Supporting Information

Stereoselective Formation of Carbon-Carbon Bonds via S_N2-Displacement: Synthesis of Substituted Cycloalkyl[*b*]indoles.

Michael C. Hillier, Jean-François Marcoux, Dalian Zhao, Edward J. J. Grabowski,*

Arlene E. McKeown, Richard D. Tillyer

Department of Process Research, Merck & Co., P. O. Box 2000, Rahway, NJ 07065

Contents:	Page:
General Methods	S2
¹ H NMR of 7a,b,d,e	S3-S6
¹ H NMR of 10a,d,e	S7-S9
¹ H NMR of 11	S10
¹ H NMR of 12	S11
¹ H NMR of 13a-13f	S12-S17
¹ H NMR of 14a-14f	S18-S23
¹ H NMR of 15	S24
¹ H NMR of 16a-16f	S25-S30
¹ H NMR of 17a-17f	S31-S36
X-Ray Structural Data for 14a	S37-S46

General Methods. Unless otherwise stated, solvents and reagents were reagent grade and used without further purification. Melting points were determined on a Büchi B-545 melting point apparatus and are uncorrected. Infrared (IR) spectra were recorded using PTFE or NaCl plate IR cards and evaporating from the solvent indicated, and are reported in wavenumbers (cm^{-1}). ^1H (400 MHz) and ^{13}C (100 MHz) NMR spectra were obtained using a 400 MHz instrument, and chemical shifts are reported in parts per million (ppm). Coupling constants are reported in hertz (Hz). Spectral splitting patterns are designated as: app, apparent; s, singlet; br, broad; d, doublet; t, triplet; q, quartet; m, multiplet; and comp, complex multiplet. Hi-resolution mass spectra was obtained using an electrospray mass spectrometer. Determination of enantiomeric ratios for chiral products was carried out using a supercritical fluid chromatography (SFC) apparatus with the indicated column under the conditions and using the columns indicated.

710568-153
isomer mixture
DMSO-d6
nmr 400a h-1

71081-75
cyclopentyl
keto hydrazone
DMSO-d6
nmr 400b h-1

Current Data Parameters
NAME 71081-075
EXTNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050511
Time 17.00
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 16
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 15384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
P1P 13.000 ppm
P1 5198.31 Hz
P2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.33125 ppm/cm
HZCM 132.45694 Hz/cm

71262-003
crude
mono fluoro
hydrazone
DMSO-d6
nmr 400c h1

71262-015
crude
difluoro
hydrazine
DMSO-d6
H1-NMR

Current Data Parameters
NAME 71262-015
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050518
Time 9.01
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6578.947 Hz
F2 160.115 MHz
AQ 2.4904180 sec
RG 57
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.00 usec
PL1 4.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8702027 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

ID NMR plot parameters
CX 40.00 cm
P1H 13.800 ppm
F1 5518.21 Hz
F2H -0.250 ppm
F2 -99.97 Hz
PPM 0.35125 ppm/cm
HZ 140.45435 Hz/cm

ketoindole
DMSO-d6
nmr400b h-1

71262-017
ketocindole
DMSO-d6
nmr400c h1

71081-039
purified
CDCl₃
nmr 400b h-1

nmr400b h-1

Current Data Parameters
NAME 75282-92F3
EXNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20020814

Time 17.02
INSTRUM Spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 16
DS 2
SWH 6578.947 Hz
FIDRES 0.700774 Hz
AQ 2.4904180 sec
RG 161.3
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 10.50 usec
PL 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters

SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

ID NMR plot parameters

CX 40.00 cm
FLP 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPKM 0.25000 ppm/cm
HZCM 99.96751 Hz/cm

nmr400b h-1

Current Data Parameters
NAME 75282-103Ae
EXTNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20020905
Time_ 11.58
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 406.4
IN 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====

NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters

SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.09 Hz
GB 0
PC 1.00

1D NMR plot parameters

CA 40.00 cm
F1P 13.500 ppm
F1 5398.25 Hz
F2P 0.500 ppm
F2 199.93 Hz
PRGCM 0.32500 ppm/cm
HZCM 129.95776 Hz/cm

75282-113-2
98 wt.8.
CDCl₃
nmr 400a h-1

Current Data Parameters
NAME 75282-113-2
EXTNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030120
Time_ 7.23
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 4
SMA 6578.947 Hz
FIDRES 0.700774 Hz
AQ 2.4904180 sec
RG 203.2
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

