were used to locate the Cl atom and most C atoms from the E-map. Repeated difference Fourier maps allowed recognition of all expected C, N, O and Cl atoms. Following anisotropic refinement of all non-H atoms, ideal H-atom positions were calculated. Final refinement was anisotropic for all non-H atoms, and isotropic-riding for H atoms. No anomalous bond lengths or thermal parameters were noted. All ORTEP diagrams have been drawn with 50% probability ellipsoids. The relatively high R is most certainly due to the weakly diffracting crystal.

Equations of interest:

$R_{int} = \Sigma \mid F_o^2 - \langle F_o^2 \rangle \mid / \Sigma \mid F_o^2 \mid$ $R1 = \Sigma \mid \mid F_o \mid - \mid F_c \mid \mid / \Sigma \mid F_o \mid$

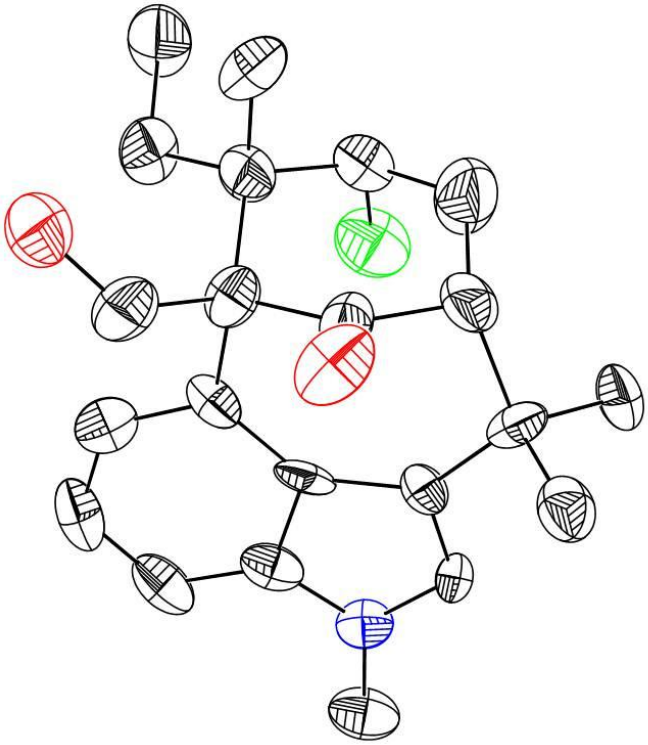
$wR2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2)^2]]^{1/2}$ $GooF = S = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)^{1/2}$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$; n = number of independent reflections;

q, a, b, P as defined in [1] p = number of parameters refined.

References

[1] All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).



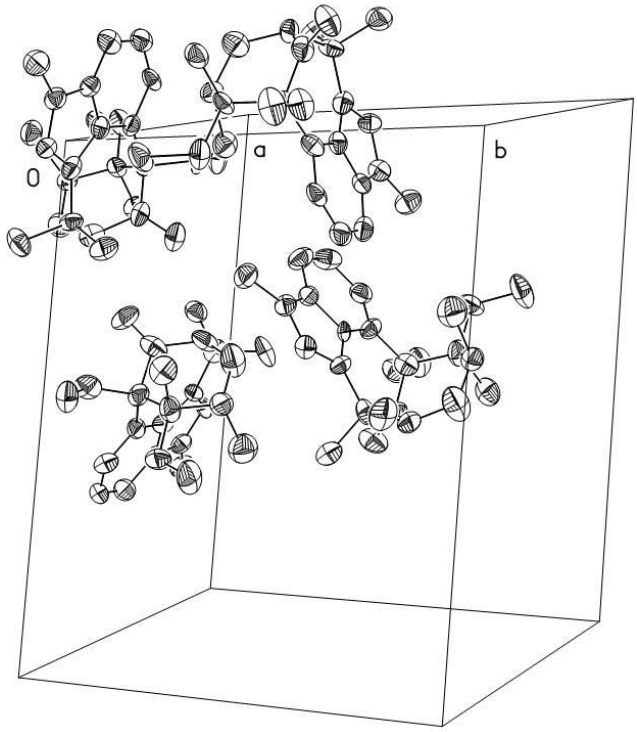


Table 1. Crystal and structure refinement for Bhat07.

