Supplementary materials

General:

^{1}H and ^{13}C NMR spectra were measured on a Varian Unity Innova at 500 MHz, or on a Bruker AC at 250 MHz. UV/Vis spectra were recorded on a Beckman DU-650 spectrophotometer. Low resolution CI mass spectra were obtained on a Finnigan MATTSQ 700 mass spectrometer. High resolution CI mass spectra were obtained on a VGZAB2-E mass spectrometer. All solvents and chemicals were obtained commercially and used as received. UV-Vis titration experiments were carried out in spectrophotometric grade acetone. The tetrabutylammonium anions were purchased from Fluka and dried overnight under vacuum at 40° C prior to use.

Experimentals:

Synthesis and characterization of 1 and 5:

A solution of tripyrrane **2** (5.33 mmoles) in dry dichloromethane (200 ml) was degassed by bubling with argon for 10 minutes. Pentafluorobenzaldehyde (5.90 mmoles) was then added and the reaction mixture was stirred under argon for 10-12 hours. DDQ (5.33 mmoles) was simply added to the solution that was then stirred for another 2 hours. After washing with aqueous saturated sodium bicarbonate and drying with brine, the organic phase was dried over anhydrous $MgSO_4$, filtered and evaporated under reduced vacuum. **1** and **5** were then isolated (in 10 and < 1 % yield, respectively) through silica gel chromatography using hexane as the eluent with an increasing amount of ethyl acetate (0 to 10 %).

1 : ^{1}H NMR ($CDCl_3$, 25°C) : δ = 1.67 (bs, H_2O); 1.71 (s, 24 H, -Me); 5.32 (s, CH_2Cl_2); 5.98 (d, $^{4}J_{\beta H\text{-}NH}$ =2.5 Hz, 4H, βH), 6.12 (d, $^{3}J_{\beta H\text{-}\beta H}$ = 4 Hz, 4H, βH), 6.23 (d, $^{3}J_{\beta H\text{-}\beta H}$ = 4 Hz, 4H, βH), 7.97 (bs, NH), 12.34 (bs, NH).

^{1}H NMR (CD_2Cl_2, -80°C) : δ = 1.60 (s, 12 H, -Me) ; 1.80 (s, 12 H, -Me); 3.70 (bs, H_2O), 5.33 (s, CH_2Cl_2), 5.94 (bs, 4H, βH); 6.15 (bs, 4H, βH); 6.27 (bs, 4H, βH); 9.35 (bs, 2H, NH); 12.45 (s, 2H, NH)

^{13}C NMR ($CDCl_3$, 25°C) : δ = 27.1 (-**C**H_3), 37.0 (-$(Me)_2$**C**-), 102.8 (βH), 116.1 (βH), 126.8 (βH), 137.8, 138.8, 164.9.

^{19}F NMR ($CDCl_3$, 25°C) : -162.6 (bs, 4F), -153.4 (t, *J* = 21 Hz, 2F), -138.6 (d, *J* = 21 Hz, 4F).

UV /Vis (CH_3COCH_3): λ_{max} (ε) = 449 (52, 562)

After addition of 90 molar equivalents of concentrated H_2SO_4, the extinction coefficient of the diprotonated form was calculated assuming total conversion of the free base: 499 (76, 273)

HR-MS (CI) for $C_{50}H_{41}N_6$ ($[M+H]^+$) calcd : 915.3233 ; found : 915.3231

Elemental analysis calculated for $C_{50}H_{40}N_6 \cdot H_2O \cdot CH_2Cl_2$: C, 60.18, H, 4.36, N, 8.26, F, 18.67 (%). Found : C, 60.47, H, 4.47, N, 8.22, F, 18.46 (%).

5 : ^{1}H NMR ($CDCl_3$, 25°C) : δ = 1.58 (s, 36 H, -Me) ; 5.86 (d, $^{2}J_{\beta\mathbf{H}\text{-}\beta\mathbf{H}}$ = 4.5 Hz, 6H), 5.99 (d, $^{4}J_{\beta\mathbf{H}\text{-}\mathrm{N}\mathbf{H}}$ = 3 Hz, 6H); 6.12 (d, $^{2}J_{\beta\mathbf{H}\text{-}\beta\mathbf{H}}$ = 4.5 Hz, 6H); 7.46 (bs, 3H, NH); 12.62 (bs, 3H, NH).