ID NMR plot parameters
CX 40.00 cm
FLP 10.500 ppm
F1 4202.63 Hz
F2P -0.250 ppm
F2 -100.06 Hz
PPIVM 0.26875 ppm/cm
HZCM 107.56721 Hz/cm

ppm

Current Data Parameters
NAME 71081-221
EXNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040409

Time 14.00
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 4
SWH 6578.947 Hz
F2 - 100.626 MHz
AQ 2.4904180 sec
RG 80.6
DM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====

MTC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.252014 MHz

F2 - Processing parameters

SI 16384
SF 400.2500933 MHz
WDW Hanning
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CA 400.000 MHz
F1P 10.500 ppm
F1 4202.63 Hz
F2P -10.250 ppm
F2 -100.06 Hz
PCKM 0.26875 ppm/cm
HCHN 107.56721 Hz/cm

7.90921
7.73330
7.73079
7.72873
7.71322
7.71070
7.70868
7.737740
7.736842
7.29307
7.29024
7.28533
7.27815
7.27097
7.26366
7.25779
7.25221
7.20727
7.19741
7.18726
7.17714
7.06799

5.71615
5.64304
5.59318
5.54437
5.36819

3.94334
3.67250
3.49892
3.32034
3.23241
3.10973
3.09112
3.08896
3.08199
3.07872
3.07412
3.06802
3.03287
3.03167
3.02619
3.02122
3.01807
3.01284
3.01067
3.00850
2.91834
2.86686
2.07611

1.59982

nmr400a h-1

ppm

70854-237
after lc and rx
CDCl₃
nmr 400b h-1

71081-185
rx'd prod
CDCl₃
nmr400c h1

Current Data Parameters
NAME 71081-185
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040122
Time 13:32
INSTRUM spect
PROBHD 5 mm old QNP
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 2
SWH 6561.680 Hz
FIDRES 0.707747 Hz
AQ 2.4959716 sec
RG 181
FW 76.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 13.80 usec
PL1 -5.00 dB
SFO1 400.1322007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.250 ppm
F2 -100.03 Hz
PPMCM 0.26875 ppm/cm
HZCM 107.53494 Hz/cm

71262-377
CDCl₃
nmr400c h1

70568-207
second crop
CDCl₃
nmr 400c h1

Current Data Parameters
NAME 70568-207-2
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030120
Time 12.21
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 2
SFO 6561.680 Hz
FIDRES 0.200247 Hz
AQ 2.4969716 sec
RG 228.1
RM 16.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -5.00 dB
SFO1 400.1322007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300056 MHz
WDW no
SSB 0
GB 0.00 Hz
CB 0
PC 1.00

1D NMR plot parameters
CA 40.00 cm
FLP 10.500 ppm
F1P 4201.37 Hz
F2P -0.250 ppm
FZP -100.03 Hz
PPMX 0.26875 ppm/cm
HZCM 107.53494 Hz/cm

71081-237
OH
DMSO-d6
TMR 400c H1

Current Data Parameters
NAME 71081-207
EXNO 9
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040701
Time 7.14
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 4
SHE 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 90.5
RW 76.000 usec
LB 6.00 usec
GB 300.0 K
PC 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 14.00 usec
PL1 -5.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

ID NMR plot parameters
CX 40.00 cm
F1P 10.500 ppm
F1 4202.63 Hz
F2P -0.250 ppm
F2 -100.06 Hz
PPMXX 0.26875 ppm/cm
HZCM 107.56721 Hz/cm

71497-71
CDCl₃
nmr 400a h-1

Current Data Parameters
NAME 71497-71
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041124
Time 15:31
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 4
SMH 6578.947 Hz
STH 0.700774 Hz
AQ 2.4904180 sec
RG 143.7
IM 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.1000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL -3.00 dB
SFO1 400.2522014 MHz

F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 40.00 cm
FLP 10.500 ppm
F1 4202.63 Hz
F2 -100.06 Hz
PPMCH 0.26875 ppm/cm
HZCH 107.56721 Hz/cm