Identification Code	Bhat07	
Empirical formula	$2C_{22}H_{26}ClNO_2$	
Formula weight	743.78	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space Group	$P2_1$	
Unit cell dimensions	a = 9.746(3) Å	α = 90.0 °
	b = 13.298(4) Å	β = 106.377(5) °
	c = 15.127(5) Å	γ = 90.0 °
Volume	1880.9(11) $Å^3$	
Z	2	
Density (calculated)	1.313 Mg/m^3	
Absorption coefficient	0.219 mm^{-1}	
F(000)	792	
Crystal size, color, habit	0.48 x 0.12 x 0.12mm, clear, long fragment	
Theta range for data collection	1.40 – 23.60 °	
Index ranges	$-10 \le h \le 10$, $-14 \le k \le 14$, $-16 \le l \le 16$	
Reflections collected	15,755	
Independent reflections	5,546 (R_{int} = 0.0554)	
Reflections with $I > 4\sigma(F_o)$	3,028	
Flack parameter	0.18(13)	
Absorption correction	SADABS based on redundant diffractions	
Max. and min. transmission	1.0, 0.451	
Refinement method	Full-matrix least squares on F^2	
Weighting scheme	$w = q\,[\sigma^2(F_o^2) + (aP)^2 + bP]^{-1}$ where:	
	$P = (F_o^2 + 2F_c^2)/3$, a = 0.0634, b = 0.0, q =1	
Data / restraints / parameters	5546 / 1 / 480	
Goodness-of-fit on F^2	0.925	
Final R indices [I > 2 sigma(I)]	R1 = 0.0771, wR2 = 0.1571	
R indices (all data)	R1 = 0.1319, wR2 = 0.1771	
Largest diff. peak and hole	0.287, -0.503 $eÅ^{-3}$	

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for Bhat07. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
C(1)	991(9)	4398(6)	2643(5)	55(3)	
C(2)	2206(8)	5443(6)	4047(5)	34(2)	
C(3)	3543(8)	5756(5)	4514(5)	33(2)	
C(4)	4437(9)	5412(5)	3994(5)	36(2)	
C(5)	3562(10)	4837(6)	3243(5)	40(2)	
C(6)	4096(11)	4339(6)	2592(6)	53(3)	
C(7)	5567(10)	4387(6)	2729(6)	52(2)	
C(8)	6421(9)	4949(6)	3459(5)	44(2)	
C(9)	5958(9)	5451(6)	4095(5)	38(2)	
C(10)	7043(8)	5977(6)	4887(5)	41(2)	
C(11)	8218(10)	5217(7)	5157(6)	53(3)	
C(12)	6467(9)	5986(8)	5751(6)	48(2)	
C(13)	5351(9)	6791(6)	5761(6)	51(2)	
C(14)	3772(8)	6345(6)	5390(6)	42(2)	
C(15)	2675(9)	7197(7)	5271(6)	62(3)	
C(16)	3552(9)	5637(6)	6145(5)	48(2)	
C(17)	5717(11)	7738(9)	5437(7)	77(3)	
C(18)	6339(9)	7820(7)	4675(6)	48(2)	
C(19)	7496(9)	7059(6)	4698(6)	48(2)	
C(20)	8005(10)	7107(6)	3826(5)	52(2)	
C(21)	8461(11)	8148(6)	3598(7)	75(3)	
C(22)	8726(8)	7398(7)	5573(6)	57(3)	
C(23)	14653(8)	6650(6)	11587(5)	50(2)	
C(24)	13081(8)	6211(5)	10012(5)	36(2)	
C(25)	11660(8)	6121(6)	9564(5)	37(2)	
C(26)	10929(8)	6378(5)	10259(5)	33(2)	
C(27)	11968(8)	6579(6)	11062(5)	31(2)	
C(28)	11683(9)	6821(5)	11863(6)	43(2)	
C(29)	10242(10)	6890(6)	11840(6)	44(2)	
C(30)	9200(9)	6709(5)	11071(6)	37(2)	
C(31)	9459(8)	6418(5)	10222(5)	34(2)	
C(32)	8205(8)	6203(6)	9378(5)	32(2)	
C(33)	7128(9)	7090(6)	9410(6)	45(2)	
C(34)	7542(8)	5131(6)	9340(5)	34(2)	
C(35)	7070(9)	4894(6)	10196(6)	48(2)	
C(36)	6318(10)	3872(6)	10177(6)	58(3)	
C(37)	6188(8)	5055(6)	8460(5)	45(2)	
C(38)	8550(8)	4350(6)	9161(5)	38(2)	
C(39)	9114(9)	4565(7)	8337(6)	51(2)	
C(40)	9493(8)	5648(6)	8161(5)	40(2)	
C(41)	11145(8)	5957(6)	8536(5)	36(2)	
C(42)	12034(8)	5129(7)	8236(6)	56(3)	
C(43)	11339(8)	6953(6)	8057(5)	51(2)	
C(44)	8585(9)	6395(6)	8487(6)	40(2)	
Cl(1)	4875(3)	7798(2)	3644(2)	79(1)	
Cl(2)	10030(2)	4075(2)	10180(2)	49(1)	
N(1)	2196(7)	4882(5)	3291(4)	37(2)	
N(2)	13291(6)	6442(5)	10905(4)	37(2)	
O(1)	9461(7)	5344(5)	5123(4)	64(2)	
O(2)	6868(6)	5358(5)	6346(4)	58(2)	
O(3)	5907(6)	6995(4)	9383(4)	56(2)	
O(4)	8120(6)	7118(4)	8014(4)	50(2)	

