Calix[4]bipyrrole - A big, flexible yet effective chloride-selective anion receptor

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Supporting Information

I. ^{1}H NMR titration studies

Proton NMR-based titration studies were carried out using a Varian Unity Plus 400 MHz NMR spectrometer. All anions were used in the form of their tetrabutylammonium salts. Tetrabutylammonium chloride was dried under vacuum at 40°C for 24 h before use. All other reagents were obtained from commercial sources and used without drying or purification. The receptor solutions were titrated by adding known quantities of a concentrated solution of the anions in question. The anion solutions used to effect the titration contained the receptors at the same concentration as the receptor solutions into which they were being titrated so as to obviate a need to account for dilution effects during the titrations. The data were fit to a 1:1 binding

profile according to the method of Wilcox[1] using the changes in the pyrrolic NH resonances in the ^{1}H NMR spectra.

^{1}H NMR titrations for compound 3 (Figure 1-2)

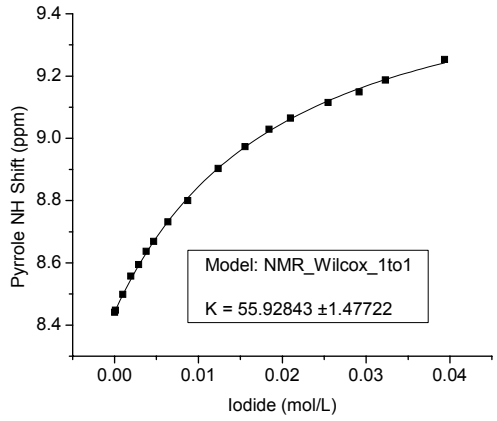


Figure 1 ^{1}H NMR spectroscopic titration curve for compound **3** and iodide anion in CD_3CN. [**3**] = 2.00×10^{-3} M. [I^-] = 0 – 39.4×10^{-3} M. The curve shows the fit of the experimental data to a 1:1 binding profile.

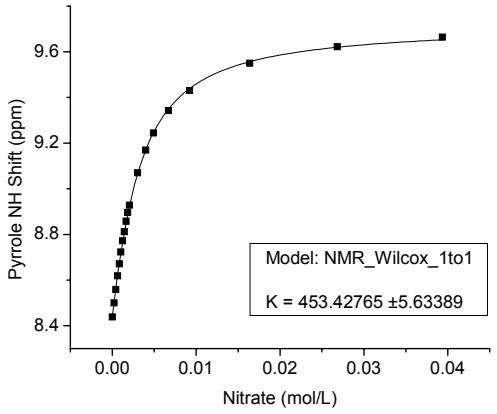


Figure. 2 ^{1}H NMR spectroscopic titration curve for compound **3** and nitrate anion in CD_3CN. [**3**] = 1.89×10^{-3} M. [NO_3^-] = 0 – 39.4×10^{-3} M. The curve shows the fit of the experimental data to a 1:1 binding profile.

^{1}H NMR titrations for compound 2 and 1 (Figure 3-4)

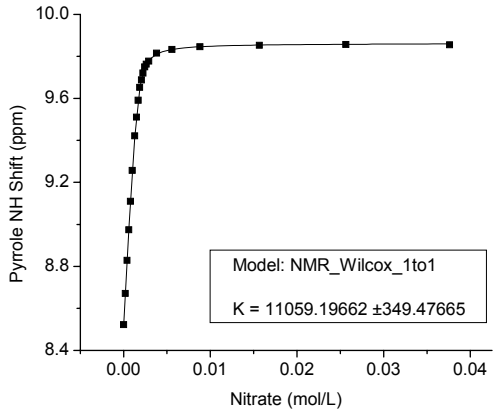


Figure 3 ^{1}H NMR spectroscopic titration curve for compound **2** and nitrate anion in CD_3CN. [**2**] = 2.25 × 10^{-3} M. [I^-] = 0 – 37.6 × 10^{-3} M. The curve shows the fit of the experimental data to a 1:1 binding profile.

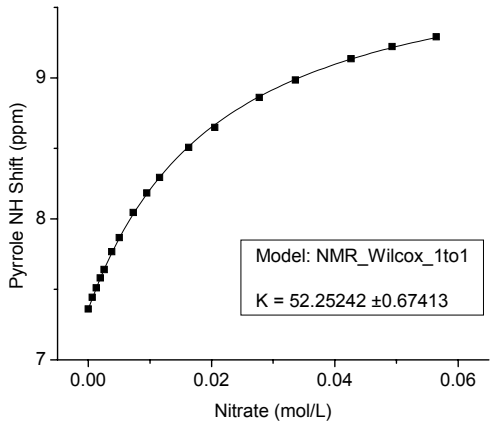


Figure 4 ^{1}H NMR spectroscopic titration curve for compound **1** and nitrate anion in CD_3CN. [**1**] = 2.32 × 10^{-3} M. [NO_3^-] = 0 – 56.5 × 10^{-3} M. The curve shows the fit of the experimental data to a 1:1 binding profile.

References:

1. Wilcox, C. S. in *Frontiers in Supramolecular Organic Chemistry and Photochemistry*; Schneider, H. J., Dürr, H., Eds.; VCH: Weinheim, **1991**.

II. ITC titration studies

Microcalorimetric titrations (experimental temperature: 30 ºC) were performed using an isothermal titration calorimeter (ITC) purchased from Microcal Inc., MA. The ORIGIN software provided by Microcal Inc. was used to calculate the biniding constant (K_a) and the enthalpy change (ΔH). The solvent, CH_3CN, was HPLC grade (Fisher) but was not further dried or purified before use.

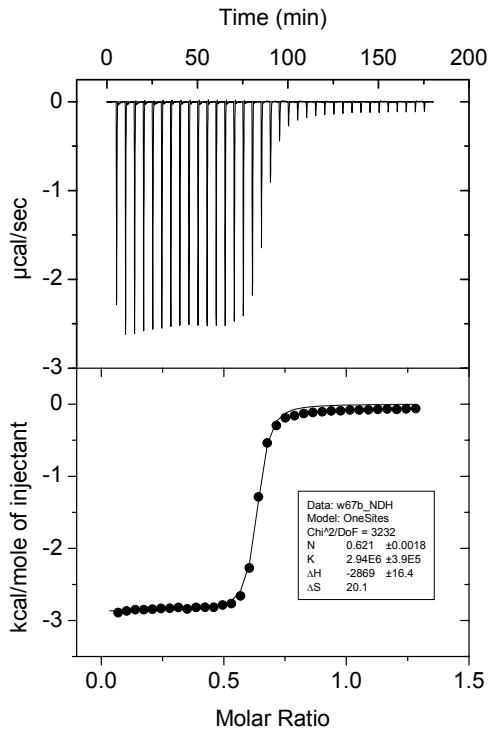


Figure 5 ITC titration curves produced from the titration of compound **3** (0.366 mM) with chloride anion (3.60 mM) in CH_3CN at 30 ºC. The curve shows the fit of the experimental data to a 1:1 binding profile.

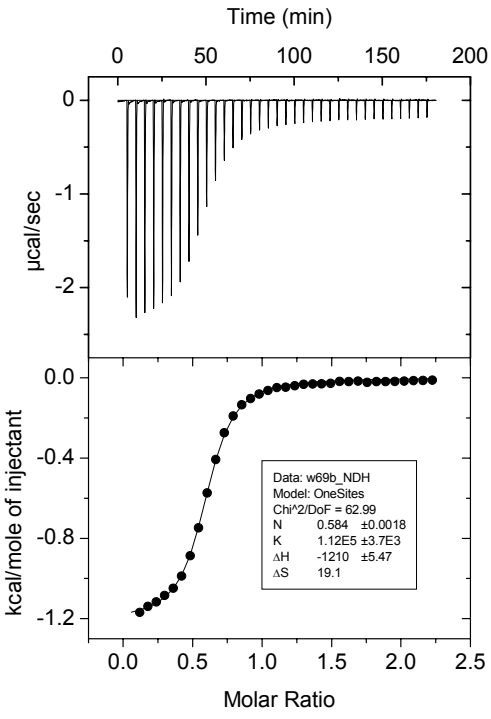


Figure 6 ITC titration curves produced from the titration of compound **3** (0.474 mM) with bromide anion (6.68 mM) in CH_3CN at 30 ºC. The curve shows the fit of the experimental data to a 1:1 binding profile.

III. X-ray experimental section

Crystallographic Material for **3**·Cl^-:

- X-ray Experimental.
- Table 1. Crystallographic Data for **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.
- Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.
- Table 3. Bond Lengths (Å) and Angles (°) for the non-hydrogen atoms of **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.
- Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.
- Table 5. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.
- Table 6. Torsion Angles (°) for the non-hydrogen atoms of **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.
- Table 7. Hydrogen Bond Lengths (Å) and Angles (°) for **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.
- Figure 1. View of **3**·Clshowing the atom labeling scheme.
- Figure 2. View illustrating the cation in **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.
- Figure 3. Unit cell packing diagram for **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.

X-ray Experimental for **3**·$C_{16}H_{36}NCl$·$2C_4H_{10}O$: $C_{68}H_{104}ClN_9O_2$, crystals grew as large, light brown needles by vapor diffusion of diethyl ether into a dichloromethane solution of **3** and tetrabutylammonium chloride (1:1 molar ratio). The data crystal was cut into an irregular shaped block that had approximate dimensions; 0.30 × 0.30 × 0.15 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoKα radiation (λ = 0.71073Å). A total of 373 frames of data were collected using ω-scans with a scan range of 1° and a counting time of 118 seconds per frame. The data were collected at 153 K using an Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.[1] The structure was solved by direct methods using SIR97[2] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[3] The hydrogen atoms on carbon were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms).

Two molecules of diethyl ether were also present in the lattice. These molecules were disordered. The contribution to the scattering due to the solvate molecules was removed by use of the utility SQUEEZE[4] as incorporated in WinGX.[5]

The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.02*P)^2]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.139, with R(F) equal to 0.0754 and a goodness of fit, S, = 1.89. Definitions used for calculating R(F), $R_w(F^2)$ and the goodness of fit, S, are given below.[6] The data were corrected for secondary extinction effects. The correction takes the form: $F_{corr} = kF_c/[1 + (7.1(4)x10^{-6})* F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. The absolute structure of the crystal was determined by the method of Flack.[7] The Flack x

parameter refined to 0.10(8) for the reported structure. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[8] All figures were generated using SHELXTL/PC.[9] Tables of positional and thermal parameters, bond lengths and angles, torsion angles, figures and lists of observed and calculated structure factors are located in tables 1 through 7.

References

1. DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.
2. SIR97. (1999). A program for crystal structure solution. Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. and Spagna, R. J. Appl. Cryst. 32, 115-119.
3. Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.
4. Spek, A. L. (1998). PLATON, A Multipurpose Crystallographic Tool. Utrecht University, The Netherlands.
5. WinGX 1.64. (1999). An Integrated System of Windows Programs for the Solution, Refinement and Analysis of Single Crystal X-ray Diffraction Data. Farrugia, L. J. J. Appl. Cryst. 32. 837-838.
6. $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ where w is the weight given each reflection. $R(F) = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|\}$ for reflections with $F_o > 4(\sigma(F_o))$.

$S = [\Sigma w(|F_o|^2 - |F_c|^2)^2/(n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.

7. Flack, H. D. (1983). Acta Cryst. A39, 876-881.
8. International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.
9. Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Table 1. Crystal data and structure refinement for **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.

Empirical formula	C68 H104 Cl N9 O2	
Formula weight	1115.05	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P212121	
Unit cell dimensions	a = 15.1706(3) Å	α= 90°.
	b = 20.1092(4) Å	β= 90°.
	c = 21.6320(5) Å	γ = 90°.
Volume	6599.2(2) $Å^3$	
Z	4	
Density (calculated)	1.122 Mg/m^3	
Absorption coefficient	0.107 mm^{-1}	
F(000)	2432	
Crystal size	0.3 x 0.3 x 0.15 mm	
Theta range for data collection	3.00 to 25.07°.	
Index ranges	-18<=h<=18, -23<=k<=23, -25<=l<=25	
Reflections collected	11343	
Independent reflections	11343	
Completeness to theta = 25.07°	98.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	11343 / 0 / 633	
Goodness-of-fit on F^2	1.849	
Final R indices [I>2sigma(I)]	R1 = 0.0754, wR2 = 0.1322	
R indices (all data)	R1 = 0.1756, wR2 = 0.1387	
Absolute structure parameter	0.10(8)	
Extinction coefficient	$7.1(4)x10^{-6}$	
Largest diff. peak and hole	0.241 and -0.186 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl1	2084(1)	2698(1)	2323(1)	59(1)
N1	1788(2)	3157(2)	814(2)	59(1)
N2	2275(2)	1752(2)	1007(2)	54(1)
N3	1318(3)	1079(2)	2153(2)	52(1)
N4	200(3)	1945(2)	2932(2)	60(1)
N5	1331(4)	2941(2)	3830(2)	75(1)
N6	3301(4)	2859(2)	3675(2)	84(2)
N7	4048(3)	3657(2)	2502(2)	75(1)
N8	2746(3)	4222(2)	1636(2)	69(1)
C1	1541(3)	3773(3)	579(3)	59(1)
C2	1540(3)	3712(3)	-58(3)	69(2)
C3	1808(3)	3046(2)	-199(3)	62(1)
C4	1957(3)	2708(3)	337(2)	51(1)
C5	2214(3)	2052(2)	435(2)	50(1)
C6	2497(3)	1570(3)	22(2)	61(1)
C7	2674(3)	979(3)	345(3)	65(2)
C8	2533(3)	1084(2)	980(3)	67(2)
C9	2562(3)	643(2)	1524(2)	50(1)
C10	1694(3)	577(2)	1815(2)	49(1)
C11	1093(4)	88(2)	1811(2)	62(1)
C12	320(4)	270(2)	2141(2)	60(1)
C13	474(3)	911(2)	2356(2)	51(1)
C14	-41(4)	1316(2)	2749(2)	52(1)
C15	-865(3)	1187(3)	3018(3)	75(2)
C16	-1091(4)	1772(3)	3380(3)	77(2)
C17	-447(4)	2236(3)	3321(2)	66(2)
C18	-291(4)	2926(2)	3585(3)	77(2)
C19	460(5)	2955(2)	4024(4)	75(2)

C20	508(6)	3009(3)	4640(4)	97(2)
C21	1370(7)	3028(3)	4818(3)	115(3)
C22	1913(6)	2990(2)	4304(3)	79(2)
C23	2841(6)	3030(2)	4229(3)	85(2)
C24	3489(6)	3219(3)	4644(3)	95(2)
C25	4296(6)	3154(3)	4337(4)	110(2)
C26	4199(5)	2937(3)	3721(4)	87(2)
C27	4816(4)	2822(3)	3194(3)	85(2)
C28	4819(5)	3433(3)	2816(3)	88(2)
C29	5470(5)	3858(4)	2617(4)	118(2)
C30	5102(6)	4343(4)	2220(4)	120(3)
C31	4240(5)	4226(3)	2148(3)	83(2)
C32	3553(5)	4538(3)	1790(3)	76(2)
C33	3501(5)	5147(3)	1488(3)	81(2)
C34	2707(4)	5204(3)	1135(2)	67(2)
C35	2270(4)	4615(3)	1242(2)	66(2)
C36	1382(4)	4379(3)	987(2)	68(2)
C37	2916(3)	-57(2)	1323(2)	72(2)
C38	3244(3)	936(2)	1982(2)	68(2)
C39	-148(3)	3423(2)	3046(2)	80(2)
C40	-1163(4)	3115(2)	3919(3)	97(2)
C41	4535(3)	2231(2)	2827(2)	89(2)
C42	5719(4)	2739(3)	3462(3)	124(2)
C43	756(3)	4190(2)	1518(2)	84(2)
C44	954(3)	4928(2)	592(2)	84(2)
N1A	4611(2)	4067(2)	-186(2)	58(1)
C1A	3769(3)	4415(2)	-317(2)	64(1)
C2A	3739(3)	4816(2)	-922(2)	70(2)
C3A	2900(4)	5159(3)	-1001(3)	78(2)
C4A	2813(4)	5473(2)	-1639(3)	89(2)
C5A	5358(3)	4562(2)	-135(2)	66(2)
C6A	5284(4)	5090(2)	340(3)	80(2)
C7A	6107(3)	5517(3)	349(3)	79(2)
C8A	6072(4)	6057(3)	863(3)	106(2)
C9A	4457(3)	3701(2)	424(2)	65(2)
C10A	5270(3)	3312(2)	673(2)	75(2)

C11A	5005(4)	2825(3)	1169(3)	86(2)
C12A	4526(4)	2223(3)	933(3)	117(2)
C13A	4848(3)	3573(3)	-696(2)	74(2)
C14A	4218(4)	3018(3)	-795(3)	112(2)
C15A	4471(4)	2611(3)	-1355(3)	114(2)
C16A	3757(7)	2066(4)	-1465(4)	222(5)

Table 3. Bond lengths [Å] and angles [°] for **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.