70456 methoxy
CDCl₃
nmr400c h1

ta Parameters
methoxy
9
1

station Parameters
20020702
15.38

5 mm QNP 1H
zg30
32768
CDCl₃
32

2

6561.680 Hz
0.200247 Hz
2.4969716 sec
128

76.200 usec
6.00 usec
300.0 K
0.10000000 sec

0.10000000 sec

== CHANNEL f1 =====
1H
10.00 usec
-5.00 dB
400.1322007 MHz

400.1322007 MHz

ssing Parameters
16384
400.1300056 MHz
no
0
0
0
0
1.00

0.00 Hz
0.00 Hz
0
1.00

it Parameters
40.00 cm
10.500 ppm
4201.37 Hz
-0.250 ppm
-100.03 Hz
0.26875 ppm/cm
107.53494 Hz/cm

TEMP adduct
'839
CDCl₃
nmr400c h1

71262-253
CDCl₃
mmr400c h1

ppm

7.48732
7.46882
7.26880
7.23252
7.21557
7.21134
7.20029
7.19706
7.18059
7.16246
7.13893
7.11072
7.09674
7.09366
7.06035
7.05712
7.04090
7.03858
6.88026
6.86302

5.81752
5.77453

5.30592
5.26296
4.41562
4.30501
4.29786
4.27999
4.06616
4.04839
4.03939
4.03054
4.02152
4.01263
4.00356
3.98375
3.97463
3.96581
3.95675
3.94808
3.93892
3.92126
3.22629
2.84618
2.84309
2.82629
2.12527
2.11845
2.05509
1.84394
1.83858
1.83429
1.56072

70854-195
purified product
CDCl₃
nm: 400c h1

Current Data Parameters
NAME 75282-195
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters

Date_ 20030307
Time 11.04
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 2
SWH 6561.680 Hz
F2 - 100.626186 MHz
AQ 2.4969716 sec
RG 362
DM 76.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====

MUCL 1H
PL 10.00 usec
PL1 -5.00 dB
SFO1 400.1322007 MHz

F2 - Processing parameters

SI 16384
SF 400.1300656 MHz
WDW po
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CV 40.00 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P 10.350 ppm
F2 100.626186 MHz
FREQM 0.26675 ppm/cm
HSCN 107.53494 Hz/cm

71262-259
pure
CDCl₃
nmr400c h1

Current Data Parameters
 NAME 71262-309
 EXPMO 34
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040812
 Time 7.36
 INSTRUM spect
 PROBRID 5 mm old QNP
 PULPROG zg30
 TD 32768
 SOLVENT CDCl3
 NS 32
 DS 2
 SSWH 6561.680 Hz
 AQ 2.4969716 sec
 RG 128
 DM 76.200 usec
 DE 6.00 usec
 TE 300.0 K
 D1 0.1000000 sec

===== CHANNEL f1 =====
 NUC1 ¹H
 P1 10.00 usec
 PL1 -5.00 dB
 SFO1 400.1322007 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300056 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 40.00 cm
 P1P 10.500 ppm
 F1 4201.17 Hz
 F2P -0.250 ppm
 F2 -100.03 Hz
 PPM2N 0.26275 ppm/cm
 HZCN 107.53494 Hz/cm

71497-083
purified
CDCl₃
nmr400c h1

Current Data Parameters
NAME 71497-083
EXPNO 40
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041028
Time 8.11
INSTRUM spect
PROBHD 5 mm old QNP
PULPROG zg30
TD 32768
SOLVENT CDCl₃
NS 32
DS 2
SWH 6561.680 Hz
AQ 2.4969716 sec
RG 128
DM 76.200 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 ¹H
P1 10.00 usec
PL -5.00 dB
SFO1 400.132007 MHz

F2 - Processing parameters
SI 16384
SF 400.130056 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

ID NMR plot parameters
CX 40.00 cm
FLP 10.500 ppm
FI 4201.37 Hz
F2P -0.250 ppm
F2 -100.03 Hz
PRGM 0.26075 ppm/cm
HZCH 107.53494 Hz/cm

71801-221
purified
cyclopentidine
acid
MeOD-d3
nmr-400c hl

71262-55
acid
CDCl₃
nmr400c h1

Current Data Parameters
NAME 71262-065
EXTNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040628
Time 12.35
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6578.947 Hz
FIDRES 0.700764 Hz
AQ 2.490480 sec
RG 181
IN 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz

F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

ID NMR plot parameters
CX 40.00 cm
F1P 10.500 ppm
F1 4198.64 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PRCM 0.26875 ppm/cm
HZCM 107.46507 Hz/cm

X-ray crystallographic determination information for **14a**

Figure 1: ORTEP view of compound **14a**

(3*R*)-5-Bromo-4-(4-chlorobenzyl)-7-fluoro-1,2,3,4-tetrahydrocyclopenta[*b*]indol-3-ol (**14a**)

A needle of C₁₈H₁₄BrClFNO, approximate dimensions 0.50 mm x 0.07 mm x 0.04 mm was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 223(2) K on a Bruker SMART APEX CCD area detector system equipped with a graphite monochromator and a Mo K α fine-focus sealed tube (λ = 0.71073 Å) operated at 1.5 kW power (50 kV, 30 mA).

A full quadrant of data (total of 2400 frames) was collected with an exposure time of 5 sec/frame. The frames were integrated with the Bruker SAINT software package using a narrow frame integration algorithm. The integration of the data using an orthorhombic unit cell yielded a total of 17094 reflections to a maximum θ angle of 26.39°, of which 3275 were independent (completeness = 99.6%, R_{int} = 5.32%). Data were corrected for absorption effects using the multi-scan technique (SADABS).

The structure was solved and refined using the Bruker SHELXTL software package, using the space group P2₁2₁2₁, with Z = 4 for the formula unit, C₁₈H₁₄BrClFNO. The final anisotropic full-matrix least-squares refinement on F^2 with 209 variables converged at R_1 = 3.59%, for the observed data and wR_2 = 8.08% for all data. The goodness-of-fit was 0.995. The Flack parameter x associated with the correct stereochemistry assignment is 0.026(12). The largest peak on the final difference electron density map was 0.487 e⁻/Å³ and the largest hole was -0.244 e⁻/Å³. On the basis of the final model, the calculated density was 1.637 g/cm³. See the following tables for information pertaining to the final specifications of the solved structure.

ORTEP view of molecule C₁₈H₁₄BrClFNO. Anisotropic atomic displacement ellipsoids for the non-hydrogen atoms are shown at the 50% probability level.

Table 1. Crystal data and structure refinement for compound 14a.

Identification code	compound14a	
Empirical formula	C ₁₈ H ₁₄ Br Cl F N O	
Formula weight	394.66	
Temperature	223(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P212121	
Unit cell dimensions	a = 5.2721(13) Å	α = 90°.
	b = 12.916(3) Å	β = 90°.
	c = 23.515(6) Å	γ = 90°.
Volume	1601.3(7) Å ³	
Z	4	
Density (calculated)	1.637 Mg/m ³	
Absorption coefficient	2.747 mm ⁻¹	
F(000)	792	
Crystal size	0.50 x 0.07 x 0.04 mm ³	
Theta range for data collection	1.73 to 26.39°.	
Index ranges	-6 ≤ h ≤ 6, -16 ≤ k ≤ 15, -29 ≤ l ≤ 29	
Reflections collected	17094	
Independent reflections	3275 [R(int) = 0.0532]	
Completeness to theta = 26.39°	99.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	3275 / 0 / 209	
Goodness-of-fit on F ²	0.995	
Final R indices [I > 2σ(I)]	R1 = 0.0359, wR2 = 0.0756	
R indices (all data)	R1 = 0.0483, wR2 = 0.0808	
Absolute structure parameter	0.026(12)	
Largest diff. peak and hole	0.487 and -0.244 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for aem0581n. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
Br(1)	10137(1)	4610(1)	7505(1)	43(1)
C(1)	8212(6)	3396(2)	7359(1)	32(1)
C(2)	6302(6)	3161(3)	7734(1)	36(1)
C(3)	4831(6)	2289(3)	7653(1)	41(1)
C(4)	5192(7)	1611(2)	7213(1)	37(1)
C(5)	7145(6)	1841(2)	6827(1)	28(1)
C(6)	8023(6)	1341(2)	6324(1)	30(1)
C(7)	7670(7)	361(2)	5983(1)	37(1)
C(8)	9708(9)	474(3)	5519(2)	60(1)
C(9)	11100(7)	1508(2)	5583(1)	36(1)
C(10)	9924(6)	1922(2)	6114(1)	30(1)
C(11)	8656(6)	2749(2)	6890(1)	28(1)
C(12)	12455(6)	3511(2)	6340(1)	31(1)
C(13)	11675(6)	4563(2)	6112(1)	30(1)
C(14)	9559(7)	4717(2)	5782(1)	36(1)
C(15)	8915(7)	5701(2)	5593(2)	41(1)
C(16)	10416(8)	6524(2)	5736(1)	39(1)
C(17)	12576(8)	6392(3)	6051(1)	45(1)
C(18)	13192(7)	5407(2)	6240(1)	39(1)
Cl(1)	9504(2)	7769(1)	5537(1)	63(1)
F(1)	2950(4)	2098(2)	8037(1)	55(1)
N(1)	10355(5)	2787(2)	6441(1)	28(1)
O(1)	10898(4)	2177(2)	5103(1)	36(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for aem0581n.