Table 3. Bond lengths [Å] and angles [°] for Bhat07.

C(1)-N(1)	1.451(9)	C(23)-N(2)	1.459(9)
C(2)-C(3)	1.360(10)	C(24)-N(2)	1.345(9)
C(2)-N(1)	1.363(9)	C(24)-C(25)	1.365(10)
C(3)-C(4)	1.404(10)	C(25)-C(26)	1.467(10)
C(3)-C(14)	1.501(10)	C(25)-C(41)	1.509(10)
C(4)-C(5)	1.434(10)	C(26)-C(27)	1.371(10)
C(4)-C(9)	1.448(11)	C(26)-C(31)	1.418(10)
C(5)-N(1)	1.355(10)	C(27)-C(28)	1.355(10)
C(5)-C(6)	1.404(11)	C(27)-N(2)	1.387(9)
C(6)-C(7)	1.391(11)	C(28)-C(29)	1.399(11)
C(7)-C(8)	1.397(11)	C(29)-C(30)	1.333(10)
C(8)-C(9)	1.350(10)	C(30)-C(31)	1.431(10)
C(9)-C(10)	1.526(10)	C(31)-C(32)	1.524(10)
C(10)-C(11)	1.497(11)	C(32)-C(44)	1.517(10)
C(10)-C(19)	1.554(11)	C(32)-C(34)	1.560(10)
C(10)-C(12)	1.560(11)	C(32)-C(33)	1.589(10)
C(11)-O(1)	1.238(9)	C(33)-O(3)	1.185(8)
C(12)-O(2)	1.210(10)	C(34)-C(38)	1.505(10)
C(12)-C(13)	1.529(12)	C(34)-C(35)	1.524(10)
C(13)-C(17)	1.433(13)	C(34)-C(37)	1.591(10)
C(13)-C(14)	1.596(11)	C(35)-C(36)	1.541(11)
C(14)-C(15)	1.533(11)	C(38)-C(39)	1.525(10)
C(14)-C(16)	1.542(10)	C(38)-Cl(2)	1.826(7)
C(17)-C(18)	1.451(11)	C(39)-C(40)	1.529(11)
C(18)-C(19)	1.509(11)	C(40)-C(44)	1.503(11)
C(18)-Cl(1)	1.793(8)	C(40)-C(41)	1.602(10)
C(19)-C(20)	1.536(11)	C(41)-C(42)	1.546(10)
C(19)-C(22)	1.580(11)	C(41)-C(43)	1.547(11)
C(20)-C(21)	1.522(11)	C(44)-O(4)	1.208(9)