N1-C1	1.389(5)	C7-C8	1.405(6)
N1-C4	1.396(5)	C7-H7	0.96
N1-H1N	0.90	C8-C9	1.475(6)
N2-C5	1.379(5)	C9-C10	1.465(6)
N2-C8	1.400(5)	C9-C38	1.548(6)
N2-H2N	0.90	C9-C37	1.568(5)
N3-C10	1.371(5)	C10-C11	1.341(6)
N3-C13	1.396(5)	C11-C12	1.420(6)
N3-H3N	0.90	C11-H11	0.96
N4-C14	1.375(5)	C12-C13	1.390(5)
N4-C17	1.419(5)	C12-H12	0.96
N4-H4N	0.90	C13-C14	1.412(6)
N5-C22	1.358(7)	C14-C15	1.403(6)
N5-C19	1.388(6)	C15-C16	1.456(6)
N5-H5N	0.90	C15-H15	0.96
N6-C26	1.375(6)	C16-C17	1.356(6)
N6-C23	1.429(7)	C16-H16	0.96
N6-H6N	0.90	C17-C18	1.519(7)
N7-C31	1.408(6)	C18-C19	1.483(8)
N7-C28	1.425(6)	C18-C39	1.552(6)
N7-H7N	0.90	C18-C40	1.555(6)
N8-C35	1.369(6)	C19-C20	1.339(7)
N8-C32	1.419(7)	C20-C21	1.364(8)
N8-H8N	0.90	C20-H20	0.96
C1-C2	1.384(6)	C21-C22	1.386(8)
C1-C36	1.524(6)	C21-H21	0.96
C2-C3	1.431(6)	C22-C23	1.420(8)
C2-H2	0.96	C23-C24	1.383(8)
C3-C4	1.362(6)	C24-C25	1.399(8)
C3-H3	0.96	C24-H24	0.96
C4-C5	1.392(6)	C25-C26	1.410(8)
C5-C6	1.387(6)	C25-H25	0.96
C6-C7	1.404(6)	C26-C27	1.492(8)
C6-H6	0.96	C27-C28	1.476(8)

C27-C41	1.491(7)	C43-H43C	0.96
C27-C42	1.496(7)	C44-H44A	0.96
C28-C29	1.375(7)	C44-H44B	0.96
C29-C30	1.415(8)	C44-H44C	0.96
C29-H29	0.96	N1A-C1A	1.485(5)
C30-C31	1.339(7)	N1A-C5A	1.512(5)
C30-H30	0.96	N1A-C13A	1.528(5)
C31-C32	1.442(7)	N1A-C9A	1.530(5)
C32-C33	1.390(7)	C1A-C2A	1.537(6)
C33-C34	1.430(7)	C1A-H1AA	0.96
C33-H33	0.96	C1A-H1AB	0.96
C34-C35	1.379(6)	C2A-C3A	1.458(6)
C34-H34	0.96	C2A-H2AA	0.96
C35-C36	1.531(7)	C2A-H2AB	0.96
C36-C44	1.539(6)	C3A-C4A	1.523(6)
C36-C43	1.540(6)	C3A-H3AA	0.96
C37-H37A	0.96	C3A-H3AB	0.96
C37-H37B	0.96	C4A-H4AA	0.96
C37-H37C	0.96	C4A-H4AB	0.96
C38-H38A	0.96	C4A-H4AC	0.96
C38-H38B	0.96	C5A-C6A	1.480(6)
C38-H38C	0.96	C5A-H5AA	0.96
C39-H39A	0.96	C5A-H5AB	0.96
C39-H39B	0.96	C6A-C7A	1.516(6)
C39-H39C	0.96	C6A-H6AA	0.96
C40-H40A	0.96	C6A-H6AB	0.96
C40-H40B	0.96	C7A-C8A	1.554(6)
C40-H40C	0.96	C7A-H7AA	0.96
C41-H41A	0.96	C7A-H7AB	0.96
C41-H41B	0.96	C8A-H8AA	0.96
C41-H41C	0.96	C8A-H8AB	0.96
C42-H42A	0.96	C8A-H8AC	0.96
C42-H42B	0.96	C9A-C10A	1.557(6)
C42-H42C	0.96	C9A-H9AA	0.96
C43-H43A	0.96	C9A-H9AB	0.96
C43-H43B	0.96	C10A-C11A	1.508(6)

C10A-H10A	0.96	C13A-H13B	0.96
C10A-H10B	0.96	C14A-C15A	1.513(7)
C11A-C12A	1.502(6)	C14A-H14A	0.96
C11A-H11B	0.96	C14A-H14B	0.96
C11A-H11C	0.96	C15A-C16A	1.559(9)
C12A-H12B	0.96	C15A-H15B	0.96
C12A-H12C	0.96	C15A-H15C	0.96
C12A-H12D	0.96	C16A-H16B	0.96
C13A-C14A	1.484(6)	C16A-H16C	0.96
C13A-H13A	0.96	C16A-H16D	0.96

C1-N1-C4	110.8(4)	C2-C1-C36	130.3(5)
C1-N1-H1N	124.7	N1-C1-C36	123.0(5)
C4-N1-H1N	124.5	C1-C2-C3	107.2(5)
C5-N2-C8	113.7(4)	C1-C2-H2	125.0
C5-N2-H2N	123.1	C3-C2-H2	127.8
C8-N2-H2N	123.2	C4-C3-C2	109.4(5)
C10-N3-C13	111.8(4)	C4-C3-H3	124.7
C10-N3-H3N	124.2	C2-C3-H3	125.9
C13-N3-H3N	124.0	C3-C4-C5	130.5(5)
C14-N4-C17	111.5(4)	C3-C4-N1	106.0(4)
C14-N4-H4N	124.2	C5-C4-N1	123.4(5)
C17-N4-H4N	124.4	N2-C5-C6	104.5(4)
C22-N5-C19	112.9(6)	N2-C5-C4	124.9(4)
C22-N5-H5N	123.7	C6-C5-C4	130.5(5)
C19-N5-H5N	123.4	C5-C6-C7	109.2(4)
C26-N6-C23	113.3(6)	C5-C6-H6	125.2
C26-N6-H6N	123.5	C7-C6-H6	125.5
C23-N6-H6N	123.2	C6-C7-C8	109.3(4)
C31-N7-C28	110.3(5)	C6-C7-H7	126.1
C31-N7-H7N	124.7	C8-C7-H7	124.5
C28-N7-H7N	125.0	N2-C8-C7	103.1(5)
C35-N8-C32	110.0(5)	N2-C8-C9	123.5(5)
C35-N8-H8N	124.8	C7-C8-C9	133.3(5)
C32-N8-H8N	125.2	C10-C9-C8	111.8(4)
C2-C1-N1	106.6(4)	C10-C9-C38	111.1(4)

C8-C9-C38	107.5(4)	N5-C19-C18	122.5(6)
C10-C9-C37	110.2(4)	C19-C20-C21	109.7(7)
C8-C9-C37	109.2(4)	C19-C20-H20	125.4
C38-C9-C37	106.9(4)	C21-C20-H20	124.8
C11-C10-N3	105.1(4)	C20-C21-C22	109.9(7)
C11-C10-C9	132.5(5)	C20-C21-H21	126.3
N3-C10-C9	122.4(5)	C22-C21-H21	123.7
C10-C11-C12	111.7(4)	N5-C22-C21	102.9(7)
C10-C11-H11	123.8	N5-C22-C23	124.2(7)
C12-C11-H11	124.5	C21-C22-C23	132.7(8)
C13-C12-C11	105.6(4)	C24-C23-C22	130.2(7)
C13-C12-H12	126.7	C24-C23-N6	105.3(7)
C11-C12-H12	127.7	C22-C23-N6	124.5(6)
C12-C13-N3	105.8(4)	C23-C24-C25	106.7(7)
C12-C13-C14	130.0(5)	C23-C24-H24	126.1
N3-C13-C14	123.9(4)	C25-C24-H24	127.0
N4-C14-C15	106.8(5)	C24-C25-C26	112.8(7)
N4-C14-C13	123.8(5)	C24-C25-H25	123.8
C15-C14-C13	129.4(5)	C26-C25-H25	123.4
C14-C15-C16	106.4(4)	N6-C26-C25	101.9(7)
C14-C15-H15	125.7	N6-C26-C27	123.3(6)
C16-C15-H15	127.9	C25-C26-C27	134.8(8)
C17-C16-C15	109.6(5)	C28-C27-C41	111.7(5)
C17-C16-H16	125.4	C28-C27-C26	107.3(6)
C15-C16-H16	125.0	C41-C27-C26	110.5(5)
C16-C17-N4	105.7(4)	C28-C27-C42	107.7(6)
C16-C17-C18	134.8(5)	C41-C27-C42	112.2(5)
N4-C17-C18	119.5(5)	C26-C27-C42	107.2(5)
C19-C18-C17	113.4(5)	C29-C28-N7	104.1(6)
C19-C18-C39	110.4(5)	C29-C28-C27	133.9(7)
C17-C18-C39	109.0(5)	N7-C28-C27	121.6(6)
C19-C18-C40	110.3(5)	C28-C29-C30	109.6(6)
C17-C18-C40	105.4(5)	C28-C29-H29	125.9
C39-C18-C40	108.1(4)	C30-C29-H29	124.4
C20-C19-N5	104.5(7)	C31-C30-C29	109.6(6)
C20-C19-C18	133.0(8)	C31-C30-H30	124.6

C29-C30-H30	125.7	H39A-C39-H39B	109.5
C30-C31-N7	106.3(6)	C18-C39-H39C	109.6
C30-C31-C32	133.8(7)	H39A-C39-H39C	109.5
N7-C31-C32	119.9(6)	H39B-C39-H39C	109.5
C33-C32-N8	103.6(6)	C18-C40-H40A	109.7
C33-C32-C31	132.7(7)	C18-C40-H40B	109.7
N8-C32-C31	123.6(6)	H40A-C40-H40B	109.5
C32-C33-C34	111.8(5)	C18-C40-H40C	109.0
C32-C33-H33	123.4	H40A-C40-H40C	109.5
C34-C33-H33	124.8	H40B-C40-H40C	109.5
C35-C34-C33	104.3(5)	C27-C41-H41A	108.9
C35-C34-H34	127.4	C27-C41-H41B	108.6
C33-C34-H34	128.2	H41A-C41-H41B	109.5
N8-C35-C34	110.3(5)	C27-C41-H41C	111.0
N8-C35-C36	120.7(5)	H41A-C41-H41C	109.5
C34-C35-C36	129.0(6)	H41B-C41-H41C	109.5
C1-C36-C35	108.4(4)	C27-C42-H42A	111.8
C1-C36-C44	108.6(4)	C27-C42-H42B	107.4
C35-C36-C44	110.4(5)	H42A-C42-H42B	109.5
C1-C36-C43	109.4(4)	C27-C42-H42C	109.2
C35-C36-C43	110.5(4)	H42A-C42-H42C	109.5
C44-C36-C43	109.4(4)	H42B-C42-H42C	109.5
C9-C37-H37A	110.0	C36-C43-H43A	108.8
C9-C37-H37B	109.0	C36-C43-H43B	109.1
H37A-C37-H37B	109.5	H43A-C43-H43B	109.5
C9-C37-H37C	109.4	C36-C43-H43C	110.5
H37A-C37-H37C	109.5	H43A-C43-H43C	109.5
H37B-C37-H37C	109.5	H43B-C43-H43C	109.5
C9-C38-H38A	109.9	C36-C44-H44A	110.9
C9-C38-H38B	108.2	C36-C44-H44B	110.2
H38A-C38-H38B	109.5	H44A-C44-H44B	109.5
C9-C38-H38C	110.2	C36-C44-H44C	107.4
H38A-C38-H38C	109.5	H44A-C44-H44C	109.5
H38B-C38-H38C	109.5	H44B-C44-H44C	109.5
C18-C39-H39A	108.7	C1A-N1A-C5A	110.3(3)
C18-C39-H39B	110.1	C1A-N1A-C13A	111.8(4)

C5A-N1A-C13A	107.8(4)	C7A-C6A-H6AA	110.2
C1A-N1A-C9A	105.1(3)	C5A-C6A-H6AB	109.5
C5A-N1A-C9A	111.6(4)	C7A-C6A-H6AB	110.4
C13A-N1A-C9A	110.2(3)	H6AA-C6A-H6AB	108.5
N1A-C1A-C2A	115.8(4)	C6A-C7A-C8A	112.2(5)
N1A-C1A-H1AA	110.4	C6A-C7A-H7AA	109.8
C2A-C1A-H1AA	108.4	C8A-C7A-H7AA	108.8
N1A-C1A-H1AB	106.7	C6A-C7A-H7AB	110.0
C2A-C1A-H1AB	107.3	C8A-C7A-H7AB	107.8
H1AA-C1A-H1AB	107.9	H7AA-C7A-H7AB	108.2
C3A-C2A-C1A	111.9(4)	C7A-C8A-H8AA	112.4
C3A-C2A-H2AA	109.2	C7A-C8A-H8AB	107.5
C1A-C2A-H2AA	109.7	H8AA-C8A-H8AB	109.5
C3A-C2A-H2AB	109.1	C7A-C8A-H8AC	108.5
C1A-C2A-H2AB	108.3	H8AA-C8A-H8AC	109.5
H2AA-C2A-H2AB	108.5	H8AB-C8A-H8AC	109.5
C2A-C3A-C4A	112.1(5)	N1A-C9A-C10A	114.7(4)
C2A-C3A-H3AA	109.0	N1A-C9A-H9AA	107.6
C4A-C3A-H3AA	110.0	C10A-C9A-H9AA	105.9
C2A-C3A-H3AB	108.3	N1A-C9A-H9AB	110.5
C4A-C3A-H3AB	109.1	C10A-C9A-H9AB	109.6
H3AA-C3A-H3AB	108.2	H9AA-C9A-H9AB	108.1
C3A-C4A-H4AA	109.9	C11A-C10A-C9A	111.1(4)
C3A-C4A-H4AB	108.8	C11A-C10A-H10A	112.0
H4AA-C4A-H4AB	109.5	C9A-C10A-H10A	107.1
C3A-C4A-H4AC	109.7	C11A-C10A-H10B	108.3
H4AA-C4A-H4AC	109.5	C9A-C10A-H10B	109.6
H4AB-C4A-H4AC	109.5	H10A-C10A-H10B	108.6
C6A-C5A-N1A	117.8(4)	C12A-C11A-C10A	114.2(5)
C6A-C5A-H5AA	106.8	C12A-C11A-H11B	108.3
N1A-C5A-H5AA	107.1	C10A-C11A-H11B	107.7
C6A-C5A-H5AB	108.4	C12A-C11A-H11C	109.0
N1A-C5A-H5AB	108.4	C10A-C11A-H11C	109.3
H5AA-C5A-H5AB	108.0	H11B-C11A-H11C	108.1
C5A-C6A-C7A	110.7(5)	C11A-C12A-H12B	111.2
C5A-C6A-H6AA	107.5	C11A-C12A-H12C	109.0

H12B-C12A-H12C	109.5	C15A-C14A-H14B	107.7
C11A-C12A-H12D	108.2	H14A-C14A-H14B	109.1
H12B-C12A-H12D	109.5	C14A-C15A-C16A	109.0(5)
H12C-C12A-H12D	109.5	C14A-C15A-H15B	111.8
C14A-C13A-N1A	116.2(4)	C16A-C15A-H15B	107.3
C14A-C13A-H13A	110.0	C14A-C15A-H15C	107.2
N1A-C13A-H13A	109.0	C16A-C15A-H15C	112.7
C14A-C13A-H13B	105.0	H15B-C15A-H15C	108.9
N1A-C13A-H13B	108.2	C15A-C16A-H16B	111.2
H13A-C13A-H13B	108.2	C15A-C16A-H16C	111.2
C13A-C14A-C15A	111.0(5)	H16B-C16A-H16C	109.5
C13A-C14A-H14A	110.8	C15A-C16A-H16D	105.9
C15A-C14A-H14A	111.4	H16B-C16A-H16D	109.5
C13A-C14A-H14B	106.8	H16C-C16A-H16D	109.5

Table 4. Anisotropic displacement parameters ($Å^2$ x 10^3) for **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$. The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [h^2 $a^{*2}U^{11}$ +...+ 2 h k a* b* U^{12}].

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl1	76(1)	55(1)	45(1)	0(1)	3(1)	-9(1)
N1	73(3)	64(3)	38(3)	5(3)	0(2)	3(2)
N2	52(3)	66(3)	44(3)	-13(2)	6(2)	4(2)
N3	60(3)	40(2)	55(3)	1(2)	-14(2)	-15(2)
N4	68(3)	49(3)	62(3)	0(2)	2(2)	-12(2)
N5	136(5)	50(3)	37(3)	-6(2)	7(4)	-7(3)
N6	132(5)	76(3)	45(4)	5(3)	-26(3)	-25(3)
N7	93(4)	63(3)	68(4)	7(3)	-2(3)	-27(3)
N8	96(4)	56(3)	53(3)	-2(2)	-6(3)	-7(3)
C1	63(4)	68(4)	47(4)	7(3)	2(3)	15(3)
C2	66(4)	89(5)	51(4)	6(3)	2(3)	-8(3)
C3	81(4)	54(3)	51(4)	-13(3)	3(3)	-10(3)
C4	66(3)	62(4)	25(3)	-4(3)	4(3)	-15(3)
C5	56(3)	54(4)	41(4)	0(3)	2(3)	-6(3)
C6	71(4)	75(4)	38(3)	-7(3)	9(3)	0(3)
C7	57(3)	70(4)	69(5)	-21(3)	11(3)	2(3)
C8	70(4)	46(3)	85(5)	-23(3)	9(3)	19(3)
C9	48(4)	45(3)	57(4)	4(3)	-7(3)	3(2)
C10	53(4)	31(3)	62(4)	-3(3)	-1(3)	8(3)
C11	86(4)	46(3)	54(4)	-10(3)	-13(3)	4(3)
C12	75(4)	54(4)	51(4)	3(3)	-5(3)	-5(3)
C13	63(4)	45(3)	44(3)	6(3)	-9(3)	-3(3)
C14	72(4)	32(3)	53(3)	5(3)	0(3)	10(3)
C15	59(4)	66(4)	100(5)	11(3)	23(4)	-2(3)
C16	60(4)	69(4)	101(5)	16(4)	29(3)	2(3)
C17	80(4)	64(4)	54(4)	3(3)	28(3)	8(4)
C18	100(5)	44(3)	86(5)	-4(3)	44(4)	16(3)
C19	91(6)	69(4)	66(6)	-20(3)	28(5)	3(4)
C20	147(7)	93(5)	50(6)	-17(4)	23(5)	-1(5)

C21	185(8)	110(5)	51(5)	5(4)	36(6)	-52(6)
C22	116(6)	78(4)	42(5)	3(3)	-5(5)	-27(4)
C23	159(8)	58(4)	38(5)	1(3)	-11(6)	-8(4)
C24	131(6)	105(5)	51(5)	-2(4)	-18(5)	-15(5)
C25	136(7)	106(5)	87(6)	-2(5)	-58(6)	-26(5)
C26	88(6)	94(5)	79(6)	-2(4)	-38(5)	-14(4)
C27	87(5)	96(5)	71(5)	12(4)	-22(4)	0(4)
C28	101(5)	85(5)	77(5)	-1(4)	-32(4)	-26(4)
C29	116(6)	120(6)	116(6)	12(5)	-22(5)	-40(6)
C30	130(7)	106(6)	124(7)	3(5)	-44(5)	-69(5)
C31	111(6)	72(4)	64(5)	-11(4)	-20(4)	-26(4)
C32	104(6)	67(5)	56(4)	-8(4)	6(4)	-10(4)
C33	138(6)	46(4)	60(4)	-5(3)	41(4)	-21(4)
C34	84(4)	51(4)	66(4)	9(3)	10(4)	5(3)
C35	90(5)	72(4)	36(4)	-13(3)	8(4)	6(4)
C36	93(5)	65(4)	44(4)	7(3)	6(4)	18(3)
C37	49(3)	59(3)	107(5)	-6(3)	0(3)	14(3)
C38	43(3)	76(3)	85(4)	14(3)	-3(3)	4(3)
C39	113(5)	46(3)	81(4)	9(3)	29(3)	-8(3)
C40	115(5)	62(4)	115(5)	-16(3)	48(4)	6(3)
C41	97(4)	85(4)	84(5)	6(4)	-23(4)	3(3)
C42	102(6)	162(6)	108(6)	13(5)	-31(4)	-10(5)
C43	97(4)	93(4)	62(4)	2(3)	30(4)	14(3)
C44	112(5)	76(4)	62(4)	4(3)	-2(3)	24(3)
N1A	36(3)	77(3)	60(3)	9(3)	11(2)	3(2)
C1A	62(4)	68(3)	61(4)	7(3)	6(3)	7(3)
C2A	60(4)	75(4)	75(4)	3(3)	-14(3)	2(3)
C3A	66(4)	85(4)	84(5)	0(3)	4(4)	3(3)
C4A	90(4)	69(4)	107(5)	8(3)	-20(4)	2(3)
C5A	67(4)	76(4)	56(4)	13(3)	4(3)	1(3)
C6A	99(5)	74(4)	69(4)	18(3)	9(4)	14(4)
C7A	65(4)	84(4)	88(5)	13(4)	-3(3)	-20(3)
C8A	105(5)	97(5)	115(6)	-4(4)	-11(4)	5(4)
C9A	56(4)	73(3)	67(4)	3(3)	16(3)	2(3)
C10A	73(4)	80(4)	72(4)	27(3)	10(3)	10(3)
C11A	80(4)	96(5)	83(5)	18(4)	27(3)	21(4)