Br(1)-C(1)	1.900(3)
C(1)-C(2)	1.373(4)
C(1)-C(11)	1.403(4)
C(2)-C(3)	1.381(5)
C(2)-H(2)	0.9400
C(3)-F(1)	1.363(4)
C(3)-C(4)	1.370(5)
C(4)-C(5)	1.403(4)
C(4)-H(4)	0.9400
C(5)-C(6)	1.425(4)
C(5)-C(11)	1.426(4)
C(6)-C(10)	1.347(4)
C(6)-C(7)	1.510(4)
C(7)-C(8)	1.539(5)
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(8)-C(9)	1.532(5)
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(9)-O(1)	1.425(3)
C(9)-C(10)	1.491(4)
C(9)-H(9)	0.9900
C(10)-N(1)	1.376(3)
C(11)-N(1)	1.386(4)
C(12)-N(1)	1.468(4)
C(12)-C(13)	1.517(4)
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(13)-C(14)	1.374(4)
C(13)-C(18)	1.384(4)
C(14)-C(15)	1.389(4)
C(14)-H(14)	0.9400
C(15)-C(16)	1.368(5)
C(15)-H(15)	0.9400

C(16)-C(17)	1.369(5)
C(16)-Cl(1)	1.742(3)
C(17)-C(18)	1.386(5)
C(17)-H(17)	0.9400
C(18)-H(18)	0.9400
O(1)-H(1)	0.8300

C(2)-C(1)-C(11)	119.7(3)
C(2)-C(1)-Br(1)	117.2(2)
C(11)-C(1)-Br(1)	123.1(2)
C(1)-C(2)-C(3)	120.2(3)
C(1)-C(2)-H(2)	119.9
C(3)-C(2)-H(2)	119.9
F(1)-C(3)-C(4)	119.1(3)
F(1)-C(3)-C(2)	117.7(3)
C(4)-C(3)-C(2)	123.2(3)
C(3)-C(4)-C(5)	117.1(3)
C(3)-C(4)-H(4)	121.5
C(5)-C(4)-H(4)	121.5
C(4)-C(5)-C(6)	132.7(3)
C(4)-C(5)-C(11)	121.1(3)
C(6)-C(5)-C(11)	106.1(3)
C(10)-C(6)-C(5)	107.1(3)
C(10)-C(6)-C(7)	111.3(3)
C(5)-C(6)-C(7)	141.3(3)
C(6)-C(7)-C(8)	102.2(3)
C(6)-C(7)-H(7A)	111.3
C(8)-C(7)-H(7A)	111.3
C(6)-C(7)-H(7B)	111.3
C(8)-C(7)-H(7B)	111.3
H(7A)-C(7)-H(7B)	109.2
C(9)-C(8)-C(7)	110.3(3)
C(9)-C(8)-H(8A)	109.6
C(7)-C(8)-H(8A)	109.6
C(9)-C(8)-H(8B)	109.6
C(7)-C(8)-H(8B)	109.6