^{13}C NMR (250 MHz, $CDCl_3$, 25°C) : δ = 27.4 (-**C**H_3); 37.2 (-$(Me)_2$**C**-); 103.7 (βH); 116.6 (βH); 126.3(βH); 137.3; 139.1; 165.4.

^{19}F NMR ($CDCl_3$, 25°C) : -162.1 (t, *J* = 19 Hz, 6F), -153.4 (t, *J* = 21 Hz, 3F), -139.5 (d, *J* = 17 Hz, 6F).

UV /Vis (CH_2Cl_2): λ_{max} (ε) = 444 (46, 215)

HR-MS (CI) for $C_{75}H_{61}N_9$ ($[M+H]^+$) calcd : 1372.4810 ; found : 1372.4841

Synthesis and characterization of 2 and 3:

Acetone (1 equiv.) and pyrrole (2.5 equiv.) were mixed at room temperature under nitrogen atmosphere. After TFA (0.025 equiv) was added, the mixture was stirred for two hours until an excess of triethyamine was added to quench the reaction. Excess pyrrole was then removed by distillation and the resulting oil was first purified using a filter column (silica - hexane/CH_2Cl_2 (50/50) as the eluent). Both products were then isolated through silica gel chromatography using hexane as the eluent with an increasing amount of dichloromethane (0 to 50 %). **2** and **3** were obtained as a white solid in 5 and 25 % yield, respectively.

2: ^{1}H NMR (CD_3CN, 25°C) : δ = 1.53 (s, -CH_3, 12H), 5.75 (d, $^{4}J_{\beta\mathbf{H}\text{-}\mathrm{N}\mathbf{H}}$ = 2.9 Hz, 2H, βH), 5.80 (m, 2H), 5.92 (m, 2H), 6.54 (m, 2H), 8.10 (bs, 1H, NH), 8.72 (bs, 2H, NH).

^{13}C NMR ($CDCl_3$, 25°C) : δ = 29.3 (-**C**H_3), 35.4 (-$(Me)_2$**C**-), 103.5, 103.6, 107.9, 116.7, 138.3, 139.1.

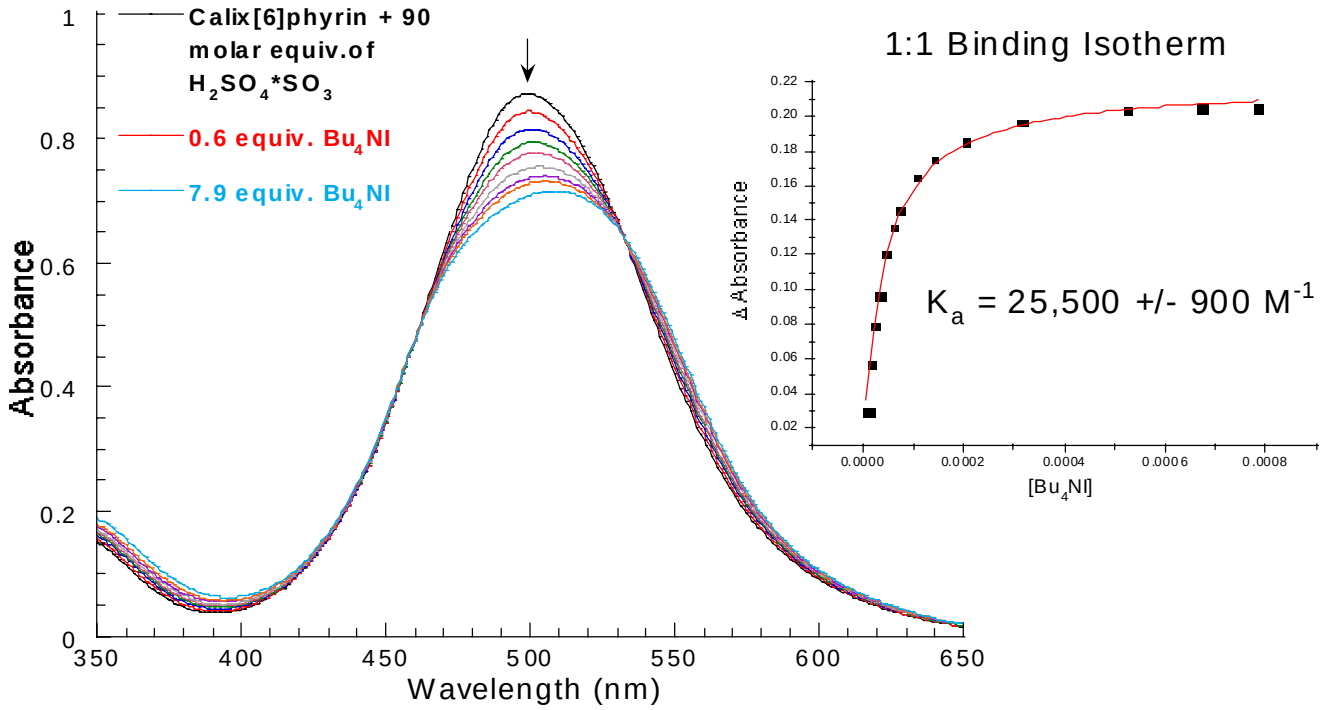
HR-MS (CI) for $C_{18}H_{24}N_3$ ($[M+H]^+$) calcd :281.1891; found : 281.1894