C12A	82(5)	96(5)	174(7)	15(5)	-1(4)	-23(4)
C13A	71(4)	89(4)	62(4)	4(3)	19(3)	18(3)
C14A	109(5)	113(5)	113(6)	-39(5)	42(4)	-40(4)
C15A	149(6)	68(4)	127(6)	-25(4)	46(5)	-10(4)
C16A	348(13)	145(7)	173(9)	-78(6)	114(9)	-79(8)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.

	x	y	z	U(eq)
H1N	1832	3060	1219	70
H2N	2157	1965	1363	65
H3N	1583	1470	2234	62
H4N	703	2147	2816	72
H5N	1493	2902	3431	90
H6N	3028	2715	3331	101
H7N	3514	3464	2528	90
H8N	2572	3822	1776	82
H2	1391	4061	-342	83
H3	1893	2867	-606	74
H6	2547	1629	-417	73
H7	2887	571	170	79
H11	1182	-335	1616	74
H12	-206	12	2199	72
H15	-1201	785	2969	90
H16	-1615	1821	3624	92
H20	14	3052	4914	116
H21	1584	3077	5234	138
H24	3394	3392	5052	114
H25	4857	3224	4530	132
H29	6087	3821	2710	141
H30	5409	4719	2051	144
H33	3959	5476	1502	98
H34	2502	5579	901	80
H37A	2519	-256	1031	108
H37B	3486	-4	1137	108
H37C	2965	-338	1680	108
H38A	3054	1367	2120	102

H38B	3289	642	2329	102
H38C	3810	975	1786	102
H39A	392	3312	2839	120
H39B	-629	3395	2758	120
H39C	-109	3867	3206	120
H40A	-1268	2812	4254	146
H40B	-1122	3560	4077	146
H40C	-1641	3089	3629	146
H41A	3943	2300	2683	133
H41B	4923	2186	2479	133
H41C	4558	1834	3073	133
H42A	5763	2344	3709	186
H42B	6125	2712	3123	186
H42C	5857	3121	3711	186
H43A	668	4573	1777	126
H43B	200	4052	1349	126
H43C	1001	3835	1759	126
H44A	849	5321	834	125
H44B	1328	5037	248	125
H44C	403	4758	442	125
H1AA	3618	4707	18	77
H1AB	3321	4079	-344	77
H2AA	3833	4524	-1267	84
H2AB	4206	5137	-913	84
H3AA	2846	5495	-687	94
H3AB	2433	4843	-944	94
H4AA	2253	5691	-1675	133
H4AB	3277	5792	-1692	133
H4AC	2861	5135	-1950	133
H5AA	5405	4782	-527	80
H5AB	5895	4322	-64	80
H6AA	5205	4875	732	96
H6AB	4774	5358	257	96
H7AA	6181	5732	-44	95
H7AB	6616	5245	425	95
H8AA	6592	6329	870	159

H8AB	5566	6331	783	159
H8AC	6005	5840	1255	159
H9AA	4006	3374	354	78
H9AB	4250	4003	735	78
H10A	5684	3635	822	90
H10B	5540	3068	341	90
H11B	4624	3054	1453	103
H11C	5520	2684	1392	103
H12B	4362	1932	1265	176
H12C	4007	2364	715	176
H12D	4911	1991	653	176
H13A	5427	3399	-618	89
H13B	4861	3808	-1082	89
H14A	3625	3183	-827	134
H14B	4260	2734	-439	134
H15B	5024	2387	-1298	137
H15C	4524	2913	-1697	137
H16B	3882	1813	-1831	333
H16C	3712	1773	-1117	333
H16D	3210	2300	-1518	333

Table 6. Torsion angles [°] for **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$.

C4-N1-C1-C2	0.9(5)	C8-C9-C10-N3	-73.0(6)
C4-N1-C1-C36	-175.3(4)	C38-C9-C10-N3	47.1(6)
N1-C1-C2-C3	-1.1(5)	C37-C9-C10-N3	165.4(4)
C36-C1-C2-C3	174.7(5)	N3-C10-C11-C12	0.4(5)
C1-C2-C3-C4	0.9(6)	C9-C10-C11-C12	-176.9(5)
C2-C3-C4-C5	179.0(4)	C10-C11-C12-C13	0.0(5)
C2-C3-C4-N1	-0.3(5)	C11-C12-C13-N3	-0.4(5)
C1-N1-C4-C3	-0.4(5)	C11-C12-C13-C14	-174.6(4)
C1-N1-C4-C5	-179.8(4)	C10-N3-C13-C12	0.7(5)
C8-N2-C5-C6	-3.8(5)	C10-N3-C13-C14	175.3(4)
C8-N2-C5-C4	178.9(4)	C17-N4-C14-C15	0.4(5)
C3-C4-C5-N2	-174.6(5)	C17-N4-C14-C13	179.4(4)
N1-C4-C5-N2	4.6(7)	C12-C13-C14-N4	179.1(4)
C3-C4-C5-C6	8.8(9)	N3-C13-C14-N4	5.9(7)
N1-C4-C5-C6	-172.0(4)	C12-C13-C14-C15	-2.0(8)
N2-C5-C6-C7	3.4(5)	N3-C13-C14-C15	-175.3(5)
C4-C5-C6-C7	-179.4(5)	N4-C14-C15-C16	-1.1(5)
C5-C6-C7-C8	-2.1(5)	C13-C14-C15-C16	180.0(4)
C5-N2-C8-C7	2.5(5)	C14-C15-C16-C17	1.5(6)
C5-N2-C8-C9	-173.9(4)	C15-C16-C17-N4	-1.2(6)
C6-C7-C8-N2	-0.2(5)	C15-C16-C17-C18	-178.6(6)
C6-C7-C8-C9	175.7(5)	C14-N4-C17-C16	0.6(5)
N2-C8-C9-C10	60.0(6)	C14-N4-C17-C18	178.4(5)
C7-C8-C9-C10	-115.2(6)	C16-C17-C18-C19	111.6(7)
N2-C8-C9-C38	-62.2(5)	N4-C17-C18-C19	-65.5(6)
C7-C8-C9-C38	122.6(6)	C16-C17-C18-C39	-125.0(6)
N2-C8-C9-C37	-177.8(4)	N4-C17-C18-C39	57.9(7)
C7-C8-C9-C37	6.9(7)	C16-C17-C18-C40	-9.2(9)
C13-N3-C10-C11	-0.7(5)	N4-C17-C18-C40	173.7(4)
C13-N3-C10-C9	176.9(4)	C22-N5-C19-C20	-0.6(6)
C8-C9-C10-C11	103.9(6)	C22-N5-C19-C18	178.4(4)
C38-C9-C10-C11	-136.0(5)	C17-C18-C19-C20	-106.1(7)
C37-C9-C10-C11	-17.7(7)	C39-C18-C19-C20	131.2(6)

C40-C18-C19-C20	11.8(8)	C26-C27-C28-N7	-64.1(7)
C17-C18-C19-N5	75.3(6)	C42-C27-C28-N7	-179.2(5)
C39-C18-C19-N5	-47.4(6)	N7-C28-C29-C30	2.8(7)
C40-C18-C19-N5	-166.8(4)	C27-C28-C29-C30	175.4(7)
N5-C19-C20-C21	0.1(7)	C28-C29-C30-C31	-1.8(8)
C18-C19-C20-C21	-178.7(6)	C29-C30-C31-N7	0.0(7)
C19-C20-C21-C22	0.3(8)	C29-C30-C31-C32	-177.9(6)
C19-N5-C22-C21	0.8(6)	C28-N7-C31-C30	1.8(6)
C19-N5-C22-C23	-175.9(5)	C28-N7-C31-C32	-180.0(5)
C20-C21-C22-N5	-0.6(7)	C35-N8-C32-C33	2.6(5)
C20-C21-C22-C23	175.6(6)	C35-N8-C32-C31	-173.7(5)
N5-C22-C23-C24	164.7(5)	C30-C31-C32-C33	-14.7(11)
C21-C22-C23-C24	-10.9(11)	N7-C31-C32-C33	167.6(5)
N5-C22-C23-N6	-16.5(8)	C30-C31-C32-N8	160.4(6)
C21-C22-C23-N6	167.9(6)	N7-C31-C32-N8	-17.2(8)
C26-N6-C23-C24	-0.6(6)	N8-C32-C33-C34	-2.2(6)
C26-N6-C23-C22	-179.7(5)	C31-C32-C33-C34	173.6(6)
C22-C23-C24-C25	178.7(5)	C32-C33-C34-C35	1.0(6)
N6-C23-C24-C25	-0.3(6)	C32-N8-C35-C34	-2.2(6)
C23-C24-C25-C26	1.1(8)	C32-N8-C35-C36	178.1(4)
C23-N6-C26-C25	1.2(6)	C33-C34-C35-N8	0.7(6)
C23-N6-C26-C27	-176.8(5)	C33-C34-C35-C36	-179.5(5)
C24-C25-C26-N6	-1.4(7)	C2-C1-C36-C35	-104.7(6)
C24-C25-C26-C27	176.3(6)	N1-C1-C36-C35	70.5(6)
N6-C26-C27-C28	82.5(7)	C2-C1-C36-C44	15.3(8)
C25-C26-C27-C28	-94.8(8)	N1-C1-C36-C44	-169.4(4)
N6-C26-C27-C41	-39.4(8)	C2-C1-C36-C43	134.7(5)
C25-C26-C27-C41	143.2(7)	N1-C1-C36-C43	-50.1(6)
N6-C26-C27-C42	-162.1(5)	N8-C35-C36-C1	-64.9(6)
C25-C26-C27-C42	20.6(10)	C34-C35-C36-C1	115.4(5)
C31-N7-C28-C29	-2.8(6)	N8-C35-C36-C44	176.2(4)
C31-N7-C28-C27	-176.6(5)	C34-C35-C36-C44	-3.5(7)
C41-C27-C28-C29	-114.5(8)	N8-C35-C36-C43	55.1(6)
C26-C27-C28-C29	124.3(8)	C34-C35-C36-C43	-124.7(5)
C42-C27-C28-C29	9.2(10)	C5A-N1A-C1A-C2A	60.9(5)
C41-C27-C28-N7	57.1(8)	C13A-N1A-C1A-C2A	-59.1(5)

C9A-N1A-C1A-C2A	-178.7(4)
N1A-C1A-C2A-C3A	-177.9(4)
C1A-C2A-C3A-C4A	-171.7(4)
C1A-N1A-C5A-C6A	58.5(5)
C13A-N1A-C5A-C6A	-179.1(4)
C9A-N1A-C5A-C6A	-57.9(5)
N1A-C5A-C6A-C7A	177.0(4)
C5A-C6A-C7A-C8A	-177.4(4)
C1A-N1A-C9A-C10A	-178.0(4)
C5A-N1A-C9A-C10A	-58.4(5)
C13A-N1A-C9A-C10A	61.4(5)
N1A-C9A-C10A-C11A	-164.7(4)
C9A-C10A-C11A-C12A	73.4(6)
C1A-N1A-C13A-C14A	-62.0(6)
C5A-N1A-C13A-C14A	176.6(4)
C9A-N1A-C13A-C14A	54.5(6)
N1A-C13A-C14A-C15A	173.6(5)
C13A-C14A-C15A-C16A	-176.3(6)

Table 7. Hydrogen bonds for **3**·$C_{16}H_{36}NCl$·$2C_4H_{10}O$ [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1N...Cl1	0.90	2.53	3.422(4)	173.7
N2-H2N...Cl1	0.90	2.55	3.436(4)	169.0
N3-H3N...Cl1	0.90	2.59	3.475(4)	167.9
N4-H4N...Cl1	0.90	2.60	3.491(4)	171.8
N5-H5N...Cl1	0.90	2.59	3.488(5)	173.9
N6-H6N...Cl1	0.90	2.61	3.475(5)	161.7
N7-H7N...Cl1	0.90	2.70	3.572(4)	163.8
N8-H8N...Cl1	0.90	2.66	3.551(4)	173.3

Figure 1. View of the chloride salt of the macrocycle in **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$ showing the atom labeling scheme. Displacement ellipsoids are scaled to the 30% probability level. Most hydrogen atoms have been removed for clarity. Dashed lines are indicative of N-H···Cl H-bonding interactions. The geometry of these interactions are listed in Table 7. The N···Cl contacts range from 3.422(4) Å for N1 to 3.572(4)Å for N7.

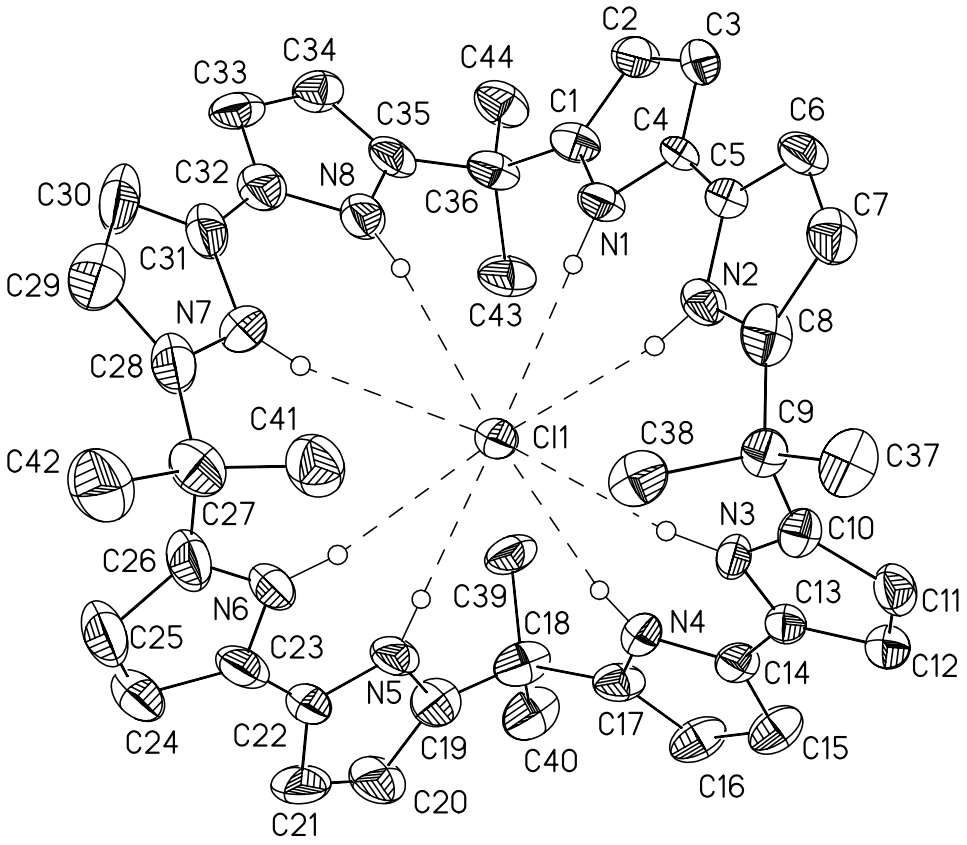


Figure 2. View illustrating the cation in **3**·$C_{16}H_{36}NCl·2C_4H_{10}O$. Displacement ellipsoids are scaled to the 30% probability level. The hydrogen atoms have been removed for clarity.

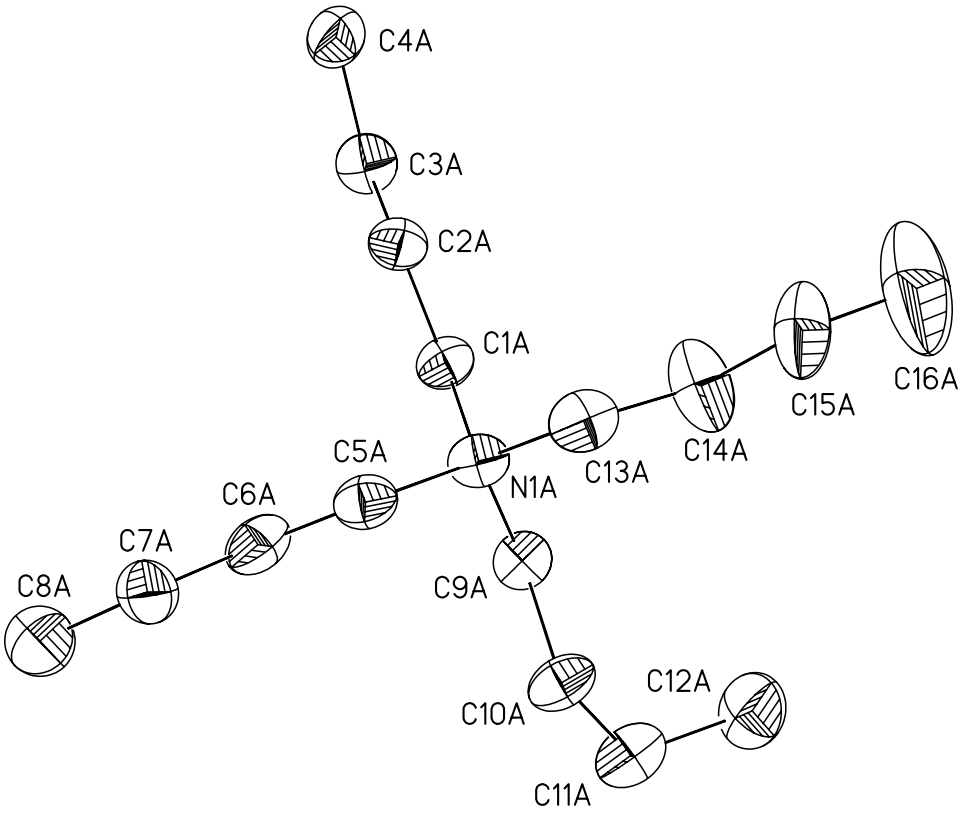
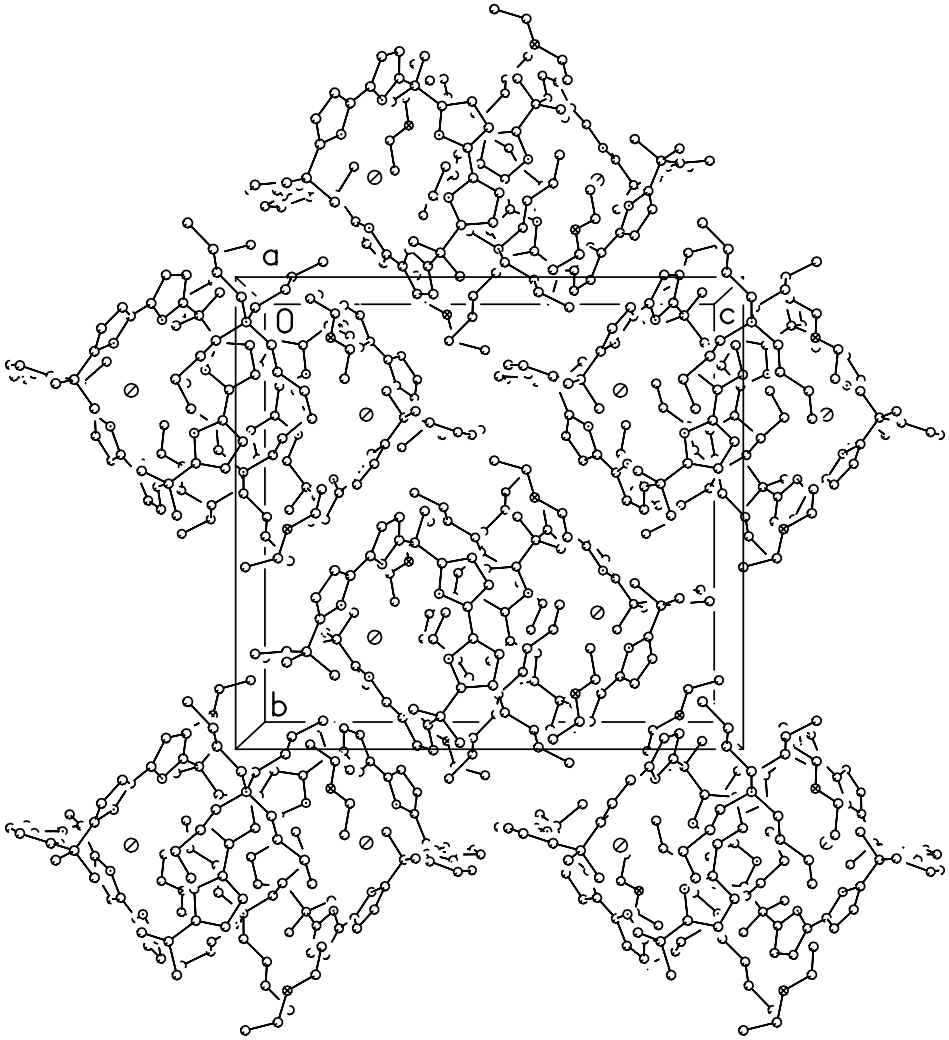


Figure 3. Unit cell packing diagram for **3**·$C_{16}H_{36}NCl$·$2C_4H_{10}O$. The view is approximately down the **a** axis.