H(8A)-C(8)-H(8B)	108.1
O(1)-C(9)-C(10)	114.5(2)
O(1)-C(9)-C(8)	114.4(3)
C(10)-C(9)-C(8)	101.3(3)
O(1)-C(9)-H(9)	108.8
C(10)-C(9)-H(9)	108.8
C(8)-C(9)-H(9)	108.8
C(6)-C(10)-N(1)	111.7(3)
C(6)-C(10)-C(9)	114.7(3)
N(1)-C(10)-C(9)	133.6(3)
N(1)-C(11)-C(1)	133.3(3)
N(1)-C(11)-C(5)	108.2(2)
C(1)-C(11)-C(5)	118.6(3)
N(1)-C(12)-C(13)	115.0(2)
N(1)-C(12)-H(12A)	108.5
C(13)-C(12)-H(12A)	108.5
N(1)-C(12)-H(12B)	108.5
C(13)-C(12)-H(12B)	108.5
H(12A)-C(12)-H(12B)	107.5
C(14)-C(13)-C(18)	118.5(3)
C(14)-C(13)-C(12)	123.3(3)
C(18)-C(13)-C(12)	118.2(3)
C(13)-C(14)-C(15)	120.8(3)
C(13)-C(14)-H(14)	119.6
C(15)-C(14)-H(14)	119.6
C(16)-C(15)-C(14)	119.4(3)
C(16)-C(15)-H(15)	120.3
C(14)-C(15)-H(15)	120.3
C(15)-C(16)-C(17)	121.2(3)
C(15)-C(16)-Cl(1)	119.4(3)
C(17)-C(16)-Cl(1)	119.3(3)
C(16)-C(17)-C(18)	118.8(3)
C(16)-C(17)-H(17)	120.6
C(18)-C(17)-H(17)	120.6
C(13)-C(18)-C(17)	121.2(3)
C(13)-C(18)-H(18)	119.4

C(17)-C(18)-H(18)	119.4
C(10)-N(1)-C(11)	106.9(2)
C(10)-N(1)-C(12)	123.4(3)
C(11)-N(1)-C(12)	129.3(2)
C(9)-O(1)-H(1)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for aem0581n. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	46(1)	35(1)	47(1)	-13(1)	-1(1)	0(1)
C(1)	32(2)	31(2)	34(2)	2(1)	-6(1)	6(1)
C(2)	38(2)	38(2)	31(2)	1(2)	-1(2)	11(2)
C(3)	36(2)	49(2)	39(2)	14(1)	6(2)	4(2)
C(4)	35(2)	32(2)	45(2)	11(1)	-1(2)	-4(2)
C(5)	31(2)	23(2)	31(2)	10(1)	-7(1)	2(1)
C(6)	37(2)	20(2)	32(2)	3(1)	-8(2)	3(1)
C(7)	40(2)	23(2)	48(2)	-1(2)	-10(2)	-1(2)
C(8)	102(4)	31(2)	46(2)	-6(2)	13(3)	-14(3)
C(9)	47(2)	25(2)	36(2)	2(1)	-6(2)	11(2)
C(10)	36(2)	20(1)	33(2)	4(1)	-3(2)	5(2)
C(11)	29(2)	25(2)	29(2)	3(1)	-5(1)	8(1)
C(12)	30(2)	24(2)	39(2)	4(1)	-1(2)	3(1)
C(13)	31(2)	28(2)	33(2)	1(1)	4(1)	1(2)
C(14)	43(2)	22(2)	43(2)	-1(1)	-3(2)	-6(2)
C(15)	48(2)	31(2)	44(2)	4(2)	-7(2)	2(2)
C(16)	60(3)	22(2)	37(2)	2(1)	5(2)	1(2)
C(17)	56(2)	30(2)	50(2)	-3(2)	3(2)	-15(2)
C(18)	38(2)	33(2)	45(2)	1(2)	-8(2)	-13(2)
Cl(1)	94(1)	24(1)	70(1)	10(1)	2(1)	4(1)
F(1)	46(1)	67(2)	53(1)	12(1)	19(1)	2(1)
N(1)	30(2)	22(1)	33(1)	-1(1)	-1(1)	1(1)
O(1)	39(1)	37(1)	33(1)	5(1)	3(1)	8(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for aem0581n.

	x	y	z	U(eq)
H(2)	5994	3596	8047	43
H(4)	4177	1017	7170	45
H(7A)	5965	324	5818	44
H(7B)	7967	-256	6217	44
H(8A)	10925	-97	5548	71
H(8B)	8908	439	5143	71
H(9)	12917	1368	5657	43
H(12A)	13635	3194	6069	37
H(12B)	13371	3612	6698	37
H(14)	8534	4149	5683	43
H(15)	7461	5800	5368	49
H(17)	13623	6960	6138	54
H(18)	14667	5310	6458	46
H(1)	9410	2378	5071	54