Elemental analysis calculated for $C_{18}H_{23}N_3$: C, 76.83, H, 8.24, N, 14.93 (%).
Found : C, 76.36, H, 8.39, N, 14.76 (%).

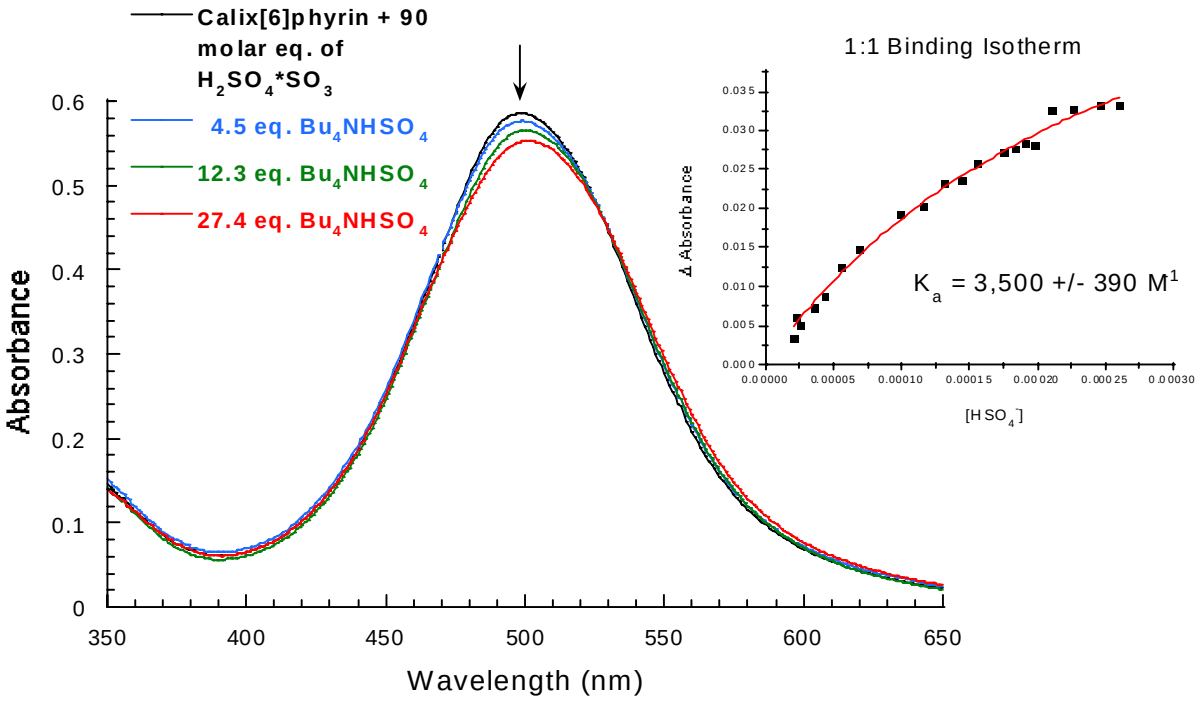
3: For caracterization data see Littler, B. J.; Miller, M. A.; Hung, C.-H.; Wagner, R. W.; O'Shea, D. F.; Boyle, P. D.; Lindsey, J. S. *J. Org. Chem.* **1999**, *64*, 1391.