Crystallographic Material for **3**·Br^-:

- X-ray Experimental.
- Table 1. Crystallographic Data for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.
- Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.
- Table 3. Bond Lengths (Å) and Angles (°) for the non-hydrogen atoms of 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.
- Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.
- Table 5. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.
- Table 6. Torsion Angles (°) for the non-hydrogen atoms of 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.
- Table 7. Hydrogen Bond Lengths (Å) and Angles (°) for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.
- Figure 1. View of the macrocycle-Br complex 1 in 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2 showing the atom labeling scheme.
- Figure 2. View of the macrocycle-Br complex 2 in 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2 showing the atom labeling scheme.
- Figure 3. View of one cation with atom labeling scheme
- Figure 4. View of one cation with atom labeling scheme.
- Figure 5. Unit cell packing diagram for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.

X-ray Experimental for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2: $C_{52.50}H_{69}BrClN_9$, crystals grew as pale yellow needles by slow evaporation of a dichloromethane solution containing **3** and tetraethylammonium bromide (1:1 molar ratio). The data crystal was a needle that had approximate dimensions; 0.51 × 0.11 × 0.05 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoKα radiation (λ = 0.71073Å). A total of 321 frames of data were collected using ω-scans with a scan range of 0.7° and a counting time of 172 seconds per frame. The data were collected at 153 K using an Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.[1] The structure was solved by direct methods using SIR97[2] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[3] The hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms).

The methylene groups of one of the tetraethyl ammonium ion were disordered. The disorder consisted of an inversion at the nitrogen atom of the methylene carbon atoms. The methyl groups appeared not to be involved in the disorder. The cation was modeled by assigning a variable, x, to the site occupancy factors for one set of four methylene atoms and (1-x) to the site occupancy factors for the second set of atoms. The site occupancy factors were refined while refining a common isotropic displacement parameter for the 8 methylene carbon atoms in the cation. The geometry at the methylene carbon atoms was restrained to be equal for the 8 atoms throughout refinement. In this way, the site occupancy factors for the major component of the disorder refined to 60(2)%.

The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0209*P)^2 + (3.4021*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.114, with R(F) equal to 0.0647 and a goodness of fit, S, = 1.11. Definitions used for calculating R(F), $R_w(F^2)$ and the goodness of fit, S, are given below.[4] The data were checked for secondary extinction effects but no correction was necessary. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[5] All figures were generated using SHELXTL/PC.[6] Tables of positional and thermal parameters, bond lengths and angles, torsion angles, figures and lists of observed and calculated structure factors are located in tables 1 through 8.

References

1. DENZO-SMN. (1997). Z. Otwinowski and W. Minor, Methods in Enzymology, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.
2. SIR97. (1999). A program for crystal structure solution. Altomare A., Burla M.C., Camalli M., Cascarano G.L., Giacovazzo C. , Guagliardi A., Moliterni A.G.G., Polidori G.,Spagna R. J. Appl. Cryst. 32, 115-119.
3. Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.
4. $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ where w is the weight given each reflection. $R(F) = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|\}$ for reflections with $F_o > 4(\sigma(F_o))$.

$S = [\Sigma w(|F_o|^2 - |F_c|^2)^2/(n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.

5. International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.
6. Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

Table 1. Crystal data and structure refinement for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.

Empirical formula	C52.50 H69 Br Cl N9	
Formula weight	941.53	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P21/c	
Unit cell dimensions	a = 21.4853(3) Å	α= 90°.
	b = 15.7904(2) Å	β= 91.265(1)°.
	c = 29.3770(5) Å	γ = 90°.
Volume	9964.1(3) $Å^3$	
Z	8	
Density (calculated)	1.255 Mg/m^3	
Absorption coefficient	0.923 mm^{-1}	
F(000)	3992	
Crystal size	0.50 × 0.11 × 0.05 mm	
Theta range for data collection	2.91 to 25.00°.	
Index ranges	-25<=h<=25, -18<=k<=18, -34<=l<=34	
Reflections collected	31386	
Independent reflections	17418 [R(int) = 0.1174]	
Completeness to theta = 25.00°	99.2 %	
Absorption correction	None	
Refinement method	Full-matrix-block least-squares on F^2	
Data / restraints / parameters	17418 / 128 / 1180	
Goodness-of-fit on F^2	1.090	
Final R indices [I>2sigma(I)]	R1 = 0.0647, wR2 = 0.0916	
R indices (all data)	R1 = 0.1693, wR2 = 0.1145	
Largest diff. peak and hole	0.485 and -0.567 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br1	10429(1)	2882(1)	1010(1)	26(1)
Cl1C	9495(1)	3067(1)	2142(1)	44(1)
Cl2C	9359(1)	2186(1)	2992(1)	73(1)
N1	10656(2)	1391(2)	1876(1)	28(1)
N2	11141(2)	3099(2)	2092(1)	28(1)
N3	11803(2)	4026(2)	1236(1)	29(1)
N4	11764(2)	3081(2)	362(1)	27(1)
N5	10457(2)	3250(2)	-184(1)	31(1)
N6	9410(2)	4066(2)	291(1)	26(1)
N7	8759(2)	2995(2)	1101(1)	26(1)
N8	9314(2)	1324(2)	1301(1)	27(1)
C1	10411(2)	584(3)	1884(2)	31(1)
C2	10496(2)	304(3)	2322(2)	38(1)
C3	10787(2)	942(3)	2581(2)	40(1)
C4	10887(2)	1618(3)	2299(2)	30(1)
C5	11168(2)	2439(3)	2394(2)	30(1)
C6	11500(2)	2715(3)	2772(2)	43(1)
C7	11665(2)	3562(3)	2692(2)	46(2)
C8	11441(2)	3799(3)	2268(2)	35(1)
C9	11499(2)	4616(3)	1999(2)	37(1)
C10	11950(2)	4492(3)	1614(2)	32(1)
C11	12556(2)	4745(4)	1566(2)	52(2)
C12	12771(2)	4428(3)	1151(2)	50(2)
C13	12296(2)	3977(3)	943(2)	29(1)
C14	12261(2)	3566(3)	503(2)	28(1)
C15	12667(2)	3588(3)	146(2)	37(1)
C16	12403(2)	3116(3)	-212(2)	34(1)
C17	11841(2)	2796(3)	-79(2)	29(1)

C18	11360(2)	2261(3)	-324(2)	34(1)
C19	10803(2)	2790(3)	-486(2)	28(1)
C20	10514(2)	2883(3)	-901(2)	34(1)
C21	9991(2)	3403(3)	-853(2)	35(1)
C22	9963(2)	3637(3)	-407(2)	25(1)
C23	9506(2)	4146(3)	-171(2)	25(1)
C24	9059(2)	4691(3)	-335(2)	30(1)
C25	8688(2)	4940(3)	29(2)	33(1)
C26	8906(2)	4549(3)	418(2)	28(1)
C27	8670(2)	4538(3)	899(2)	30(1)
C28	8388(2)	3676(3)	988(2)	27(1)
C29	7796(2)	3370(3)	958(2)	34(1)
C30	7804(2)	2500(3)	1059(2)	31(1)
C31	8407(2)	2271(3)	1154(2)	24(1)
C32	8684(2)	1459(3)	1273(2)	25(1)
C33	8411(2)	701(3)	1365(2)	42(2)
C34	8888(2)	100(3)	1450(2)	50(2)
C35	9449(2)	493(3)	1407(2)	32(1)
C36	10111(2)	176(3)	1470(2)	36(1)
C37	11750(2)	5308(3)	2316(2)	51(2)
C38	10861(2)	4889(3)	1804(2)	37(1)
C39	11131(2)	1544(3)	-11(2)	42(2)
C40	11661(2)	1859(3)	-745(2)	54(2)
C41	9186(2)	4730(3)	1250(2)	39(1)
C42	8151(2)	5219(3)	940(2)	50(2)
C43	10486(2)	358(3)	1037(2)	40(1)
C44	10093(2)	-795(3)	1542(2)	54(2)
N1B	8252(2)	3048(3)	4636(1)	40(1)
C1B	8184(3)	2144(4)	4780(3)	46(2)
C1BA	7602(4)	2670(6)	4702(6)	40(5)
C2B	7548(2)	1757(4)	4717(2)	69(2)
C3B	8947(3)	3308(5)	4670(2)	46(2)
C3BA	8684(5)	2774(7)	5022(4)	23(4)
C4B	9250(2)	3292(4)	5111(2)	67(2)
C5B	7896(3)	3615(4)	4971(2)	45(2)
C5BA	8094(8)	3962(6)	4534(4)	49(5)

C6B	7966(3)	4523(4)	4925(2)	75(2)
C7B	8044(3)	3267(5)	4158(2)	56(2)
C7BA	8497(8)	2480(10)	4258(3)	64(6)
C8B	8278(4)	2683(5)	3802(2)	114(3)
Br2	5727(1)	3748(1)	3690(1)	28(1)
N1'	6757(2)	5309(2)	4035(1)	26(1)
N2'	7158(2)	4218(2)	3259(1)	23(1)
N3'	6081(2)	3761(2)	2513(1)	28(1)
N4'	4879(2)	4515(2)	2730(1)	33(1)
N5'	4134(2)	3636(3)	3571(1)	35(1)
N6'	4737(2)	2132(3)	3987(1)	40(1)
N7'	5891(2)	2505(2)	4690(2)	35(1)
N8'	6050(2)	4341(2)	4848(1)	30(1)
C1'	6703(2)	5904(3)	4374(2)	27(1)
C2'	7226(2)	6395(3)	4360(2)	33(1)
C3'	7603(2)	6094(3)	4009(2)	33(1)
C4'	7309(2)	5411(3)	3809(2)	24(1)
C5'	7494(2)	4906(3)	3421(2)	22(1)
C6'	7992(2)	4992(3)	3148(2)	33(1)
C7'	7960(2)	4348(3)	2813(2)	35(1)
C8'	7439(2)	3870(3)	2887(2)	26(1)
C9'	7152(2)	3146(3)	2617(2)	29(1)
C10'	6622(2)	3470(3)	2323(2)	31(1)
C11'	6549(2)	3590(4)	1867(2)	49(2)
C12'	5964(2)	3953(4)	1780(2)	51(2)
C13'	5675(2)	4058(3)	2186(2)	31(1)
C14'	5074(2)	4421(3)	2292(2)	32(1)
C15'	4603(2)	4724(3)	2017(2)	48(2)
C16'	4121(2)	5007(3)	2297(2)	48(2)
C17'	4293(2)	4884(3)	2740(2)	34(1)
C18'	3977(2)	5054(3)	3184(2)	38(1)
C19'	3737(2)	4234(3)	3381(2)	37(1)
C20'	3151(2)	3898(4)	3402(2)	54(2)
C21'	3194(2)	3086(4)	3599(2)	53(2)
C22'	3810(2)	2924(4)	3708(2)	37(1)
C23'	4107(2)	2190(4)	3908(2)	39(1)

C24'	3864(3)	1432(4)	4043(2)	60(2)
C25'	4354(3)	917(4)	4205(2)	58(2)
C26'	4900(3)	1353(3)	4175(2)	45(2)
C27'	5563(2)	1141(3)	4316(2)	43(2)
C28'	5767(2)	1654(3)	4720(2)	40(1)
C29'	5859(3)	1458(4)	5167(2)	58(2)
C30'	6024(3)	2186(4)	5409(2)	55(2)
C31'	6048(2)	2846(3)	5106(2)	33(1)
C32'	6202(2)	3726(3)	5170(2)	29(1)
C33'	6486(2)	4147(3)	5525(2)	38(1)
C34'	6500(2)	5016(3)	5424(2)	39(1)
C35'	6231(2)	5124(3)	5004(2)	30(1)
C36'	6166(2)	5893(3)	4700(2)	28(1)
C37'	6918(2)	2438(3)	2938(2)	39(1)
C38'	7652(2)	2766(3)	2309(2)	43(2)
C39'	3421(2)	5654(3)	3093(2)	57(2)
C40'	4437(2)	5495(3)	3516(2)	39(1)
C41'	5994(2)	1307(3)	3909(2)	49(2)
C42'	5605(3)	187(3)	4437(2)	62(2)
C43'	6193(2)	6695(3)	5001(2)	47(2)
C44'	5534(2)	5883(3)	4440(2)	35(1)
C1C	9355(2)	2095(3)	2407(2)	35(1)
N1A	6275(2)	7009(3)	2771(2)	43(1)
C1A	6008(3)	6899(4)	3244(2)	65(2)
C2A	5767(3)	7719(4)	3459(2)	94(3)
C3A	6813(2)	7630(3)	2780(2)	38(1)
C4A	7334(2)	7410(3)	3107(2)	54(2)
C5A	6491(3)	6136(3)	2633(2)	58(2)
C6A	6811(3)	6086(4)	2177(2)	90(2)
C7A	5799(2)	7374(3)	2435(2)	55(2)
C8A	5208(3)	6874(5)	2375(3)	126(4)

Table 3. Bond lengths [Å] and angles [°] for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.

Cl1C-C1C	1.751(4)	C6-C7	1.404(7)
Cl2C-C1C	1.722(5)	C6-H6	0.96
N1-C4	1.375(6)	C7-C8	1.376(6)
N1-C1	1.378(5)	C7-H7	0.96
N1-H1N	0.90	C8-C9	1.520(7)
N2-C5	1.370(5)	C9-C10	1.518(6)
N2-C8	1.376(5)	C9-C37	1.525(6)
N2-H2N	0.90	C9-C38	1.537(6)
N3-C10	1.365(5)	C10-C11	1.372(6)
N3-C13	1.382(5)	C11-C12	1.404(7)
N3-H3N	0.90	C11-H11	0.96
N4-C14	1.370(5)	C12-C13	1.376(6)
N4-C17	1.384(6)	C12-H12	0.96
N4-H4N	0.90	C13-C14	1.448(6)
N5-C22	1.377(5)	C14-C15	1.379(6)
N5-C19	1.377(5)	C15-C16	1.400(6)
N5-H5N	0.90	C15-H15	0.96
N6-C26	1.381(5)	C16-C17	1.375(6)
N6-C23	1.383(5)	C16-H16	0.96
N6-H6N	0.90	C17-C18	1.504(6)
N7-C28	1.374(5)	C18-C19	1.527(6)
N7-C31	1.380(5)	C18-C40	1.544(6)
N7-H7N	0.90	C18-C39	1.546(6)
N8-C32	1.370(5)	C19-C20	1.366(6)
N8-C35	1.378(5)	C20-C21	1.402(6)
N8-H8N	0.90	C20-H20	0.96
C1-C2	1.368(6)	C21-C22	1.363(6)
C1-C36	1.508(7)	C21-H21	0.96
C2-C3	1.403(7)	C22-C23	1.457(6)
C2-H2	0.96	C23-C24	1.370(6)
C3-C4	1.372(6)	C24-C25	1.405(6)
C3-H3	0.96	C24-H24	0.96
C4-C5	1.455(6)	C25-C26	1.372(6)
C5-C6	1.378(6)	C25-H25	0.96

C26-C27	1.512(6)	C43-H43A	0.96
C27-C28	1.515(6)	C43-H43B	0.96
C27-C41	1.527(6)	C43-H43C	0.96
C27-C42	1.555(6)	C44-H44A	0.96
C28-C29	1.361(6)	C44-H44B	0.96
C29-C30	1.405(6)	C44-H44C	0.96
C29-H29	0.96	N1B-C1B	1.497(6)
C30-C31	1.368(6)	N1B-C7B	1.502(6)
C30-H30	0.96	N1B-C5BA	1.511(8)
C31-C32	1.453(6)	N1B-C3BA	1.512(7)
C32-C33	1.363(6)	N1B-C7BA	1.530(8)
C33-C34	1.414(7)	N1B-C1BA	1.537(8)
C33-H33	0.96	N1B-C5B	1.546(6)
C34-C35	1.365(6)	N1B-C3B	1.550(6)
C34-H34	0.96	C1B-C2B	1.507(6)
C35-C36	1.514(6)	C1B-C1BA	1.516(13)
C36-C44	1.549(6)	C1B-C3BA	1.615(11)
C36-C43	1.549(6)	C1B-C7BA	1.771(16)
C37-H37A	0.96	C1B-H1BA	0.96
C37-H37B	0.96	C1B-H1BB	0.96
C37-H37C	0.96	C1B-H2BF	1.3741
C38-H38A	0.96	C1B-H3BC	1.4555
C38-H38B	0.96	C1B-H7BC	1.4564
C38-H38C	0.96	C1BA-C2B	1.447(8)
C39-H39A	0.96	C1BA-C5B	1.797(14)
C39-H39B	0.96	C1BA-H1BC	0.96
C39-H39C	0.96	C1BA-H1BD	0.96
C40-H40A	0.96	C2B-H2BD	0.96
C40-H40B	0.96	C2B-H2BE	0.96
C40-H40C	0.96	C2B-H2BF	0.96
C41-H41A	0.96	C2B-H2BA	0.96
C41-H41B	0.96	C2B-H2BB	0.96
C41-H41C	0.96	C2B-H2BC	0.96
C42-H42A	0.96	C3B-C4B	1.437(6)
C42-H42B	0.96	C3B-H3BA	0.96
C42-H42C	0.96	C3B-H3BB	0.96