C(3)-C(2)-N(1)	112.5(7)	C(19)-C(18)-Cl(1)	114.5(6)
C(2)-C(3)-C(4)	105.4(7)	C(18)-C(19)-C(20)	111.9(7)
C(2)-C(3)-C(14)	120.0(7)	C(18)-C(19)-C(10)	112.1(7)
C(4)-C(3)-C(14)	134.6(7)	C(20)-C(19)-C(10)	112.0(7)
C(3)-C(4)-C(5)	107.0(7)	C(18)-C(19)-C(22)	102.3(7)
C(3)-C(4)-C(9)	135.1(7)	C(20)-C(19)-C(22)	110.8(7)
C(5)-C(4)-C(9)	117.7(8)	C(10)-C(19)-C(22)	107.3(7)
N(1)-C(5)-C(6)	128.5(8)	C(21)-C(20)-C(19)	114.7(8)
N(1)-C(5)-C(4)	108.0(7)	N(2)-C(24)-C(25)	111.3(7)
C(6)-C(5)-C(4)	123.6(9)	C(24)-C(25)-C(26)	104.8(7)
C(7)-C(6)-C(5)	116.4(8)	C(24)-C(25)-C(41)	121.3(7)
C(6)-C(7)-C(8)	120.1(8)	C(26)-C(25)-C(41)	133.2(7)
C(9)-C(8)-C(7)	125.7(8)	C(27)-C(26)-C(31)	120.9(7)
C(8)-C(9)-C(4)	116.5(7)	C(27)-C(26)-C(25)	107.1(7)
C(8)-C(9)-C(10)	119.3(8)	C(31)-C(26)-C(25)	132.0(7)
C(4)-C(9)-C(10)	124.1(7)	C(28)-C(27)-C(26)	123.5(8)
C(11)-C(10)-C(9)	102.2(7)	C(28)-C(27)-N(2)	128.2(7)
C(11)-C(10)-C(19)	116.1(7)	C(26)-C(27)-N(2)	108.2(7)
C(9)-C(10)-C(19)	116.7(7)	C(27)-C(28)-C(29)	116.8(8)
C(11)-C(10)-C(12)	101.8(7)	C(30)-C(29)-C(28)	121.5(8)
C(9)-C(10)-C(12)	109.1(7)	C(29)-C(30)-C(31)	123.3(8)
C(19)-C(10)-C(12)	109.6(7)	C(26)-C(31)-C(30)	114.0(7)
O(1)-C(11)-C(10)	125.3(9)	C(26)-C(31)-C(32)	126.1(7)
O(2)-C(12)-C(13)	123.8(8)	C(30)-C(31)-C(32)	119.9(7)
O(2)-C(12)-C(10)	120.1(8)	C(44)-C(32)-C(31)	111.9(6)
C(13)-C(12)-C(10)	116.0(8)	C(44)-C(32)-C(34)	108.8(6)
C(17)-C(13)-C(12)	111.5(8)	C(31)-C(32)-C(34)	115.4(6)
C(17)-C(13)-C(14)	121.0(8)	C(44)-C(32)-C(33)	103.4(6)
C(12)-C(13)-C(14)	110.8(7)	C(31)-C(32)-C(33)	102.5(6)
C(3)-C(14)-C(15)	110.9(7)	C(34)-C(32)-C(33)	114.2(6)
C(3)-C(14)-C(16)	108.6(6)	O(3)-C(33)-C(32)	125.8(8)
C(15)-C(14)-C(16)	107.4(7)	C(38)-C(34)-C(35)	112.2(6)
C(3)-C(14)-C(13)	113.2(6)	C(38)-C(34)-C(32)	110.7(6)
C(15)-C(14)-C(13)	109.7(7)	C(35)-C(34)-C(32)	112.2(6)
C(16)-C(14)-C(13)	106.7(6)	C(38)-C(34)-C(37)	103.6(6)
C(13)-C(17)-C(18)	122.5(10)	C(35)-C(34)-C(37)	108.7(6)
C(17)-C(18)-C(19)	114.1(8)	C(32)-C(34)-C(37)	109.1(6)
C(17)-C(18)-Cl(1)	106.4(7)	C(34)-C(35)-C(36)	115.0(7)

C(34)-C(38)-C(39)	114.9(6)	C(42)-C(41)-C(40)	107.6(6)
C(34)-C(38)-Cl(2)	112.8(5)	C(43)-C(41)-C(40)	107.2(6)
C(39)-C(38)-Cl(2)	110.2(6)	O(4)-C(44)-C(40)	119.3(8)
C(38)-C(39)-C(40)	118.7(7)	O(4)-C(44)-C(32)	121.0(8)
C(44)-C(40)-C(39)	111.8(7)	C(40)-C(44)-C(32)	119.7(7)
C(44)-C(40)-C(41)	109.9(6)	C(5)-N(1)-C(2)	107.1(6)
C(39)-C(40)-C(41)	116.6(7)	C(5)-N(1)-C(1)	124.4(7)
C(25)-C(41)-C(42)	110.8(6)	C(2)-N(1)-C(1)	128.5(7)
C(25)-C(41)-C(43)	108.1(6)	C(24)-N(2)-C(27)	108.5(6)
C(42)-C(41)-C(43)	108.6(6)	C(24)-N(2)-C(23)	127.1(7)
C(25)-C(41)-C(40)	114.3(6)	C(27)-N(2)-C(23)	123.9(7)