UV/Vis Titration Data[1]

Titration of the Diprotonated Form of Calix[6]phyrin with Tetrabutylammonium Iodide in Acetone at 1.4 x 10^{-5} M



Titration of the Diprotonated Form of Calix[6]phyrin with Tetrabutylammonium Hydrogen Sulfate in Acetone at 9.5 x 10^{-6} M



Since the calix[6]phyrin was protonated with 90 equivalents of H_2SO_4 and exists in its diprotonated from, we infer the existence of two equivalents of HSO_4^- produced as the result of this protonation. These two equivalents were added to those produced by [Bu_4NHSO_4] addition in order to calculate the association constant[1]. The effects of sulfuric acid ionization and further production of HSO_4^- were ignored. However, attempts to fit the binding profile using additional putative equivalents of HSO_4^- resulted in an increasingly poor fit. We take this as an indication that either a minimal amount of HSO_4^- is produced as the result of solvent protonation or that the resulting species, hydrogen sulfate anion and protonated acetone, are tightly ion paired.

Crystallographic Material for 1.

X-ray Experimental.

Table 1. Crystallographic Data for **1**.

Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of **1**.

Table 3. Bond Lengths (Å) and Angles (^{o}) for the non-hydrogen atoms of **1**.

Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of **1**.

Table 5. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of **1**.

Figure 1. View of **1** showing the atom labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. Most hydrogen atoms have been removed for clarity.

Figure 2. View of **1** showing the hydrogen bonding interactions between the macrocycle and the water molecule. Thermal ellipsoids are scaled to the 50% probability level. Most hydrogen atoms have been removed for clarity. The relevant H-bonding interactions are: N2-H2N···O1W, N···O 3.072(2)Å, H···O 2.22(2)Å, N-H···O 167(2)°; N5-H5N···O1W, N···O 3.056(2)Å, H···O 2.14(2)Å, N-H···O 172(2)°; O1W-H1WA···N6, O···N 2.917(2)Å, H···N 2.07(3)Å, O-H···N 156(3)°; O1W-H1WB···N3, O···N 2.916(2)Å, H···N 2.12(3)Å, O-H···N 150(3)°.

Figure 3. Unit cell packing diagram for **1**. The view is approximately down the **a** axis

X-ray Experimental for **1** ($C_{50}H_{40}N_6F_{10} - H_2O - CH_2Cl_2$): Crystals grew as small, well formed orange prisms by slow evaporation of a dichloromethane/hexane solution. The data crystal was an orange prism of approximate dimensions; 0.22 x 0.18 x 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 521 frames of data were collected using ω-scans with a scan range of 1° and a counting time of 29 seconds per frame. The data were collected at –120 °C using a 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.[2] The structure was solved by direct methods using SIR92[3] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[4] 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). The hydrogen atoms on the pyrrole nitrogen atoms and on the water molecule were observed in a ΔF map and refined with isotropic displacement parameters. A molecule of methylene chloride was found. The molecule was disordered by a small rotation about the Cl-C bond between Cl1a and C1a. The disorder resulted in two Cl positions for the second chlorine atom of the solvent molecule. The site occupancy factors for the two affected atoms were refined while refining a common isotropic displacement parameter for the two atoms. The site occupancy factor refined to 78(2)% for Cl2a. Subsequently, the site occupancy factors were fixed and the isotropic displacement factors were refined independently. Finally, both atoms were refined anisotropically while constraining the atoms to be approximately isotropic. The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.037*P)^2 + (0.4359*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.113, with R(F) equal to 0.0485 and a goodness of fit, S, = 1.120. Definitions used for calculating R(F),$R_w(F^2)$ and the goodness of fit, S, are given below.[5] The data were corrected for secondary extinction effects. The correction takes the form: $F_{corr} = kF_c/[1 + (3.5(5)x10^{-6})* F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[6] All figures were generated using SHELXTL/PC.[7] Tables of positional and thermal parameters, bond lengths and angles, figures and lists of observed and calculated structure factors are located in tables 1 through 6.

Table 1. Crystal data and structure refinement for **1**.