C3BA-C4B	1.485(7)	N2'-C8'	1.376(5)
C3BA-H3BC	0.96	N2'-C5'	1.383(5)
C3BA-H3BD	0.96	N2'-H2'N	0.90
C4B-H4BD	0.96	N3'-C13'	1.366(5)
C4B-H4BE	0.96	N3'-C10'	1.380(5)
C4B-H4BF	0.96	N3'-H3'N	0.90
C4B-H4BA	0.96	N4'-C14'	1.371(5)
C4B-H4BB	0.96	N4'-C17'	1.389(5)
C4B-H4BC	0.96	N4'-H4'N	0.90
C5B-C6B	1.447(6)	N5'-C19'	1.383(6)
C5B-C5BA	1.467(14)	N5'-C22'	1.387(6)
C5B-H5BA	0.96	N5'-H5'N	0.90
C5B-H5BB	0.96	N6'-C23'	1.371(6)
C5BA-C6B	1.483(8)	N6'-C26'	1.388(6)
C5BA-H5BC	0.96	N6'-H6'N	0.90
C5BA-H5BD	0.96	N7'-C31'	1.371(6)
C6B-H6BD	0.96	N7'-C28'	1.374(6)
C6B-H6BE	0.96	N7'-H7'N	0.90
C6B-H6BF	0.96	N8'-C35'	1.372(5)
C6B-H6BA	0.96	N8'-C32'	1.387(5)
C6B-H6BB	0.96	N8'-H8'N	0.90
C6B-H6BC	0.96	C1'-C2'	1.368(6)
C7B-C8B	1.490(6)	C1'-C36'	1.516(6)
C7B-H7BA	0.96	C2'-C3'	1.407(6)
C7B-H7BB	0.96	C2'-H2'	0.96
C7BA-H7BC	0.96	C3'-C4'	1.375(6)
C7BA-H7BD	0.96	C3'-H3'	0.96
C8B-H8BD	0.96	C4'-C5'	1.453(6)
C8B-H8BE	0.96	C5'-C6'	1.359(6)
C8B-H8BF	0.96	C6'-C7'	1.415(6)
C8B-H8BA	0.96	C6'-H6'	0.96
C8B-H8BB	0.96	C7'-C8'	1.372(6)
C8B-H8BC	0.96	C7'-H7'	0.96
N1'-C1'	1.376(5)	C8'-C9'	1.515(6)
N1'-C4'	1.382(5)	C9'-C10'	1.504(6)
N1'-H1'N	0.90	C9'-C38'	1.541(6)

C9'-C37'	1.554(6)	C31'-C32'	1.439(7)
C10'-C11'	1.359(6)	C32'-C33'	1.370(6)
C11'-C12'	1.399(6)	C33'-C34'	1.405(7)
C11'-H11'	0.96	C33'-H33'	0.96
C12'-C13'	1.367(6)	C34'-C35'	1.363(6)
C12'-H12'	0.96	C34'-H34'	0.96
C13'-C14'	1.452(6)	C35'-C36'	1.510(6)
C14'-C15'	1.368(6)	C36'-C44'	1.544(6)
C15'-C16'	1.410(6)	C36'-C43'	1.546(6)
C15'-H15'	0.96	C37'-H37D	0.96
C16'-C17'	1.359(6)	C37'-H37E	0.96
C16'-H16'	0.96	C37'-H37F	0.96
C17'-C18'	1.506(6)	C38'-H38D	0.96
C18'-C19'	1.514(7)	C38'-H38E	0.96
C18'-C40'	1.541(6)	C38'-H38F	0.96
C18'-C39'	1.544(6)	C39'-H39D	0.96
C19'-C20'	1.368(6)	C39'-H39E	0.96
C20'-C21'	1.410(7)	C39'-H39F	0.96
C20'-H20'	0.96	C40'-H40D	0.96
C21'-C22'	1.380(7)	C40'-H40E	0.96
C21'-H21'	0.96	C40'-H40F	0.96
C22'-C23'	1.441(7)	C41'-H41D	0.96
C23'-C24'	1.368(7)	C41'-H41E	0.96
C24'-C25'	1.406(7)	C41'-H41F	0.96
C24'-H24'	0.96	C42'-H42D	0.96
C25'-C26'	1.364(7)	C42'-H42E	0.96
C25'-H25'	0.96	C42'-H42F	0.96
C26'-C27'	1.514(7)	C43'-H43D	0.96
C27'-C28'	1.495(7)	C43'-H43E	0.96
C27'-C41'	1.550(6)	C43'-H43F	0.96
C27'-C42'	1.549(6)	C44'-H44D	0.96
C28'-C29'	1.358(7)	C44'-H44E	0.96
C29'-C30'	1.394(7)	C44'-H44F	0.96
C29'-H29'	0.96	C1C-H1CA	0.96
C30'-C31'	1.371(7)	C1C-H1CB	0.96
C30'-H30'	0.96	N1A-C5A	1.513(6)

N1A-C3A	1.517(5)	C4A-H4AC	0.96
N1A-C7A	1.520(6)	C5A-C6A	1.522(8)
N1A-C1A	1.523(6)	C5A-H5AA	0.96
C1A-C2A	1.535(8)	C5A-H5AB	0.96
C1A-H1AA	0.96	C6A-H6AA	0.96
C1A-H1AB	0.96	C6A-H6AB	0.96
C2A-H2AA	0.96	C6A-H6AC	0.96
C2A-H2AB	0.96	C7A-C8A	1.503(7)
C2A-H2AC	0.96	C7A-H7AA	0.96
C3A-C4A	1.499(6)	C7A-H7AB	0.96
C3A-H3AA	0.96	C8A-H8AA	0.96
C3A-H3AB	0.96	C8A-H8AB	0.96
C4A-H4AA	0.96	C8A-H8AC	0.96
C4A-H4AB	0.96		

C4-N1-C1	110.8(4)	C32-N8-C35	111.5(4)
C4-N1-H1N	124.8	C32-N8-H8N	124.5
C1-N1-H1N	124.4	C35-N8-H8N	124.0
C5-N2-C8	110.7(4)	C2-C1-N1	105.8(5)
C5-N2-H2N	124.8	C2-C1-C36	131.6(5)
C8-N2-H2N	124.5	N1-C1-C36	122.5(5)
C10-N3-C13	111.7(4)	C1-C2-C3	109.1(5)
C10-N3-H3N	124.1	C1-C2-H2	124.7
C13-N3-H3N	124.2	C3-C2-H2	126.3
C14-N4-C17	110.9(4)	C4-C3-C2	107.7(5)
C14-N4-H4N	124.4	C4-C3-H3	125.3
C17-N4-H4N	124.7	C2-C3-H3	126.9
C22-N5-C19	110.4(4)	C3-C4-N1	106.6(5)
C22-N5-H5N	124.6	C3-C4-C5	130.4(5)
C19-N5-H5N	125.0	N1-C4-C5	123.0(4)
C26-N6-C23	110.5(4)	N2-C5-C6	107.2(4)
C26-N6-H6N	124.4	N2-C5-C4	122.9(4)
C23-N6-H6N	125.1	C6-C5-C4	129.9(5)
C28-N7-C31	111.1(4)	C5-C6-C7	107.1(5)
C28-N7-H7N	124.4	C5-C6-H6	126.5
C31-N7-H7N	124.5	C7-C6-H6	126.4

C8-C7-C6	109.0(5)	C17-C18-C19	111.4(4)
C8-C7-H7	124.3	C17-C18-C40	108.6(4)
C6-C7-H7	126.7	C19-C18-C40	108.4(4)
N2-C8-C7	105.9(5)	C17-C18-C39	110.5(4)
N2-C8-C9	121.9(4)	C19-C18-C39	109.2(4)
C7-C8-C9	132.1(5)	C40-C18-C39	108.7(4)
C10-C9-C8	109.8(4)	C20-C19-N5	106.1(4)
C10-C9-C37	108.9(4)	C20-C19-C18	132.4(5)
C8-C9-C37	108.9(4)	N5-C19-C18	121.4(4)
C10-C9-C38	109.7(4)	C19-C20-C21	108.8(4)
C8-C9-C38	110.5(4)	C19-C20-H20	124.8
C37-C9-C38	109.1(4)	C21-C20-H20	126.4
N3-C10-C11	106.0(5)	C22-C21-C20	108.0(4)
N3-C10-C9	122.4(4)	C22-C21-H21	125.3
C11-C10-C9	131.4(5)	C20-C21-H21	126.7
C10-C11-C12	108.4(5)	C21-C22-N5	106.7(4)
C10-C11-H11	125.1	C21-C22-C23	130.7(4)
C12-C11-H11	126.4	N5-C22-C23	122.5(4)
C13-C12-C11	108.4(5)	C24-C23-N6	106.5(4)
C13-C12-H12	125.6	C24-C23-C22	130.8(4)
C11-C12-H12	125.9	N6-C23-C22	122.3(4)
C12-C13-N3	105.4(5)	C23-C24-C25	108.2(4)
C12-C13-C14	130.8(5)	C23-C24-H24	125.5
N3-C13-C14	123.7(4)	C25-C24-H24	126.4
N4-C14-C15	106.7(4)	C26-C25-C24	108.7(4)
N4-C14-C13	123.1(4)	C26-C25-H25	124.8
C15-C14-C13	130.2(5)	C24-C25-H25	126.5
C14-C15-C16	107.7(4)	C25-C26-N6	106.2(4)
C14-C15-H15	125.5	C25-C26-C27	131.9(4)
C16-C15-H15	126.8	N6-C26-C27	121.8(4)
C17-C16-C15	109.1(5)	C26-C27-C28	108.4(4)
C17-C16-H16	125.3	C26-C27-C41	112.0(4)
C15-C16-H16	125.7	C28-C27-C41	110.4(4)
C16-C17-N4	105.7(4)	C26-C27-C42	108.7(4)
C16-C17-C18	132.0(5)	C28-C27-C42	108.5(4)
N4-C17-C18	122.4(4)	C41-C27-C42	108.8(4)

C29-C28-N7	105.9(4)	C9-C38-H38A	108.7
C29-C28-C27	133.2(4)	C9-C38-H38B	109.8
N7-C28-C27	120.9(4)	H38A-C38-H38B	109.5
C28-C29-C30	109.1(4)	C9-C38-H38C	109.9
C28-C29-H29	124.7	H38A-C38-H38C	109.5
C30-C29-H29	126.2	H38B-C38-H38C	109.5
C31-C30-C29	107.9(4)	C18-C39-H39A	109.1
C31-C30-H30	125.1	C18-C39-H39B	109.8
C29-C30-H30	127.0	H39A-C39-H39B	109.5
C30-C31-N7	106.1(4)	C18-C39-H39C	109.5
C30-C31-C32	131.5(4)	H39A-C39-H39C	109.5
N7-C31-C32	122.4(4)	H39B-C39-H39C	109.5
C33-C32-N8	106.2(4)	C18-C40-H40A	109.1
C33-C32-C31	130.4(4)	C18-C40-H40B	109.4
N8-C32-C31	123.4(4)	H40A-C40-H40B	109.4
C32-C33-C34	108.2(4)	C18-C40-H40C	109.9
C32-C33-H33	125.5	H40A-C40-H40C	109.5
C34-C33-H33	126.3	H40B-C40-H40C	109.5
C35-C34-C33	108.4(4)	C27-C41-H41A	109.8
C35-C34-H34	125.3	C27-C41-H41B	109.7
C33-C34-H34	126.2	H41A-C41-H41B	109.5
C34-C35-N8	105.7(4)	C27-C41-H41C	108.9
C34-C35-C36	131.9(4)	H41A-C41-H41C	109.5
N8-C35-C36	122.4(4)	H41B-C41-H41C	109.5
C1-C36-C35	110.0(4)	C27-C42-H42A	108.7
C1-C36-C44	108.9(4)	C27-C42-H42B	109.8
C35-C36-C44	108.5(4)	H42A-C42-H42B	109.5
C1-C36-C43	111.3(4)	C27-C42-H42C	109.9
C35-C36-C43	110.0(4)	H42A-C42-H42C	109.5
C44-C36-C43	108.1(4)	H42B-C42-H42C	109.5
C9-C37-H37A	109.7	C36-C43-H43A	109.0
C9-C37-H37B	109.7	C36-C43-H43B	110.0
H37A-C37-H37B	109.5	H43A-C43-H43B	109.5
C9-C37-H37C	109.0	C36-C43-H43C	109.4
H37A-C37-H37C	109.5	H43A-C43-H43C	109.5
H37B-C37-H37C	109.5	H43B-C43-H43C	109.5

C36-C44-H44A	110.1	H2BD-C2B-H2BE	109.5
C36-C44-H44B	108.4	C1BA-C2B-H2BF	109.6
H44A-C44-H44B	109.5	H2BD-C2B-H2BF	109.5
C36-C44-H44C	109.9	H2BE-C2B-H2BF	109.5
H44A-C44-H44C	109.5	C1B-C2B-H2BA	110.0
H44B-C44-H44C	109.5	C1B-C2B-H2BB	109.7
C1B-N1B-C7B	117.1(5)	H2BF-C2B-H2BB	100.0
C5BA-N1B-C7BA	119.7(7)	H2BA-C2B-H2BB	109.5
C3BA-N1B-C7BA	99.3(8)	C1B-C2B-H2BC	108.8
C5BA-N1B-C1BA	101.2(8)	H2BA-C2B-H2BC	109.5
C3BA-N1B-C1BA	109.7(8)	H2BB-C2B-H2BC	109.5
C7BA-N1B-C1BA	101.1(10)	C4B-C3B-N1B	118.1(5)
C1B-N1B-C5B	108.6(4)	C4B-C3B-H3BA	108.0
C7B-N1B-C5B	108.7(5)	N1B-C3B-H3BA	108.2
C1B-N1B-C3B	109.5(4)	C7BA-C3B-H3BA	114.9
C7B-N1B-C3B	105.4(4)	C4B-C3B-H3BB	107.1
C5B-N1B-C3B	107.2(5)	C3BA-C3B-H3BB	106.0
N1B-C1B-C2B	116.5(5)	N1B-C3B-H3BB	107.6
C1BA-C1B-C3BA	105.6(7)	H3BA-C3B-H3BB	107.4
N1B-C1B-H1BA	108.2	C4B-C3BA-N1B	117.4(7)
C2B-C1B-H1BA	107.7	C3B-C3BA-C1B	108.0(7)
N1B-C1B-H1BB	108.2	C3B-C3BA-H3BC	104.7
C2B-C1B-H1BB	108.6	C4B-C3BA-H3BC	106.7
H1BA-C1B-H1BB	107.5	N1B-C3BA-H3BC	107.0
C2B-C1BA-N1B	117.7(7)	C4B-C3BA-H3BD	109.2
C2B-C1BA-H1BC	108.3	N1B-C3BA-H3BD	108.6
N1B-C1BA-H1BC	108.1	H3BC-C3BA-H3BD	107.4
C5B-C1BA-H1BC	103.9	H1BA-C3BA-H4BC	114.9
C2B-C1BA-H1BD	107.2	C3BA-C4B-H4BD	109.9
C1B-C1BA-H1BD	111.2	C3BA-C4B-H4BE	110.5
N1B-C1BA-H1BD	107.8	H4BD-C4B-H4BE	109.5
H1BC-C1BA-H1BD	107.3	C3BA-C4B-H4BF	108.0
H1BC-C1BA-H2BB	101.6	H4BD-C4B-H4BF	109.5
H2BB-C1BA-H5BB	107.4	H4BE-C4B-H4BF	109.5
C1BA-C2B-H2BD	110.3	C3B-C4B-H4BA	110.8
C1BA-C2B-H2BE	108.5	C3B-C4B-H4BB	108.4

H4BA-C4B-H4BB	109.5	C5BA-C7B-C7BA	112.7(7)
C3B-C4B-H4BC	109.2	C8B-C7B-H7BA	108.1
H4BE-C4B-H4BC	112.4	N1B-C7B-H7BA	108.8
H4BA-C4B-H4BC	109.5	C8B-C7B-H7BB	108.6
H4BB-C4B-H4BC	109.5	N1B-C7B-H7BB	108.8
C6B-C5B-N1B	117.4(5)	H7BA-C7B-H7BB	107.7
C6B-C5B-H5BA	107.7	C8B-C7BA-N1B	115.7(8)
N1B-C5B-H5BA	108.1	C8B-C7BA-H7BC	107.9
C1BA-C5B-H5BA	107.0	N1B-C7BA-H7BC	107.4
C6B-C5B-H5BB	107.7	C8B-C7BA-H7BD	109.0
C5BA-C5B-H5BB	107.1	N1B-C7BA-H7BD	109.0
N1B-C5B-H5BB	108.0	H7BC-C7BA-H7BD	107.6
H5BA-C5B-H5BB	107.5	C8B-C7BA-H8BB	38.8
C6B-C5BA-N1B	117.5(8)	N1B-C7BA-H8BB	130.8
C5B-C5BA-C7B	109.9(8)	C7B-C7BA-H8BB	82.9
C5B-C5BA-H5BC	104.8	C1B-C7BA-H8BB	174.7
C6B-C5BA-H5BC	106.8	C3B-C7BA-H8BB	103.3
N1B-C5BA-H5BC	106.9	H1BB-C7BA-H8BB	144.5
C6B-C5BA-H5BD	109.1	H7BC-C7BA-H8BB	119.8
N1B-C5BA-H5BD	108.7	H7BD-C7BA-H8BB	70.3
H5BC-C5BA-H5BD	107.4	C8B-C7BA-H8BC	38.1
H6BC-C5BA-H7BA	114.3	N1B-C7BA-H8BC	127.0
C5BA-C6B-H6BD	110.3	C7B-C7BA-H8BC	81.2
C5BA-C6B-H6BE	110.2	C1B-C7BA-H8BC	110.8
H6BD-C6B-H6BE	109.5	C3B-C7BA-H8BC	161.5
C5BA-C6B-H6BF	107.9	H1BB-C7BA-H8BC	104.8
H6BD-C6B-H6BF	109.5	H7BC-C7BA-H8BC	69.8
H6BE-C6B-H6BF	109.5	H7BD-C7BA-H8BC	122.5
C5B-C6B-H6BA	110.7	H8BB-C7BA-H8BC	64.4
C5B-C6B-H6BB	108.8	C7BA-C8B-C7B	66.1(6)
H6BA-C6B-H6BB	109.5	C7BA-C8B-H8BD	109.9
C5B-C6B-H6BC	108.8	C7B-C8B-H8BD	175.9
H6BE-C6B-H6BC	111.2	C7BA-C8B-H8BE	109.9
H6BA-C6B-H6BC	109.5	C7B-C8B-H8BE	73.1
H6BB-C6B-H6BC	109.5	H8BD-C8B-H8BE	109.5
C8B-C7B-N1B	114.6(6)	C7BA-C8B-H8BF	108.7