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for Bhat07. The anisotropic displacement factor exponent takes the form:
$-2\pi^2[h^2a^{*2}U_{11}+ ... + 2hka^*b^*U_{12}]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	61(6)	49(6)	44(6)	-1(5)	-3(5)	-12(5)
C(2)	27(5)	35(5)	46(5)	-4(4)	19(4)	3(4)
C(3)	38(5)	32(5)	27(5)	-6(4)	6(4)	1(4)
C(4)	59(6)	15(4)	34(5)	0(4)	11(5)	-13(4)
C(5)	55(6)	29(5)	30(5)	5(4)	2(4)	-5(4)
C(6)	75(7)	38(6)	40(5)	-10(4)	7(5)	4(5)
C(7)	74(7)	47(6)	40(5)	1(5)	23(5)	24(5)
C(8)	44(6)	50(6)	36(5)	9(5)	8(4)	-4(5)
C(9)	46(6)	28(5)	34(5)	-6(4)	3(4)	5(4)
C(10)	29(5)	50(6)	41(5)	7(4)	6(4)	7(4)
C(11)	55(7)	58(6)	55(6)	6(5)	29(5)	-9(5)
C(12)	32(5)	72(7)	43(6)	-23(5)	12(5)	-10(5)
C(13)	70(7)	47(6)	44(6)	-17(5)	27(5)	-11(5)
C(14)	32(5)	41(6)	54(6)	7(4)	16(4)	-11(4)
C(15)	60(7)	48(6)	89(7)	-23(5)	36(6)	9(5)
C(16)	46(6)	53(6)	42(5)	-11(5)	9(4)	2(5)
C(17)	85(8)	55(7)	110(9)	8(7)	57(7)	8(6)
C(18)	56(6)	41(5)	44(6)	-3(5)	12(5)	-1(5)
C(19)	54(6)	33(5)	62(6)	-1(5)	23(5)	2(5)
C(20)	69(6)	54(6)	43(5)	10(5)	31(5)	2(5)
C(21)	99(9)	38(6)	111(9)	19(6)	64(7)	5(6)
C(22)	38(6)	66(7)	69(7)	7(5)	19(5)	-7(5)
C(23)	38(5)	61(6)	48(6)	-6(5)	6(5)	1(5)
C(24)	35(5)	40(5)	30(5)	3(4)	7(4)	6(4)
C(25)	31(5)	31(5)	40(5)	3(4)	-2(4)	2(4)
C(26)	35(5)	24(5)	40(5)	3(4)	10(4)	0(4)
C(27)	31(5)	30(4)	32(5)	7(4)	10(4)	-2(4)
C(28)	50(6)	31(5)	48(6)	-6(4)	16(5)	-9(4)
C(29)	64(7)	28(5)	42(6)	1(4)	21(5)	-1(5)
C(30)	60(6)	20(4)	43(6)	1(4)	34(5)	1(4)
C(31)	35(5)	32(5)	37(5)	12(4)	16(4)	10(4)
C(32)	32(5)	32(5)	32(5)	2(4)	8(4)	5(4)
C(33)	32(5)	35(5)	67(6)	15(5)	10(5)	15(4)
C(34)	45(5)	29(5)	38(5)	6(4)	27(4)	0(4)
C(35)	45(6)	37(5)	67(6)	-11(5)	24(5)	-11(5)
C(36)	98(8)	27(5)	65(6)	1(4)	48(6)	-5(5)
C(37)	38(5)	53(6)	45(5)	8(5)	16(4)	-8(5)
C(38)	40(5)	36(5)	32(5)	-1(4)	3(4)	2(4)
C(39)	54(6)	52(6)	52(6)	4(5)	21(5)	4(5)
C(40)	32(5)	43(5)	42(5)	1(4)	6(4)	-14(4)
C(41)	27(5)	57(6)	25(5)	8(4)	9(4)	2(4)
C(42)	30(5)	72(7)	60(6)	-31(5)	1(4)	8(5)
C(43)	43(6)	63(6)	40(5)	6(5)	3(4)	-5(5)
C(44)	39(5)	30(5)	48(6)	7(4)	6(4)	4(4)
Cl(1)	92(2)	66(2)	75(2)	1(2)	19(2)	-2(2)
Cl(2)	54(1)	37(1)	53(1)	2(1)	9(1)	4(1)
N(1)	32(4)	41(4)	33(4)	3(3)	-2(4)	-2(4)
N(2)	30(4)	43(4)	37(4)	5(3)	8(3)	3(3)

O(1)	62(5)	47(4)	83(5)	6(4)	23(4)	12(4)
O(2)	39(4)	82(5)	46(4)	23(4)	2(3)	5(3)
O(3)	29(4)	48(4)	94(5)	10(3)	25(3)	11(3)
O(4)	40(4)	43(4)	70(4)	18(3)	23(3)	9(3)