Empirical formula	C51 H44 Cl2 F10 N6 O	
Formula weight	1017.82	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 13.6366(1) Å	α= 90.476(1)°.
	b = 13.9099(2) Å	β= 108.997(1)°.
	c = 15.2690(2) Å	γ = 117.499(1)°.
Volume	2385.30(5) $Å^3$	
Z	2	
Density (calculated)	1.417 Mg/m^3	
Absorption coefficient	0.221 mm^{-1}	
F(000)	1048	
Crystal size	0.22 x 0.18 x 0.15 mm	
Theta range for data collection	2.93 to 27.50°.	
Index ranges	-15<=h<=17, -18<=k<=17, -19<=l<=19	
Reflections collected	18117	
Independent reflections	10860 [R(int) = 0.0301]	
Completeness to theta = 27.50°	99.1 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10860 / 14 / 665	
Goodness-of-fit on F^2	1.120	
Final R indices [I>2sigma(I)]	R1 = 0.0485, wR2 = 0.0997	
R indices (all data)	R1 = 0.0980, wR2 = 0.1130	
Extinction coefficient	3.5(5)x10^{-6}	
Largest diff. peak and hole	0.29 and -0.39 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **1**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl1A	1805(1)	2149(1)	1222(1)	75(1)
Cl2A	774(2)	-93(1)	1519(2)	87(1)
Cl2B	823(8)	-228(5)	1142(7)	128(3)
C1A	2056(2)	1055(2)	1553(2)	75(1)
N1	5366(1)	3568(1)	2873(1)	26(1)
N2	7625(1)	6086(1)	2937(1)	28(1)
N3	9388(1)	5044(1)	2317(1)	27(1)
N4	8690(1)	3716(1)	651(1)	26(1)
N5	5810(1)	2967(1)	-655(1)	25(1)
N6	4656(1)	2192(1)	1237(1)	25(1)
C1	5579(2)	2780(2)	3314(1)	27(1)
C2	6097(2)	3207(2)	4278(1)	34(1)
C3	6197(2)	4246(2)	4403(1)	33(1)
C4	5739(2)	4457(2)	3516(1)	26(1)
C5	5735(2)	5492(1)	3221(1)	26(1)
C6	7014(2)	6319(2)	3390(1)	26(1)
C7	7803(2)	7314(2)	3986(1)	32(1)
C8	8918(2)	7691(2)	3893(1)	35(1)
C9	8790(2)	6916(2)	3242(1)	29(1)
C10	9634(2)	6877(2)	2817(1)	31(1)
C11	9757(2)	5860(2)	3001(1)	28(1)
C12	10317(2)	5666(2)	3912(1)	37(1)
C13	10307(2)	4699(2)	3753(1)	35(1)
C14	9739(2)	4300(2)	2750(1)	28(1)
C15	9604(2)	3409(2)	2229(1)	27(1)
C16	9107(2)	3116(2)	1225(1)	27(1)
C17	8943(2)	2252(2)	620(1)	31(1)
C18	8427(2)	2344(2)	-302(1)	32(1)
C19	8274(2)	3261(2)	-271(1)	26(1)
C20	7658(2)	3688(2)	-1051(1)	27(1)
C21	6334(2)	2982(2)	-1293(1)	25(1)
C22	5446(2)	2254(2)	-2092(1)	29(1)
C23	4359(2)	1792(2)	-1938(1)	29(1)
C24	4606(2)	2250(1)	-1046(1)	24(1)
C25	3817(2)	2116(2)	-498(1)	25(1)
C26	4197(2)	1644(1)	364(1)	25(1)
C27	4111(2)	569(2)	351(1)	32(1)
C28	4542(2)	477(2)	1261(1)	31(1)
C29	4879(2)	1486(1)	1832(1)	26(1)
C30	5320(2)	1786(2)	2802(1)	27(1)
C31	5263(2)	5920(2)	3825(1)	35(1)
C32	4946(2)	5259(2)	2174(1)	31(1)
C33	10871(2)	7895(2)	3296(2)	43(1)
C34	9173(2)	6871(2)	1755(1)	37(1)
C35	10048(2)	2686(2)	2728(1)	29(1)
C36	9284(2)	1636(2)	2804(1)	34(1)
C37	9682(2)	956(2)	3248(1)	42(1)
C38	10882(2)	1318(2)	3607(2)	47(1)
C39	11667(2)	2342(2)	3530(1)	43(1)