C7B-C8B-H8BF	72.1	C23'-N6'-C26'	111.5(4)
H8BD-C8B-H8BF	109.5	C23'-N6'-H6'N	124.2
H8BE-C8B-H8BF	109.5	C26'-N6'-H6'N	124.4
C7BA-C8B-H8BA	176.1	C31'-N7'-C28'	111.8(5)
C7B-C8B-H8BA	110.4	C31'-N7'-H7'N	124.5
H8BD-C8B-H8BA	73.6	C28'-N7'-H7'N	123.8
H8BE-C8B-H8BA	69.9	C35'-N8'-C32'	110.2(4)
H8BF-C8B-H8BA	68.1	C35'-N8'-H8'N	125.0
C7BA-C8B-H8BB	70.9	C32'-N8'-H8'N	124.9
C7B-C8B-H8BB	109.2	C2'-C1'-N1'	106.4(4)
H8BD-C8B-H8BB	68.1	C2'-C1'-C36'	131.6(4)
H8BE-C8B-H8BB	177.6	N1'-C1'-C36'	121.8(4)
H8BF-C8B-H8BB	72.2	C1'-C2'-C3'	108.5(4)
H8BA-C8B-H8BB	109.5	C1'-C2'-H2'	125.7
C7BA-C8B-H8BC	73.7	C3'-C2'-H2'	125.8
C7B-C8B-H8BC	108.7	C4'-C3'-C2'	108.2(4)
H8BD-C8B-H8BC	69.9	C4'-C3'-H3'	125.5
H8BE-C8B-H8BC	68.8	C2'-C3'-H3'	126.3
H8BF-C8B-H8BC	177.5	C3'-C4'-N1'	106.1(4)
H8BA-C8B-H8BC	109.5	C3'-C4'-C5'	129.4(4)
H8BB-C8B-H8BC	109.5	N1'-C4'-C5'	124.4(4)
C1'-N1'-C4'	110.8(4)	C6'-C5'-N2'	106.7(4)
C1'-N1'-H1'N	124.5	C6'-C5'-C4'	129.9(4)
C4'-N1'-H1'N	124.6	N2'-C5'-C4'	123.5(4)
C8'-N2'-C5'	110.7(4)	C5'-C6'-C7'	108.2(4)
C8'-N2'-H2'N	125.0	C5'-C6'-H6'	125.6
C5'-N2'-H2'N	124.3	C7'-C6'-H6'	126.2
C13'-N3'-C10'	111.0(4)	C8'-C7'-C6'	108.3(4)
C13'-N3'-H3'N	124.3	C8'-C7'-H7'	125.3
C10'-N3'-H3'N	124.7	C6'-C7'-H7'	126.4
C14'-N4'-C17'	111.2(4)	C7'-C8'-N2'	106.2(4)
C14'-N4'-H4'N	124.5	C7'-C8'-C9'	131.1(4)
C17'-N4'-H4'N	124.3	N2'-C8'-C9'	122.5(4)
C19'-N5'-C22'	111.2(4)	C10'-C9'-C8'	109.7(4)
C19'-N5'-H5'N	124.1	C10'-C9'-C38'	109.0(4)
C22'-N5'-H5'N	124.7	C8'-C9'-C38'	108.7(4)

C10'-C9'-C37'	110.0(4)	C19'-C20'-C21'	108.7(5)
C8'-C9'-C37'	111.0(4)	C19'-C20'-H20'	125.1
C38'-C9'-C37'	108.4(4)	C21'-C20'-H20'	126.3
C11'-C10'-N3'	105.9(4)	C22'-C21'-C20'	108.5(5)
C11'-C10'-C9'	133.1(4)	C22'-C21'-H21'	125.5
N3'-C10'-C9'	121.0(4)	C20'-C21'-H21'	126.0
C10'-C11'-C12'	108.7(4)	C21'-C22'-N5'	105.6(5)
C10'-C11'-H11'	125.2	C21'-C22'-C23'	131.0(5)
C12'-C11'-H11'	126.1	N5'-C22'-C23'	123.4(4)
C13'-C12'-C11'	108.3(5)	C24'-C23'-N6'	105.9(5)
C13'-C12'-H12'	124.4	C24'-C23'-C22'	130.8(5)
C11'-C12'-H12'	127.3	N6'-C23'-C22'	123.3(5)
N3'-C13'-C12'	106.1(4)	C23'-C24'-C25'	108.4(5)
N3'-C13'-C14'	122.7(4)	C23'-C24'-H24'	125.3
C12'-C13'-C14'	131.2(5)	C25'-C24'-H24'	126.2
C15'-C14'-N4'	106.2(4)	C26'-C25'-C24'	108.9(5)
C15'-C14'-C13'	131.4(5)	C26'-C25'-H25'	124.6
N4'-C14'-C13'	122.4(4)	C24'-C25'-H25'	126.4
C14'-C15'-C16'	108.0(5)	C25'-C26'-N6'	105.3(5)
C14'-C15'-H15'	125.0	C25'-C26'-C27'	132.5(6)
C16'-C15'-H15'	127.0	N6'-C26'-C27'	122.2(4)
C17'-C16'-C15'	109.0(4)	C28'-C27'-C26'	110.8(4)
C17'-C16'-H16'	124.9	C28'-C27'-C41'	110.5(4)
C15'-C16'-H16'	126.1	C26'-C27'-C41'	109.0(4)
C16'-C17'-N4'	105.5(4)	C28'-C27'-C42'	109.3(5)
C16'-C17'-C18'	133.3(4)	C26'-C27'-C42'	109.1(5)
N4'-C17'-C18'	121.3(4)	C41'-C27'-C42'	108.0(4)
C17'-C18'-C19'	110.0(4)	C29'-C28'-N7'	105.1(5)
C17'-C18'-C40'	109.5(4)	C29'-C28'-C27'	132.7(5)
C19'-C18'-C40'	111.4(4)	N7'-C28'-C27'	122.3(5)
C17'-C18'-C39'	108.7(4)	C28'-C29'-C30'	109.7(5)
C19'-C18'-C39'	108.7(4)	C28'-C29'-H29'	124.7
C40'-C18'-C39'	108.4(4)	C30'-C29'-H29'	125.6
C20'-C19'-N5'	106.1(5)	C31'-C30'-C29'	107.9(5)
C20'-C19'-C18'	132.2(5)	C31'-C30'-H30'	125.2
N5'-C19'-C18'	121.7(4)	C29'-C30'-H30'	126.9

N7'-C31'-C30'	105.6(5)	C18'-C39'-H39F	109.7
N7'-C31'-C32'	123.0(5)	H39D-C39'-H39F	109.5
C30'-C31'-C32'	131.4(5)	H39E-C39'-H39F	109.5
C33'-C32'-N8'	105.9(5)	C18'-C40'-H40D	109.3
C33'-C32'-C31'	131.5(5)	C18'-C40'-H40E	109.4
N8'-C32'-C31'	122.6(5)	H40D-C40'-H40E	109.5
C32'-C33'-C34'	108.9(5)	C18'-C40'-H40F	109.7
C32'-C33'-H33'	124.9	H40D-C40'-H40F	109.5
C34'-C33'-H33'	126.2	H40E-C40'-H40F	109.5
C35'-C34'-C33'	107.7(5)	C27'-C41'-H41D	108.9
C35'-C34'-H34'	125.3	C27'-C41'-H41E	109.5
C33'-C34'-H34'	127.0	H41D-C41'-H41E	109.5
C34'-C35'-N8'	107.4(5)	C27'-C41'-H41F	110.0
C34'-C35'-C36'	131.9(5)	H41D-C41'-H41F	109.5
N8'-C35'-C36'	120.4(4)	H41E-C41'-H41F	109.5
C35'-C36'-C1'	108.6(4)	C27'-C42'-H42D	109.9
C35'-C36'-C44'	110.7(4)	C27'-C42'-H42E	109.0
C1'-C36'-C44'	111.2(4)	H42D-C42'-H42E	109.5
C35'-C36'-C43'	108.6(4)	C27'-C42'-H42F	109.5
C1'-C36'-C43'	109.4(4)	H42D-C42'-H42F	109.5
C44'-C36'-C43'	108.3(4)	H42E-C42'-H42F	109.5
C9'-C37'-H37D	109.3	C36'-C43'-H43D	109.4
C9'-C37'-H37E	109.4	C36'-C43'-H43E	109.4
H37D-C37'-H37E	109.5	H43D-C43'-H43E	109.5
C9'-C37'-H37F	109.8	C36'-C43'-H43F	109.7
H37D-C37'-H37F	109.4	H43D-C43'-H43F	109.5
H37E-C37'-H37F	109.5	H43E-C43'-H43F	109.5
C9'-C38'-H38D	108.6	C36'-C44'-H44D	108.9
C9'-C38'-H38E	110.0	C36'-C44'-H44E	110.2
H38D-C38'-H38E	109.5	H44D-C44'-H44E	109.5
C9'-C38'-H38F	109.8	C36'-C44'-H44F	109.3
H38D-C38'-H38F	109.5	H44D-C44'-H44F	109.5
H38E-C38'-H38F	109.5	H44E-C44'-H44F	109.5
C18'-C39'-H39D	108.7	Cl2C-C1C-Cl1C	112.0(3)
C18'-C39'-H39E	110.0	Cl2C-C1C-H1CA	109.6
H39D-C39'-H39E	109.4	Cl1C-C1C-H1CA	109.6

Cl2C-C1C-H1CB	108.7	H4AA-C4A-H4AB	109.5
Cl1C-C1C-H1CB	108.7	C3A-C4A-H4AC	108.4
H1CA-C1C-H1CB	108.2	H4AA-C4A-H4AC	109.5
C5A-N1A-C3A	110.7(4)	H4AB-C4A-H4AC	109.5
C5A-N1A-C7A	112.2(4)	N1A-C5A-C6A	115.6(4)
C3A-N1A-C7A	105.5(4)	N1A-C5A-H5AA	107.8
C5A-N1A-C1A	105.4(4)	C6A-C5A-H5AA	107.8
C3A-N1A-C1A	111.1(4)	N1A-C5A-H5AB	108.6
C7A-N1A-C1A	112.0(4)	C6A-C5A-H5AB	109.0
N1A-C1A-C2A	114.6(5)	H5AA-C5A-H5AB	107.8
N1A-C1A-H1AA	109.0	C5A-C6A-H6AA	110.8
C2A-C1A-H1AA	109.6	C5A-C6A-H6AB	109.4
N1A-C1A-H1AB	107.9	H6AA-C6A-H6AB	109.5
C2A-C1A-H1AB	107.8	C5A-C6A-H6AC	108.2
H1AA-C1A-H1AB	107.7	H6AA-C6A-H6AC	109.5
C1A-C2A-H2AA	109.9	H6AB-C6A-H6AC	109.5
C1A-C2A-H2AB	108.4	C8A-C7A-N1A	115.5(4)
H2AA-C2A-H2AB	109.5	C8A-C7A-H7AA	107.2
C1A-C2A-H2AC	110.1	N1A-C7A-H7AA	108.0
H2AA-C2A-H2AC	109.5	C8A-C7A-H7AB	109.1
H2AB-C2A-H2AC	109.5	N1A-C7A-H7AB	109.0
C4A-C3A-N1A	114.7(4)	H7AA-C7A-H7AB	107.8
C4A-C3A-H3AA	107.8	C7A-C8A-H8AA	111.1
N1A-C3A-H3AA	108.7	C7A-C8A-H8AB	109.6
C4A-C3A-H3AB	109.1	H8AA-C8A-H8AB	109.5
N1A-C3A-H3AB	108.6	C7A-C8A-H8AC	107.7
H3AA-C3A-H3AB	107.7	H8AA-C8A-H8AC	109.5
C3A-C4A-H4AA	110.4	H8AB-C8A-H8AC	109.5
C3A-C4A-H4AB	109.6		

Table 4. Anisotropic displacement parameters ($Å^2 \times 10^3$) for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{1}1 +...+ 2 h k a^* b^* U^{12}]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br1	23(1)	27(1)	28(1)	5(1)	-3(1)	-4(1)
Cl1C	56(1)	37(1)	39(1)	3(1)	2(1)	-4(1)
Cl2C	138(2)	44(1)	38(1)	1(1)	17(1)	-7(1)
N1	35(2)	22(2)	26(3)	11(2)	0(2)	3(2)
N2	33(2)	29(2)	21(2)	5(2)	-10(2)	0(2)
N3	20(2)	29(2)	37(3)	1(2)	-4(2)	-4(2)
N4	17(2)	28(2)	36(3)	7(2)	5(2)	3(2)
N5	33(2)	41(3)	17(2)	-4(2)	-1(2)	9(2)
N6	32(2)	22(2)	22(3)	5(2)	-4(2)	7(2)
N7	16(2)	23(2)	38(3)	1(2)	-2(2)	-2(2)
N8	24(2)	16(2)	41(3)	3(2)	4(2)	-6(2)
C1	24(3)	24(3)	46(4)	1(3)	8(3)	7(2)
C2	39(3)	30(3)	45(4)	20(3)	11(3)	14(3)
C3	44(3)	39(3)	38(4)	18(3)	11(3)	14(3)
C4	25(3)	40(3)	23(3)	10(3)	1(2)	11(3)
C5	22(3)	36(3)	30(3)	7(3)	-4(2)	6(2)
C6	46(3)	49(4)	35(4)	2(3)	-11(3)	2(3)
C7	52(4)	52(4)	31(4)	-9(3)	-21(3)	5(3)
C8	28(3)	41(3)	36(4)	-11(3)	-10(3)	1(3)
C9	38(3)	34(3)	38(4)	-9(3)	-5(3)	-3(3)
C10	34(3)	27(3)	35(4)	-5(3)	-13(3)	-5(2)
C11	28(3)	64(4)	63(5)	-12(4)	-13(3)	-20(3)
C12	28(3)	59(4)	63(5)	4(4)	0(3)	-20(3)
C13	16(3)	29(3)	42(4)	13(3)	-2(3)	-1(2)
C14	18(3)	28(3)	39(4)	11(3)	0(3)	3(2)
C15	18(3)	42(3)	50(4)	14(3)	2(3)	1(3)
C16	22(3)	43(3)	38(4)	14(3)	11(3)	4(2)
C17	26(3)	32(3)	30(3)	6(3)	1(2)	11(2)
C18	33(3)	34(3)	34(3)	-6(3)	4(3)	5(3)

C19	28(3)	33(3)	24(3)	3(3)	-1(2)	0(2)
C20	40(3)	38(3)	24(3)	-1(3)	3(3)	-6(3)
C21	37(3)	45(3)	22(3)	4(3)	-16(3)	-3(3)
C22	20(3)	27(3)	28(3)	10(3)	-8(2)	-1(2)
C23	27(3)	23(3)	23(3)	8(2)	-6(2)	-1(2)
C24	34(3)	26(3)	29(3)	9(2)	-1(3)	2(2)
C25	35(3)	26(3)	38(4)	7(3)	1(3)	6(2)
C26	30(3)	19(3)	34(3)	2(3)	3(3)	3(2)
C27	34(3)	23(3)	32(3)	3(3)	-5(3)	5(2)
C28	30(3)	31(3)	19(3)	2(2)	1(2)	7(3)
C29	28(3)	36(3)	37(4)	3(3)	5(3)	6(3)
C30	21(3)	38(3)	33(3)	-4(3)	2(2)	-2(2)
C31	26(3)	24(3)	23(3)	-3(2)	4(2)	-2(2)
C32	23(3)	22(3)	30(3)	-4(2)	6(2)	-8(2)
C33	32(3)	26(3)	68(4)	1(3)	15(3)	-12(3)
C34	47(4)	23(3)	81(5)	4(3)	21(3)	-5(3)
C35	39(3)	17(3)	40(4)	-2(2)	6(3)	-4(2)
C36	41(3)	14(3)	52(4)	9(3)	9(3)	6(2)
C37	57(4)	33(3)	61(4)	-11(3)	-20(3)	-8(3)
C38	35(3)	36(3)	40(4)	-6(3)	-7(3)	7(3)
C39	43(3)	27(3)	57(4)	2(3)	-7(3)	6(3)
C40	50(4)	62(4)	51(4)	-15(3)	7(3)	15(3)
C41	53(3)	24(3)	41(4)	0(3)	-5(3)	0(3)
C42	64(4)	35(3)	52(4)	14(3)	13(3)	21(3)
C43	45(3)	33(3)	43(4)	-5(3)	12(3)	4(3)
C44	62(4)	18(3)	83(5)	7(3)	8(3)	7(3)
N1B	23(2)	61(3)	38(3)	13(2)	15(2)	14(2)
C1B	46(4)	40(4)	52(5)	8(4)	6(4)	7(4)
C1BA	39(7)	38(7)	43(7)	1(6)	2(6)	9(6)
C2B	35(3)	96(4)	76(4)	-8(4)	2(3)	-2(3)
C3B	48(4)	46(4)	43(5)	0(4)	12(4)	0(4)
C3BA	24(6)	25(6)	20(6)	2(5)	-5(5)	6(5)
C4B	45(3)	62(4)	94(5)	21(4)	-11(3)	-2(3)
C5B	41(4)	46(4)	50(5)	1(4)	10(4)	3(4)
C5BA	43(7)	47(8)	57(8)	23(6)	-3(6)	-3(6)
C6B	63(4)	69(4)	94(5)	-5(4)	15(3)	14(3)