Table 5. Hydrogen coordinates [x 10^4] and isotropic displacement parameters [Å^2 x 10^3] for Bhat07.

	x	y	z	U(eq)
H(1A)	776	4746	2048	83
H(1B)	155	4426	2881	83
H(1C)	1229	3694	2562	83
H(2)	1373	5597	4228	41
H(6)	3486	3988	2085	64
H(7)	5992	4038	2325	63
H(8)	7415	4979	3512	53
H(11)	7993	4584	5373	64
H(13)	5471	6908	6431	62
H(15A)	2765	7648	4778	94
H(15B)	2852	7575	5849	94
H(15C)	1709	6912	5111	94
H(16A)	2633	5289	5919	71
H(16B)	3556	6030	6694	71
H(16C)	4327	5141	6304	71
H(17)	5545	8336	5734	93
H(18)	6788	8501	4714	57
H(20A)	8822	6641	3901	63
H(20B)	7223	6866	3297	63
H(21A)	7773	8649	3685	113
H(21B)	8491	8161	2956	113
H(21C)	9412	8303	4006	113
H(22A)	8830	8131	5567	85
H(22B)	9628	7080	5561	85
H(22C)	8482	7194	6132	85
H(23A)	15438	6407	11357	75
H(23B)	14682	6305	12164	75
H(23C)	14753	7376	11696	75
H(24)	13827	6121	9728	43
H(28)	12427	6938	12413	51
H(29)	10000	7069	12385	52
H(30)	8237	6778	11087	44
H(33)	7495	7756	9455	55
H(35A)	6413	5431	10278	57
H(35B)	7922	4910	10739	57
H(36A)	5462	3849	9648	88
H(36B)	6042	3786	10748	88
H(36C)	6971	3330	10123	88
H(37A)	6441	5307	7918	67
H(37B)	5405	5459	8561	67
H(37C)	5884	4351	8359	67
H(38)	7983	3714	9011	45
H(39A)	8389	4326	7778	62
H(39B)	9981	4150	8406	62
H(40)	9236	5725	7476	48
H(42A)	13019	5363	8337	85
H(42B)	11614	4980	7581	85
H(42C)	12030	4520	8599	85
H(43A)	10888	7503	8304	76
H(43B)	10890	6893	7392	76
H(43C)	12361	7093	8170	76
H(1)	9710	4853	4855	96

Table 6. Torsion angles [°] for Bhat07.