C40	11250(2)	3021(2)	3101(1)	35(1)
C41	7955(2)	3569(2)	-1918(1)	38(1)
C42	8071(2)	4912(2)	-733(1)	33(1)
C43	3921(2)	3233(2)	-222(1)	31(1)
C44	2515(2)	1296(2)	-1112(1)	35(1)
C45	5539(2)	987(2)	3377(1)	28(1)
C46	4615(2)	77(2)	3499(1)	30(1)
C47	4806(2)	-672(2)	4016(1)	34(1)
C48	5927(2)	-538(2)	4409(1)	37(1)
C49	6865(2)	359(2)	4300(1)	36(1)
C50	6669(2)	1123(2)	3811(1)	33(1)
F36	8097(1)	1239(1)	2426(1)	45(1)
F37	8918(1)	-44(1)	3336(1)	62(1)
F38	11285(1)	660(1)	4028(1)	71(1)
F39	12841(1)	2687(1)	3856(1)	66(1)
F40	12044(1)	4023(1)	3034(1)	48(1)
F46	3495(1)	-103(1)	3106(1)	42(1)
F47	3876(1)	-1552(1)	4113(1)	49(1)
F48	6099(1)	-1284(1)	4888(1)	56(1)
F49	7963(1)	488(1)	4667(1)	56(1)
F50	7611(1)	2018(1)	3757(1)	47(1)
O1W	6832(2)	4182(1)	1376(1)	34(1)

Table 3. Bond lengths [Å] and angles [°] for **1**

Cl1A-C1A	1.752(3)	C18-H18	0.96
Cl2A-Cl2B	0.638(9)	C19-C20	1.516(2)
Cl2A-C1A	1.724(3)	C20-C21	1.511(3)
Cl2B-C1A	1.712(7)	C20-C41	1.533(3)
C1A-H1AA	0.96	C20-C42	1.541(3)
C1A-H1AB	0.96	C21-C22	1.367(2)
N1-C4	1.351(2)	C22-C23	1.421(3)
N1-C1	1.386(2)	C22-H22	0.96
N1-H1N	0.88(2)	C23-C24	1.364(2)
N2-C9	1.375(2)	C23-H23	0.96
N2-C6	1.377(2)	C24-C25	1.515(2)
N2-H2N	0.87(2)	C25-C26	1.523(2)
N3-C11	1.320(2)	C25-C43	1.536(2)
N3-C14	1.414(2)	C25-C44	1.537(3)
N4-C19	1.354(2)	C26-C27	1.444(3)
N4-C16	1.384(2)	C27-C28	1.354(3)
N4-H4N	0.836(19)	C27-H27	0.96
N5-C24	1.374(2)	C28-C29	1.437(3)
N5-C21	1.378(2)	C28-H28	0.96
N5-H5N	0.92(2)	C29-C30	1.382(2)
N6-C26	1.323(2)	C30-C45	1.499(2)
N6-C29	1.412(2)	C31-H31A	0.96
C1-C2	1.393(3)	C31-H31B	0.96
C1-C30	1.413(3)	C31-H31C	0.96
C2-C3	1.393(3)	C32-H32A	0.96
C2-H2	0.96	C32-H32B	0.96
C3-C4	1.391(3)	C32-H32C	0.96
C3-H3	0.96	C33-H33A	0.96
C4-C5	1.514(2)	C33-H33B	0.96
C5-C6	1.511(3)	C33-H33C	0.96
C5-C31	1.531(3)	C34-H34A	0.96
C5-C32	1.539(3)	C34-H34B	0.96
C6-C7	1.367(3)	C34-H34C	0.96
C7-C8	1.419(3)	C35-C40	1.382(3)
C7-H7	0.96	C35-C36	1.382(3)
C8-C9	1.369(3)	C36-F36	1.348(2)
C8-H8	0.96	C36-C37	1.376(3)
C9-C10	1.514(3)	C37-F37	1.340(2)
C10-C11	1.517(3)	C37-C38	1.372(3)
C10-C34	1.536(3)	C38-F38	1.341(2)
C10-C33	1.536(3)	C38-C39	1.366(3)
C11-C12	1.448(3)	C39-F39	1.345(2)
C12-C13	1.358(3)	C39-C40	1.381(3)
C12-H12	0.96	C40-F40	1.344(2)
C13-C14	1.439(3)	C41-H41A	0.96
C13-H13	0.96	C41-H41B	0.96
C14-C15	1.371(3)	C41-H41C	0.96
C15-C16	1.426(2)	C42-H42A	0.96
C15-C35	1.494(2)	C42-H42B	0.96
C16-C17	1.396(3)	C42-H42C	0.96
C17-C18	1.391(3)	C43-H43A	0.96
C17-H17	0.96	C43-H43B	0.96
C18-C19	1.387(3)	C43-H43C	0.96