C7B	45(4)	75(5)	47(5)	19(4)	-3(4)	2(4)
C7BA	54(8)	74(9)	64(9)	4(6)	-3(6)	4(6)
C8B	112(5)	141(6)	90(5)	-14(5)	29(4)	29(4)
Br2	23(1)	31(1)	29(1)	2(1)	2(1)	-5(1)
N1'	23(2)	22(2)	33(3)	-3(2)	6(2)	-7(2)
N2'	16(2)	24(2)	30(3)	2(2)	5(2)	-3(2)
N3'	26(2)	38(2)	21(2)	-3(2)	0(2)	5(2)
N4'	20(2)	49(3)	30(3)	-1(2)	-8(2)	3(2)
N5'	18(2)	51(3)	36(3)	-10(2)	3(2)	-10(2)
N6'	35(3)	45(3)	40(3)	-5(2)	12(2)	-17(2)
N7'	39(3)	32(3)	34(3)	11(2)	5(2)	1(2)
N8'	30(2)	38(3)	22(3)	0(2)	-1(2)	-5(2)
C1'	29(3)	29(3)	24(3)	-5(2)	4(2)	3(2)
C2'	34(3)	30(3)	34(3)	-7(3)	-1(3)	-12(3)
C3'	25(3)	36(3)	39(4)	-7(3)	6(3)	-6(2)
C4'	22(3)	25(3)	25(3)	-1(2)	1(2)	-3(2)
C5'	18(3)	23(3)	25(3)	3(2)	-2(2)	-4(2)
C6'	27(3)	36(3)	35(3)	-10(3)	5(3)	-11(2)
C7'	18(3)	50(3)	39(4)	-2(3)	7(2)	-4(3)
C8'	19(3)	28(3)	30(3)	-4(2)	6(2)	7(2)
C9'	24(3)	32(3)	32(3)	-8(3)	2(2)	2(2)
C10'	20(3)	37(3)	35(4)	-12(3)	6(2)	2(2)
C11'	29(3)	93(5)	24(4)	-9(3)	8(3)	17(3)
C12'	44(4)	93(5)	16(3)	-4(3)	0(3)	10(3)
C13'	26(3)	43(3)	23(3)	-4(3)	-6(3)	3(2)
C14'	33(3)	41(3)	21(3)	-5(3)	0(3)	6(3)
C15'	49(4)	67(4)	26(3)	4(3)	-8(3)	22(3)
C16'	38(3)	67(4)	40(4)	-11(3)	-13(3)	30(3)
C17'	28(3)	40(3)	32(4)	-1(3)	-8(3)	7(3)
C18'	22(3)	56(4)	36(4)	-9(3)	-5(3)	8(3)
C19'	22(3)	60(4)	29(3)	-14(3)	-3(3)	3(3)
C20'	22(3)	84(5)	55(4)	-11(4)	-1(3)	0(3)
C21'	23(3)	81(5)	54(4)	-16(4)	1(3)	-20(3)
C22'	29(3)	55(4)	27(3)	-7(3)	3(2)	-16(3)
C23'	35(3)	47(4)	35(4)	-7(3)	12(3)	-21(3)
C24'	45(4)	80(5)	56(4)	1(4)	8(3)	-31(4)

C25'	67(4)	47(4)	59(5)	1(3)	13(4)	-24(4)
C26'	55(4)	35(3)	44(4)	0(3)	22(3)	-21(3)
C27'	50(4)	32(3)	47(4)	9(3)	21(3)	1(3)
C28'	52(4)	32(3)	35(4)	3(3)	11(3)	-7(3)
C29'	78(5)	40(4)	55(5)	17(4)	4(4)	-8(3)
C30'	72(4)	59(4)	33(4)	17(4)	7(3)	-9(4)
C31'	31(3)	46(4)	23(3)	2(3)	3(2)	0(3)
C32'	25(3)	39(3)	24(3)	5(3)	8(2)	5(3)
C33'	31(3)	55(4)	29(4)	1(3)	1(3)	16(3)
C34'	33(3)	55(4)	29(4)	-13(3)	0(3)	9(3)
C35'	23(3)	36(3)	29(3)	-8(3)	6(2)	0(2)
C36'	26(3)	31(3)	27(3)	-7(3)	1(2)	-2(2)
C37'	34(3)	33(3)	49(4)	-9(3)	-1(3)	2(2)
C38'	36(3)	50(4)	45(4)	-20(3)	6(3)	3(3)
C39'	39(3)	72(4)	61(5)	-8(4)	0(3)	21(3)
C40'	38(3)	45(3)	34(3)	-6(3)	3(3)	10(3)
C41'	64(4)	34(3)	51(4)	7(3)	22(3)	-2(3)
C42'	92(5)	27(3)	69(5)	9(3)	34(4)	-6(3)
C43'	54(4)	43(3)	45(4)	-14(3)	9(3)	0(3)
C44'	35(3)	33(3)	39(4)	-1(3)	4(3)	7(2)
C1C	32(3)	29(3)	44(3)	1(3)	3(2)	4(3)
N1A	42(3)	38(3)	47(3)	21(2)	-9(2)	-8(2)
C1A	60(4)	73(5)	64(5)	50(4)	8(4)	-11(4)
C2A	98(6)	106(6)	79(6)	37(5)	36(5)	42(5)
C3A	44(3)	20(3)	51(4)	3(3)	11(3)	-9(3)
C4A	39(3)	50(4)	73(5)	7(3)	-11(3)	-9(3)
C5A	66(4)	24(3)	84(5)	10(3)	-31(4)	-6(3)
C6A	156(7)	46(4)	68(5)	-12(4)	-23(5)	31(4)
C7A	56(4)	55(4)	52(4)	23(3)	-15(3)	0(3)
C8A	87(6)	109(7)	177(9)	59(6)	-85(6)	-36(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2 \times 10^3$) for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.

	x	y	z	U(eq)
H1N	10667	1720	1626	33
H2N	10948	3081	1816	33
H3N	11428	3788	1181	35
H4N	11433	2963	534	32
H5N	10542	3298	116	37
H6N	9643	3749	484	31
H7N	9176	3018	1134	31
H8N	9603	1723	1252	32
H2	10373	-245	2429	46
H3	10897	920	2900	48
H6	11601	2390	3040	52
H7	11893	3929	2896	55
H11	12787	5087	1780	62
H12	13180	4508	1034	60
H15	13061	3876	148	44
H16	12585	3026	-504	41
H20	10654	2633	-1179	41
H21	9705	3578	-1090	42
H24	9010	4869	-647	35
H25	8340	5320	14	40
H29	7436	3700	875	40
H30	7453	2125	1067	37
H33	7971	598	1371	50
H34	8831	-484	1530	60
H37A	12149	5143	2439	76
H37B	11791	5828	2149	76
H37C	11465	5389	2559	76
H38A	10706	4453	1604	55

H38B	10575	4970	2047	55
H38C	10901	5409	1637	55
H39A	10941	1790	251	64
H39B	10831	1200	-173	64
H39C	11478	1200	86	64
H40A	11804	2301	-941	81
H40B	12008	1515	-648	81
H40C	11362	1515	-906	81
H41A	9510	4315	1229	59
H41B	9354	5283	1195	59
H41C	9014	4712	1549	59
H42A	7829	5100	717	75
H42B	7981	5200	1239	75
H42C	8320	5771	885	75
H43A	10507	959	991	60
H43B	10900	135	1072	60
H43C	10282	98	779	60
H44A	9865	-928	1810	81
H44B	9891	-1048	1281	81
H44C	10510	-1012	1574	81
H1BA	8297	2106	5098	55
H1BB	8478	1810	4616	55
H1BC	7331	2877	4462	48
H1BD	7447	2884	4984	48
H2BD	7123	1595	4761	104
H2BE	7686	1531	4433	104
H2BF	7804	1538	4961	104
H2BA	7553	1180	4820	104
H2BB	7250	2073	4887	104
H2BC	7434	1773	4399	104
H3BA	8986	3870	4549	55
H3BB	9175	2935	4477	55
H3BC	8823	2212	4952	27
H3BD	8449	2738	5295	27
H4BD	9491	3050	5357	101
H4BE	9500	3319	4844	101

H4BF	9120	3852	5192	101
H4BA	9678	3459	5090	101
H4BB	9037	3677	5306	101
H4BC	9228	2729	5233	101
H5BA	8025	3464	5275	55
H5BB	7460	3486	4938	55
H5BC	7724	3959	4344	59
H5BD	8422	4203	4359	59
H6BD	7861	5082	4821	113
H6BE	7629	4299	5098	113
H6BF	8336	4547	5114	113
H6BA	7726	4815	5148	113
H6BB	8398	4665	4966	113
H6BC	7826	4688	4626	113
H7BA	8185	3828	4089	67
H7BB	7598	3269	4142	67
H7BC	8370	1911	4323	77
H7BD	8943	2493	4267	77
H8BD	8448	2289	3589	171
H8BE	7832	2656	3786	171
H8BF	8412	3245	3729	171
H8BA	8130	2857	3506	171
H8BB	8725	2687	3811	171
H8BC	8132	2121	3864	171
H1'N	6471	4909	3969	31
H2'N	6807	4024	3384	28
H3'N	6006	3763	2814	34
H4'N	5103	4360	2980	40
H5'N	4549	3702	3600	42
H6'N	5009	2551	3931	48
H7'N	5865	2802	4429	42
H8'N	5860	4240	4578	36
H2'	7317	6873	4553	39
H3'	7999	6320	3924	40
H6'	8308	5420	3177	39
H7'	8253	4253	2576	42

H11'	6849	3442	1643	58
H12'	5790	4118	1490	61
H15'	4609	4738	1690	57
H16'	3731	5244	2195	58
H20'	2776	4177	3300	64
H21'	2853	2706	3649	63
H24'	3432	1279	4024	72
H25'	4318	357	4328	69
H29'	5825	899	5292	69
H30'	6101	2230	5731	66
H33'	6646	3883	5798	46
H34'	6669	5460	5613	47
H37D	6606	2667	3133	58
H37E	6741	1986	2759	58
H37F	7259	2225	3121	58
H38D	7799	3202	2111	65
H38E	7993	2552	2492	65
H38F	7476	2313	2130	65
H39D	3132	5381	2887	86
H39E	3218	5778	3373	86
H39F	3565	6171	2960	86
H40D	4786	5128	3577	59
H40E	4581	6012	3383	59
H40F	4234	5619	3796	59
H41D	5975	1897	3831	74
H41E	6415	1159	3994	74
H41F	5861	973	3652	74
H42D	5340	65	4688	93
H42E	5473	-140	4177	93
H42F	6027	45	4519	93
H43D	6582	6709	5168	71
H43E	6159	7189	4812	71
H43F	5857	6688	5211	71
H44D	5512	5386	4252	53
H44E	5198	5877	4650	53
H44F	5501	6379	4251	53

H1CA	9664	1692	2319	42
H1CB	8954	1890	2306	42
H1AA	6320	6650	3441	79
H1AB	5666	6510	3220	79
H2AA	5602	7601	3753	141
H2AB	6109	8108	3491	141
H2AC	5448	7966	3267	141
H3AA	6659	8179	2861	46
H3AB	6975	7669	2478	46
H4AA	7658	7828	3095	81
H4AB	7178	7383	3410	81
H4AC	7497	6867	3024	81
H5AA	6133	5773	2618	70
H5AB	6768	5922	2867	70
H6AA	6931	5514	2111	136
H6AB	6533	6288	1941	136
H6AC	7175	6439	2192	136
H7AA	5988	7405	2142	66
H7AB	5698	7941	2526	66
H8AA	4932	7136	2156	188
H8AB	5304	6310	2277	188
H8AC	5011	6851	2665	188

Table 6. Torsion angles [°] for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2.

C4-N1-C1-C2	-0.2(5)	C37-C9-C10-C11	15.6(8)
C4-N1-C1-C36	178.5(4)	C38-C9-C10-C11	134.9(6)
N1-C1-C2-C3	0.4(5)	N3-C10-C11-C12	0.4(6)
C36-C1-C2-C3	-178.2(4)	C9-C10-C11-C12	177.1(5)
C1-C2-C3-C4	-0.5(5)	C10-C11-C12-C13	-0.2(6)
C2-C3-C4-N1	0.3(5)	C11-C12-C13-N3	-0.1(6)
C2-C3-C4-C5	179.6(4)	C11-C12-C13-C14	176.0(5)
C1-N1-C4-C3	-0.1(5)	C10-N3-C13-C12	0.3(5)
C1-N1-C4-C5	-179.4(4)	C10-N3-C13-C14	-176.2(4)
C8-N2-C5-C6	-0.7(5)	C17-N4-C14-C15	-0.4(5)
C8-N2-C5-C4	179.7(4)	C17-N4-C14-C13	177.5(4)
C3-C4-C5-N2	-168.7(5)	C12-C13-C14-N4	174.7(5)
N1-C4-C5-N2	10.4(7)	N3-C13-C14-N4	-9.8(7)
C3-C4-C5-C6	11.7(8)	C12-C13-C14-C15	-7.9(9)
N1-C4-C5-C6	-169.1(5)	N3-C13-C14-C15	167.6(4)
N2-C5-C6-C7	0.7(5)	N4-C14-C15-C16	0.6(5)
C4-C5-C6-C7	-179.7(5)	C13-C14-C15-C16	-177.1(4)
C5-C6-C7-C8	-0.5(6)	C14-C15-C16-C17	-0.6(5)
C5-N2-C8-C7	0.4(5)	C15-C16-C17-N4	0.4(5)
C5-N2-C8-C9	178.1(4)	C15-C16-C17-C18	179.2(5)
C6-C7-C8-N2	0.1(6)	C14-N4-C17-C16	0.0(5)
C6-C7-C8-C9	-177.4(5)	C14-N4-C17-C18	-179.0(4)
N2-C8-C9-C10	-70.9(5)	C16-C17-C18-C19	-102.8(6)
C7-C8-C9-C10	106.3(6)	N4-C17-C18-C19	75.8(5)
N2-C8-C9-C37	170.0(4)	C16-C17-C18-C40	16.5(7)
C7-C8-C9-C37	-12.8(8)	N4-C17-C18-C40	-164.9(4)
N2-C8-C9-C38	50.3(6)	C16-C17-C18-C39	135.6(5)
C7-C8-C9-C38	-132.6(5)	N4-C17-C18-C39	-45.7(6)
C13-N3-C10-C11	-0.4(5)	C22-N5-C19-C20	-0.9(5)
C13-N3-C10-C9	-177.5(4)	C22-N5-C19-C18	-176.8(4)
C8-C9-C10-N3	72.7(6)	C17-C18-C19-C20	127.1(5)
C37-C9-C10-N3	-168.2(4)	C40-C18-C19-C20	7.7(7)
C38-C9-C10-N3	-48.8(6)	C39-C18-C19-C20	-110.5(6)
C8-C9-C10-C11	-103.5(6)	C17-C18-C19-N5	-58.2(6)

C40-C18-C19-N5	-177.6(4)	N7-C28-C29-C30	0.8(5)
C39-C18-C19-N5	64.1(5)	C27-C28-C29-C30	176.8(5)
N5-C19-C20-C21	0.2(5)	C28-C29-C30-C31	0.2(6)
C18-C19-C20-C21	175.5(5)	C29-C30-C31-N7	-1.1(5)
C19-C20-C21-C22	0.6(6)	C29-C30-C31-C32	-179.2(5)
C20-C21-C22-N5	-1.1(5)	C28-N7-C31-C30	1.6(5)
C20-C21-C22-C23	-177.7(4)	C28-N7-C31-C32	179.9(4)
C19-N5-C22-C21	1.3(5)	C35-N8-C32-C33	0.3(6)
C19-N5-C22-C23	178.2(4)	C35-N8-C32-C31	-178.3(4)
C26-N6-C23-C24	0.5(5)	C30-C31-C32-C33	-6.8(9)
C26-N6-C23-C22	-173.2(4)	N7-C31-C32-C33	175.4(5)
C21-C22-C23-C24	-17.0(8)	C30-C31-C32-N8	171.4(5)
N5-C22-C23-C24	166.9(4)	N7-C31-C32-N8	-6.4(7)
C21-C22-C23-N6	155.0(5)	N8-C32-C33-C34	0.0(6)
N5-C22-C23-N6	-21.2(7)	C31-C32-C33-C34	178.5(5)
N6-C23-C24-C25	-0.3(5)	C32-C33-C34-C35	-0.4(7)
C22-C23-C24-C25	172.6(4)	C33-C34-C35-N8	0.6(6)
C23-C24-C25-C26	0.1(5)	C33-C34-C35-C36	178.2(5)
C24-C25-C26-N6	0.2(5)	C32-N8-C35-C34	-0.6(6)
C24-C25-C26-C27	-175.7(5)	C32-N8-C35-C36	-178.4(4)
C23-N6-C26-C25	-0.4(5)	C2-C1-C36-C35	98.2(6)
C23-N6-C26-C27	176.0(4)	N1-C1-C36-C35	-80.3(5)
C25-C26-C27-C28	107.0(6)	C2-C1-C36-C44	-20.7(7)
N6-C26-C27-C28	-68.4(5)	N1-C1-C36-C44	160.9(4)
C25-C26-C27-C41	-130.9(5)	C2-C1-C36-C43	-139.7(5)
N6-C26-C27-C41	53.7(6)	N1-C1-C36-C43	41.9(6)
C25-C26-C27-C42	-10.7(7)	C34-C35-C36-C1	-111.2(6)
N6-C26-C27-C42	173.9(4)	N8-C35-C36-C1	66.0(6)
C31-N7-C28-C29	-1.5(5)	C34-C35-C36-C44	7.9(8)
C31-N7-C28-C27	-178.1(4)	N8-C35-C36-C44	-174.9(4)
C26-C27-C28-C29	-94.9(6)	C34-C35-C36-C43	125.9(6)
C41-C27-C28-C29	142.1(5)	N8-C35-C36-C43	-56.9(6)
C42-C27-C28-C29	23.0(7)	C4'-N1'-C1'-C2'	0.6(5)
C26-C27-C28-N7	80.6(5)	C4'-N1'-C1'-C36'	-175.0(4)
C41-C27-C28-N7	-42.4(6)	N1'-C1'-C2'-C3'	-0.3(5)
C42-C27-C28-N7	-161.5(4)	C36'-C1'-C2'-C3'	174.8(5)