N(1)-C(2)-C(3)-C(4)	3.0(9)	C(11)-C(10)-C(19)-C(22)	-53.7(9)
N(1)-C(2)-C(3)-C(14)	-177.3(7)	C(9)-C(10)-C(19)-C(22)	-174.4(7)
C(2)-C(3)-C(4)-C(5)	-3.5(8)	C(12)-C(10)-C(19)-C(22)	61.0(8)
C(14)-C(3)-C(4)-C(5)	176.9(8)	C(18)-C(19)-C(20)-C(21)	53.0(11)
C(2)-C(3)-C(4)-C(9)	-178.0(8)	C(10)-C(19)-C(20)-C(21)	179.8(8)
C(14)-C(3)-C(4)-C(9)	2.4(15)	C(22)-C(19)-C(20)-C(21)	-60.4(10)
C(3)-C(4)-C(5)-N(1)	2.9(8)	N(2)-C(24)-C(25)-C(26)	3.1(9)
C(9)-C(4)-C(5)-N(1)	178.5(6)	N(2)-C(24)-C(25)-C(41)	174.4(7)
C(3)-C(4)-C(5)-C(6)	-177.5(7)	C(24)-C(25)-C(26)-C(27)	-1.2(8)
C(9)-C(4)-C(5)-C(6)	-1.9(11)	C(41)-C(25)-C(26)-C(27)	-170.9(8)
N(1)-C(5)-C(6)-C(7)	-177.7(8)	C(24)-C(25)-C(26)-C(31)	-179.5(8)
C(4)-C(5)-C(6)-C(7)	2.8(11)	C(41)-C(25)-C(26)-C(31)	10.8(15)
C(5)-C(6)-C(7)-C(8)	-2.9(12)	C(31)-C(26)-C(27)-C(28)	0.2(12)
C(6)-C(7)-C(8)-C(9)	2.2(13)	C(25)-C(26)-C(27)-C(28)	-178.3(7)
C(7)-C(8)-C(9)-C(4)	-1.1(12)	C(31)-C(26)-C(27)-N(2)	177.4(7)
C(7)-C(8)-C(9)-C(10)	176.3(7)	C(25)-C(26)-C(27)-N(2)	-1.1(8)
C(3)-C(4)-C(9)-C(8)	175.0(8)	C(26)-C(27)-C(28)-C(29)	-1.8(12)
C(5)-C(4)-C(9)-C(8)	0.9(10)	N(2)-C(27)-C(28)-C(29)	-178.4(7)
C(3)-C(4)-C(9)-C(10)	-2.3(14)	C(27)-C(28)-C(29)-C(30)	1.1(12)
C(5)-C(4)-C(9)-C(10)	-176.3(7)	C(28)-C(29)-C(30)-C(31)	1.1(12)
C(8)-C(9)-C(10)-C(11)	-42.5(9)	C(27)-C(26)-C(31)-C(30)	1.9(10)
C(4)-C(9)-C(10)-C(11)	134.6(8)	C(25)-C(26)-C(31)-C(30)	179.9(8)
C(8)-C(9)-C(10)-C(19)	85.3(9)	C(27)-C(26)-C(31)-C(32)	-179.4(7)
C(4)-C(9)-C(10)-C(19)	-97.5(9)	C(25)-C(26)-C(31)-C(32)	-1.3(13)
C(8)-C(9)-C(10)-C(12)	-149.8(8)	C(29)-C(30)-C(31)-C(26)	-2.6(11)
C(4)-C(9)-C(10)-C(12)	27.4(10)	C(29)-C(30)-C(31)-C(32)	178.6(7)
C(9)-C(10)-C(11)-O(1)	118.2(9)	C(26)-C(31)-C(32)-C(44)	-25.8(10)
C(19)-C(10)-C(11)-O(1)	-10.0(12)	C(30)-C(31)-C(32)-C(44)	152.8(7)
C(12)-C(10)-C(11)-O(1)	-129.0(9)	C(26)-C(31)-C(32)-C(34)	99.3(8)
C(11)-C(10)-C(12)-O(2)	-9.8(10)	C(30)-C(31)-C(32)-C(34)	-82.0(8)
C(9)-C(10)-C(12)-O(2)	97.7(9)	C(26)-C(31)-C(32)-C(33)	-136.0(8)
C(19)-C(10)-C(12)-O(2)	-133.3(8)	C(30)-C(31)-C(32)-C(33)	42.7(9)
C(11)-C(10)-C(12)-C(13)	172.8(7)	C(44)-C(32)-C(33)-O(3)	116.0(9)
C(9)-C(10)-C(12)-C(13)	-79.7(9)	C(31)-C(32)-C(33)-O(3)	-127.6(9)
C(19)-C(10)-C(12)-C(13)	49.3(9)	C(34)-C(32)-C(33)-O(3)	-2.1(12)
O(2)-C(12)-C(13)-C(17)	140.8(10)	C(44)-C(32)-C(34)-C(38)	56.2(8)
C(10)-C(12)-C(13)-C(17)	-41.9(10)	C(31)-C(32)-C(34)-C(38)	-70.5(8)
O(2)-C(12)-C(13)-C(14)	-81.4(10)	C(33)-C(32)-C(34)-C(38)	171.1(6)
C(10)-C(12)-C(13)-C(14)	95.9(8)	C(44)-C(32)-C(34)-C(35)	-177.6(7)
C(2)-C(3)-C(14)-C(15)	-47.4(10)	C(31)-C(32)-C(34)-C(35)	55.7(9)
C(4)-C(3)-C(14)-C(15)	132.2(9)	C(33)-C(32)-C(34)-C(35)	-62.7(8)
C(2)-C(3)-C(14)-C(16)	70.4(9)	C(44)-C(32)-C(34)-C(37)	-57.2(8)
C(4)-C(3)-C(14)-C(16)	-110.0(10)	C(31)-C(32)-C(34)-C(37)	176.1(6)
C(2)-C(3)-C(14)-C(13)	-171.2(7)	C(33)-C(32)-C(34)-C(37)	57.7(8)
C(4)-C(3)-C(14)-C(13)	8.3(12)	C(38)-C(34)-C(35)-C(36)	-58.3(9)
C(17)-C(13)-C(14)-C(3)	86.0(10)	C(32)-C(34)-C(35)-C(36)	176.3(7)
C(12)-C(13)-C(14)-C(3)	-47.2(9)	C(37)-C(34)-C(35)-C(36)	55.7(9)
C(17)-C(13)-C(14)-C(15)	-38.5(11)	C(35)-C(34)-C(38)-C(39)	-177.5(7)
C(12)-C(13)-C(14)-C(15)	-171.7(7)	C(32)-C(34)-C(38)-C(39)	-51.3(8)
C(17)-C(13)-C(14)-C(16)	-154.6(8)	C(37)-C(34)-C(38)-C(39)	65.5(8)
C(12)-C(13)-C(14)-C(16)	72.2(8)	C(35)-C(34)-C(38)-Cl(2)	-50.0(8)
C(12)-C(13)-C(17)-C(18)	39.0(13)	C(32)-C(34)-C(38)-Cl(2)	76.2(7)
C(14)-C(13)-C(17)-C(18)	-93.9(11)	C(37)-C(34)-C(38)-Cl(2)	-167.0(5)
C(13)-C(17)-C(18)-C(19)	-43.1(13)	C(34)-C(38)-C(39)-C(40)	38.9(10)
C(13)-C(17)-C(18)-Cl(1)	84.1(10)	Cl(2)-C(38)-C(39)-C(40)	-89.8(8)
C(17)-C(18)-C(19)-C(20)	174.0(8)	C(38)-C(39)-C(40)-C(44)	-30.1(10)
Cl(1)-C(18)-C(19)-C(20)	51.1(9)	C(38)-C(39)-C(40)-C(41)	97.4(9)
C(17)-C(18)-C(19)-C(10)	47.3(11)	C(24)-C(25)-C(41)-C(42)	46.5(10)
Cl(1)-C(18)-C(19)-C(10)	-75.6(8)	C(26)-C(25)-C(41)-C(42)	-145.2(8)
C(17)-C(18)-C(19)-C(22)	-67.4(9)	C(24)-C(25)-C(41)-C(43)	-72.5(9)
Cl(1)-C(18)-C(19)-C(22)	169.7(6)	C(26)-C(25)-C(41)-C(43)	95.9(10)
C(11)-C(10)-C(19)-C(18)	-165.2(7)	C(24)-C(25)-C(41)-C(40)	168.2(7)
C(9)-C(10)-C(19)-C(18)	74.1(9)	C(26)-C(25)-C(41)-C(40)	-23.4(12)
C(12)-C(10)-C(19)-C(18)	-50.6(9)	C(44)-C(40)-C(41)-C(25)	52.8(9)
C(11)-C(10)-C(19)-C(20)	68.2(10)	C(39)-C(40)-C(41)-C(25)	-75.6(9)
C(9)-C(10)-C(19)-C(20)	-52.6(10)	C(44)-C(40)-C(41)-C(42)	176.3(7)
C(12)-C(10)-C(19)-C(20)	-177.2(7)	C(39)-C(40)-C(41)-C(42)	47.8(9)