C44-H44A	0.96	C47-C48	1.368(3)
C44-H44B	0.96	C48-F48	1.338(2)
C44-H44C	0.96	C48-C49	1.374(3)
C45-C46	1.382(3)	C49-F49	1.338(2)
C45-C50	1.382(3)	C49-C50	1.381(3)
C46-F46	1.343(2)	C50-F50	1.342(2)
C46-C47	1.380(3)	O1W-H1WA	0.90(3)
C47-F47	1.349(2)	O1W-H1WB	0.88(3)
Cl2B-C1A-Cl1A	115.0(4)	C8-C9-N2	106.90(17)
Cl2A-C1A-Cl1A	111.92(18)	C8-C9-C10	132.46(18)
Cl2A-C1A-H1AA	107.6	N2-C9-C10	120.55(16)
Cl1A-C1A-H1AA	109.1	C9-C10-C11	109.54(15)
Cl2A-C1A-H1AB	111.2	C9-C10-C34	109.86(16)
Cl1A-C1A-H1AB	108.8	C11-C10-C34	110.94(15)
H1AA-C1A-H1AB	108.0	C9-C10-C33	109.34(16)
C4-N1-C1	110.52(15)	C11-C10-C33	107.83(16)
C4-N1-H1N	128.7(13)	C34-C10-C33	109.28(16)
C1-N1-H1N	120.6(13)	N3-C11-C12	111.31(16)
C9-N2-C6	110.42(16)	N3-C11-C10	122.67(16)
C9-N2-H2N	126.4(13)	C12-C11-C10	125.98(17)
C6-N2-H2N	123.2(13)	C13-C12-C11	106.81(17)
C11-N3-C14	106.27(15)	C13-C12-H12	126.6
C19-N4-C16	110.79(16)	C11-C12-H12	126.6
C19-N4-H4N	128.5(12)	C12-C13-C14	106.61(17)
C16-N4-H4N	120.6(12)	C12-C13-H13	127.2
C24-N5-C21	110.32(15)	C14-C13-H13	126.2
C24-N5-H5N	123.1(13)	C15-C14-N3	121.65(16)
C21-N5-H5N	126.6(13)	C15-C14-C13	129.21(17)
C26-N6-C29	106.05(14)	N3-C14-C13	108.95(16)
N1-C1-C2	106.24(16)	C14-C15-C16	125.73(16)
N1-C1-C30	122.23(16)	C14-C15-C35	118.98(16)
C2-C1-C30	131.49(17)	C16-C15-C35	115.24(15)
C1-C2-C3	107.97(16)	N4-C16-C17	105.98(16)
C1-C2-H2	125.7	N4-C16-C15	123.00(16)
C3-C2-H2	126.4	C17-C16-C15	131.01(16)
C4-C3-C2	107.84(16)	C18-C17-C16	107.96(16)
C4-C3-H3	126.3	C18-C17-H17	126.3
C2-C3-H3	125.9	C16-C17-H17	125.8
N1-C4-C3	107.42(16)	C19-C18-C17	108.13(16)
N1-C4-C5	121.53(16)	C19-C18-H18	125.3
C3-C4-C5	130.72(17)	C17-C18-H18	126.5
C6-C5-C4	105.99(15)	N4-C19-C18	107.14(16)
C6-C5-C31	110.68(15)	N4-C19-C20	121.85(16)
C4-C5-C31	109.51(15)	C18-C19-C20	130.72(17)
C6-C5-C32	110.47(15)	C21-C20-C19	107.20(15)
C4-C5-C32	110.89(15)	C21-C20-C41	109.49(15)
C31-C5-C32	109.26(16)	C19-C20-C41	109.20(15)
C7-C6-N2	106.87(16)	C21-C20-C42	110.91(15)
C7-C6-C5	132.64(17)	C19-C20-C42	110.86(15)
N2-C6-C5	120.36(16)	C41-C20-C42	109.14(15)
C6-C7-C8	107.92(17)	C22-C21-N5	106.66(16)
C6-C7-H7	125.8	C22-C21-C20	131.32(17)
C8-C7-H7	126.3	N5-C21-C20	122.00(16)
C9-C8-C7	107.89(17)	C21-C22-C23	108.14(16)
C9-C8-H8	125.9	C21-C22-H22	125.6
C7-C8-H8	126.2	C23-C22-H22	126.3