C1'-C2'-C3'-C4'	-0.2(6)	C10'-N3'-C13'-C14'	-178.5(4)
C2'-C3'-C4'-N1'	0.6(5)	C11'-C12'-C13'-N3'	0.0(6)
C2'-C3'-C4'-C5'	177.0(4)	C11'-C12'-C13'-C14'	178.3(5)
C1'-N1'-C4'-C3'	-0.8(5)	C17'-N4'-C14'-C15'	-0.8(6)
C1'-N1'-C4'-C5'	-177.4(4)	C17'-N4'-C14'-C13'	179.4(4)
C8'-N2'-C5'-C6'	-0.1(5)	N3'-C13'-C14'-C15'	-177.8(5)
C8'-N2'-C5'-C4'	179.2(4)	C12'-C13'-C14'-C15'	4.1(10)
C3'-C4'-C5'-C6'	-2.3(8)	N3'-C13'-C14'-N4'	2.0(7)
N1'-C4'-C5'-C6'	173.5(5)	C12'-C13'-C14'-N4'	-176.1(5)
C3'-C4'-C5'-N2'	178.5(4)	N4'-C14'-C15'-C16'	0.2(6)
N1'-C4'-C5'-N2'	-5.7(7)	C13'-C14'-C15'-C16'	-179.9(5)
N2'-C5'-C6'-C7'	0.1(5)	C14'-C15'-C16'-C17'	0.3(7)
C4'-C5'-C6'-C7'	-179.2(4)	C15'-C16'-C17'-N4'	-0.8(6)
C5'-C6'-C7'-C8'	-0.1(6)	C15'-C16'-C17'-C18'	-179.7(5)
C6'-C7'-C8'-N2'	0.0(5)	C14'-N4'-C17'-C16'	1.0(6)
C6'-C7'-C8'-C9'	175.2(5)	C14'-N4'-C17'-C18'	180.0(4)
C5'-N2'-C8'-C7'	0.0(5)	C16'-C17'-C18'-C19'	104.0(7)
C5'-N2'-C8'-C9'	-175.6(4)	N4'-C17'-C18'-C19'	-74.7(6)
C7'-C8'-C9'-C10'	-99.1(6)	C16'-C17'-C18'-C40'	-133.3(6)
N2'-C8'-C9'-C10'	75.4(5)	N4'-C17'-C18'-C40'	47.9(6)
C7'-C8'-C9'-C38'	20.0(7)	C16'-C17'-C18'-C39'	-15.0(8)
N2'-C8'-C9'-C38'	-165.5(4)	N4'-C17'-C18'-C39'	166.3(4)
C7'-C8'-C9'-C37'	139.2(5)	C22'-N5'-C19'-C20'	0.5(6)
N2'-C8'-C9'-C37'	-46.4(6)	C22'-N5'-C19'-C18'	-177.2(4)
C13'-N3'-C10'-C11'	0.0(6)	C17'-C18'-C19'-C20'	-104.3(6)
C13'-N3'-C10'-C9'	177.4(4)	C40'-C18'-C19'-C20'	134.1(6)
C8'-C9'-C10'-C11'	108.4(6)	C39'-C18'-C19'-C20'	14.7(8)
C38'-C9'-C10'-C11'	-10.5(8)	C17'-C18'-C19'-N5'	72.7(6)
C37'-C9'-C10'-C11'	-129.2(6)	C40'-C18'-C19'-N5'	-48.9(6)
C8'-C9'-C10'-N3'	-68.1(5)	C39'-C18'-C19'-N5'	-168.3(4)
C38'-C9'-C10'-N3'	173.0(4)	N5'-C19'-C20'-C21'	-0.9(6)
C37'-C9'-C10'-N3'	54.2(6)	C18'-C19'-C20'-C21'	176.5(5)
N3'-C10'-C11'-C12'	-0.1(6)	C19'-C20'-C21'-C22'	1.0(7)
C9'-C10'-C11'-C12'	-177.0(5)	C20'-C21'-C22'-N5'	-0.6(6)
C10'-C11'-C12'-C13'	0.1(7)	C20'-C21'-C22'-C23'	-178.8(5)
C10'-N3'-C13'-C12'	0.0(6)	C19'-N5'-C22'-C21'	0.0(5)

C19'-N5'-C22'-C23'	178.4(4)	C28'-C29'-C30'-C31'	-1.2(7)
C26'-N6'-C23'-C24'	-0.6(6)	C28'-N7'-C31'-C30'	0.2(6)
C26'-N6'-C23'-C22'	-178.9(5)	C28'-N7'-C31'-C32'	179.8(4)
C21'-C22'-C23'-C24'	1.1(10)	C29'-C30'-C31'-N7'	0.6(6)
N5'-C22'-C23'-C24'	-176.9(5)	C29'-C30'-C31'-C32'	-179.0(5)
C21'-C22'-C23'-N6'	178.9(5)	C35'-N8'-C32'-C33'	-0.4(5)
N5'-C22'-C23'-N6'	1.0(8)	C35'-N8'-C32'-C31'	177.8(4)
N6'-C23'-C24'-C25'	0.2(6)	N7'-C31'-C32'-C33'	-164.7(5)
C22'-C23'-C24'-C25'	178.3(5)	C30'-C31'-C32'-C33'	14.7(9)
C23'-C24'-C25'-C26'	0.3(7)	N7'-C31'-C32'-N8'	17.5(7)
C24'-C25'-C26'-N6'	-0.6(6)	C30'-C31'-C32'-N8'	-163.1(5)
C24'-C25'-C26'-C27'	176.8(6)	N8'-C32'-C33'-C34'	0.7(5)
C23'-N6'-C26'-C25'	0.8(6)	C31'-C32'-C33'-C34'	-177.4(4)
C23'-N6'-C26'-C27'	-177.0(5)	C32'-C33'-C34'-C35'	-0.7(5)
C25'-C26'-C27'-C28'	-108.5(7)	C33'-C34'-C35'-N8'	0.4(5)
N6'-C26'-C27'-C28'	68.6(6)	C33'-C34'-C35'-C36'	-173.7(4)
C25'-C26'-C27'-C41'	129.7(6)	C32'-N8'-C35'-C34'	0.0(5)
N6'-C26'-C27'-C41'	-53.2(6)	C32'-N8'-C35'-C36'	174.9(4)
C25'-C26'-C27'-C42'	11.9(8)	C34'-C35'-C36'-C1'	93.8(6)
N6'-C26'-C27'-C42'	-171.0(4)	N8'-C35'-C36'-C1'	-79.7(5)
C31'-N7'-C28'-C29'	-0.9(6)	C34'-C35'-C36'-C44'	-143.8(5)
C31'-N7'-C28'-C27'	177.7(4)	N8'-C35'-C36'-C44'	42.7(6)
C26'-C27'-C28'-C29'	106.0(7)	C34'-C35'-C36'-C43'	-25.1(7)
C41'-C27'-C28'-C29'	-133.0(6)	N8'-C35'-C36'-C43'	161.4(4)
C42'-C27'-C28'-C29'	-14.3(8)	C2'-C1'-C36'-C35'	-106.1(6)
C26'-C27'-C28'-N7'	-72.2(6)	N1'-C1'-C36'-C35'	68.4(5)
C41'-C27'-C28'-N7'	48.8(6)	C2'-C1'-C36'-C44'	131.8(5)
C42'-C27'-C28'-N7'	167.5(4)	N1'-C1'-C36'-C44'	-53.7(6)
N7'-C28'-C29'-C30'	1.2(6)	C2'-C1'-C36'-C43'	12.3(7)
C27'-C28'-C29'-C30'	-177.2(5)	N1'-C1'-C36'-C43'	-173.2(4)

Table 7. Hydrogen bonds for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N1-H1N...Br1	0.90	2.62	3.492(4)	163.6
N2-H2N...Br1	0.90	2.61	3.511(4)	174.6
N3-H3N...Br1	0.90	2.62	3.511(4)	171.4
N4-H4N...Br1	0.90	2.60	3.493(4)	170.5
N5-H5N...Br1	0.90	2.72	3.558(4)	154.7
N6-H6N...Br1	0.90	2.65	3.542(4)	174.2
N7-H7N...Br1	0.90	2.73	3.608(3)	164.6
N8-H8N...Br1	0.90	2.66	3.552(3)	173.6
N1'-H1'N...Br2	0.90	2.55	3.450(4)	172.9
N2'-H2'N...Br2	0.90	2.54	3.430(3)	169.0
N3'-H3'N...Br2	0.90	2.66	3.554(4)	177.2
N4'-H4'N...Br2	0.90	2.64	3.536(4)	174.2
N5'-H5'N...Br2	0.90	2.54	3.438(4)	175.0
N6'-H6'N...Br2	0.90	2.55	3.448(4)	174.1
N7'-H7'N...Br2	0.90	2.65	3.545(4)	175.8
N8'-H8'N...Br2	0.90	2.73	3.583(4)	158.5

Figure 1. View of the macrocycle-Br complex 1 in 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2 showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The methyl hydrogen atoms have been removed for clarity. Dashed lines are indicative of N-H···Br H-bonding interactions. The N···Br contacts range from 3.492(4)Å for N1 to 3.608(3)Å for N7. A full listing of these interactions are found in Table 7.

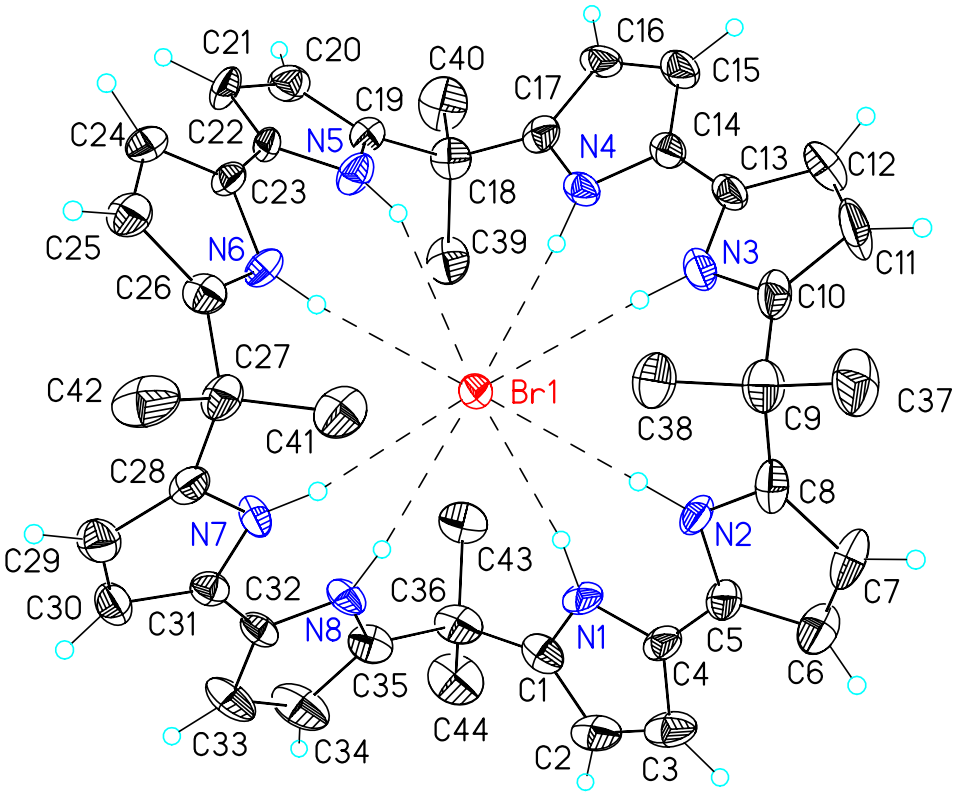


Figure 2. View of the macrocycle-Br complex 2 in 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2 showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The methyl hydrogen atoms have been removed for clarity. Dashed lines are indicative of N-H···Br H-bonding interactions. The N···Br contacts range from 3.430(3)Å for N2' to 3.583(4)Å for N8'. A full listing of these interactions are found in Table 7.

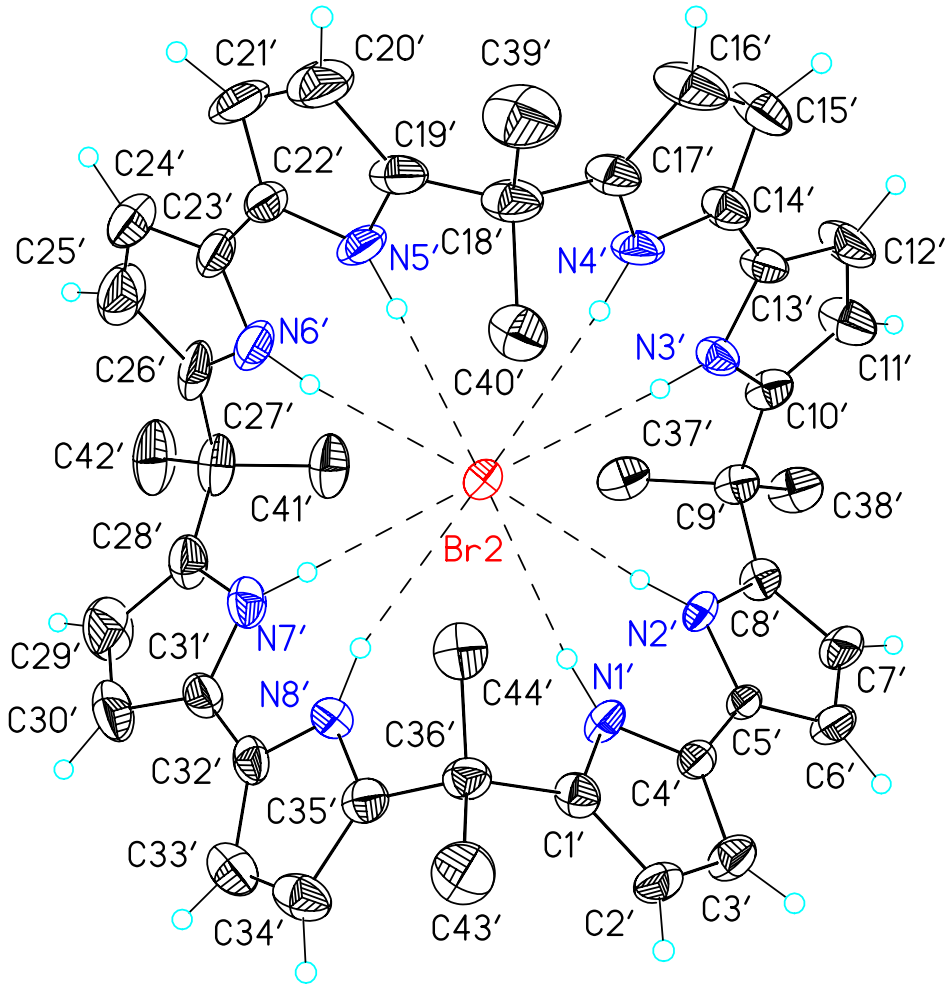


Figure 3. View of one cation with atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level.

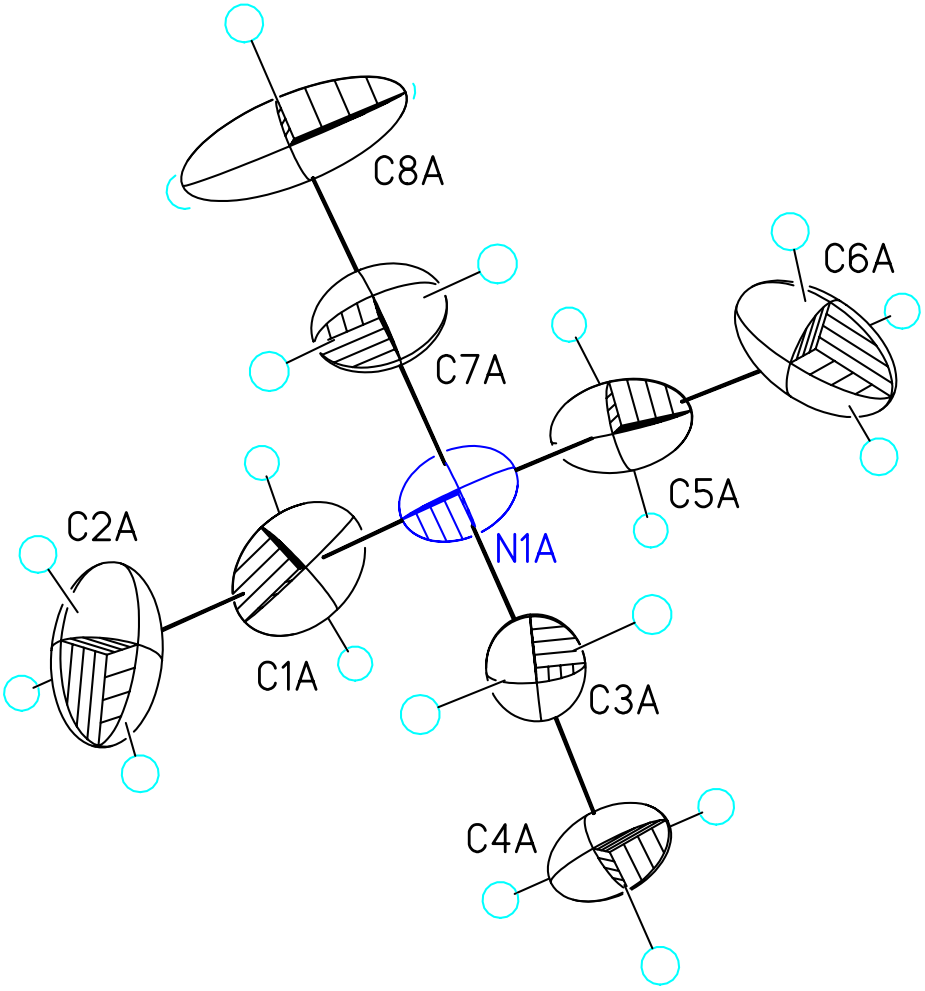


Figure 4. View of one cation with atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The methylene carbon atoms are disordered. Atoms represented by open ellipsoids have site occupancy factors of 40%.

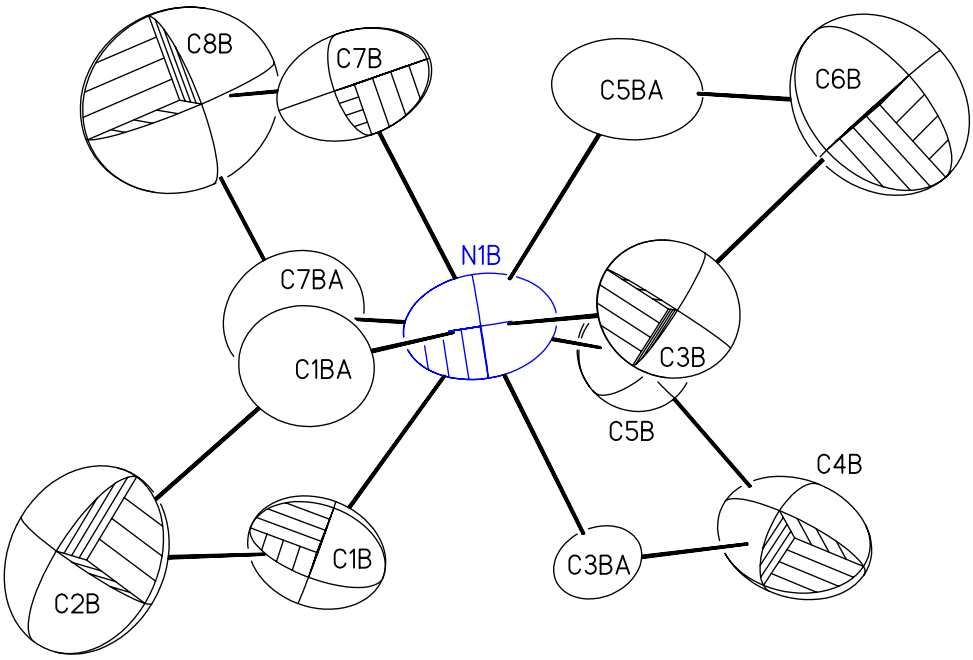


Figure 5. Unit cell packing diagram for 2[**3**·$C_8H_{20}NBr$]·CH_2Cl_2. The view is approximately down the **c** axis. Macrocycle 2 is displayed in wireframe form (near x = ½) while macrocycle 1 is shown in ball-and-stick form.