C(44)-C(40)-C(41)-C(43)	-67.0(8)	C(6)-C(5)-N(1)-C(2)	179.3(8)
C(39)-C(40)-C(41)-C(43)	164.5(7)	C(4)-C(5)-N(1)-C(2)	-1.1(8)
C(39)-C(40)-C(44)-O(4)	-138.1(8)	C(6)-C(5)-N(1)-C(1)	0.0(12)
C(41)-C(40)-C(44)-O(4)	90.8(9)	C(4)-C(5)-N(1)-C(1)	179.5(6)
C(39)-C(40)-C(44)-C(32)	38.8(10)	C(3)-C(2)-N(1)-C(5)	-1.2(9)
C(41)-C(40)-C(44)-C(32)	-92.3(8)	C(3)-C(2)-N(1)-C(1)	178.1(7)
C(31)-C(32)-C(44)-O(4)	-107.0(8)	C(25)-C(24)-N(2)-C(27)	-3.9(9)
C(34)-C(32)-C(44)-O(4)	124.3(8)	C(25)-C(24)-N(2)-C(23)	-176.5(7)
C(33)-C(32)-C(44)-O(4)	2.6(10)	C(28)-C(27)-N(2)-C(24)	-179.9(8)
C(31)-C(32)-C(44)-C(40)	76.1(9)	C(26)-C(27)-N(2)-C(24)	3.0(8)
C(34)-C(32)-C(44)-C(40)	-52.6(9)	C(28)-C(27)-N(2)-C(23)	-7.1(12)
C(33)-C(32)-C(44)-C(40)	-174.3(7)	C(26)-C(27)-N(2)-C(23)	175.9(7)