C24-C23-C22	107.62(16)	C37-C36-C35	122.6(2)
C24-C23-H23	125.7	F37-C37-C38	119.98(18)
C22-C23-H23	126.7	F37-C37-C36	121.0(2)
C23-C24-N5	107.26(16)	C38-C37-C36	119.0(2)
C23-C24-C25	132.12(17)	F38-C38-C39	119.9(2)
N5-C24-C25	120.60(15)	F38-C38-C37	119.8(2)
C24-C25-C26	107.95(14)	C39-C38-C37	120.31(18)
C24-C25-C43	109.97(14)	F39-C39-C38	120.45(19)
C26-C25-C43	111.78(15)	F39-C39-C40	119.8(2)
C24-C25-C44	109.62(15)	C38-C39-C40	119.7(2)
C26-C25-C44	108.53(15)	F40-C40-C39	118.38(19)
C43-C25-C44	108.95(15)	F40-C40-C35	119.83(17)
N6-C26-C27	111.38(15)	C39-C40-C35	121.8(2)
N6-C26-C25	122.81(15)	C20-C41-H41A	109.3
C27-C26-C25	125.81(16)	C20-C41-H41B	109.8
C28-C27-C26	106.86(16)	H41A-C41-H41B	109.5
C28-C27-H27	126.9	C20-C41-H41C	109.4
C26-C27-H27	126.2	H41A-C41-H41C	109.5
C27-C28-C29	106.67(16)	H41B-C41-H41C	109.5
C27-C28-H28	126.8	C20-C42-H42A	109.4
C29-C28-H28	126.5	C20-C42-H42B	109.1
C30-C29-N6	121.40(16)	H42A-C42-H42B	109.5
C30-C29-C28	129.54(16)	C20-C42-H42C	109.9
N6-C29-C28	109.03(15)	H42A-C42-H42C	109.5
C29-C30-C1	126.20(16)	H42B-C42-H42C	109.5
C29-C30-C45	117.62(16)	C25-C43-H43A	108.9
C1-C30-C45	116.17(15)	C25-C43-H43B	109.5
C5-C31-H31A	109.5	H43A-C43-H43B	109.5
C5-C31-H31B	109.8	C25-C43-H43C	110.0
H31A-C31-H31B	109.5	H43A-C43-H43C	109.5
C5-C31-H31C	109.1	H43B-C43-H43C	109.5
H31A-C31-H31C	109.5	C25-C44-H44A	109.0
H31B-C31-H31C	109.5	C25-C44-H44B	109.7
C5-C32-H32A	109.8	H44A-C44-H44B	109.5
C5-C32-H32B	109.6	C25-C44-H44C	109.7
H32A-C32-H32B	109.5	H44A-C44-H44C	109.5
C5-C32-H32C	109.0	H44B-C44-H44C	109.5
H32A-C32-H32C	109.5	C46-C45-C50	116.81(17)
H32B-C32-H32C	109.5	C46-C45-C30	121.27(17)
C10-C33-H33A	109.4	C50-C45-C30	121.92(17)
C10-C33-H33B	109.6	F46-C46-C47	118.18(17)
H33A-C33-H33B	109.5	F46-C46-C45	120.09(16)
C10-C33-H33C	109.5	C47-C46-C45	121.72(19)
H33A-C33-H33C	109.5	F47-C47-C48	120.13(17)
H33B-C33-H33C	109.5	F47-C47-C46	119.66(19)
C10-C34-H34A	109.0	C48-C47-C46	120.19(19)
C10-C34-H34B	109.6	F48-C48-C47	119.8(2)
H34A-C34-H34B	109.5	F48-C48-C49	120.72(19)
C10-C34-H34C	109.8	C47-C48-C49	119.49(17)
H34A-C34-H34C	109.5	F49-C49-C48	119.99(17)
H34B-C34-H34C	109.5	F49-C49-C50	120.3(2)
C40-C35-C36	116.59(17)	C48-C49-C50	119.70(19)
C40-C35-C15	121.28(17)	F50-C50-C49	118.26(18)
C36-C35-C15	122.08(17)	F50-C50-C45	119.75(17)
F36-C36-C37	117.54(18)	C49-C50-C45	121.99(19)
F36-C36-C35	119.88(17)	H1WA-O1W-H1WB	99(2)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **1**. 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}
Cl1A	85(1)	75(1)	96(1)	36(1)	56(1)	49(1)
Cl2A	70(1)	68(1)	103(1)	27(1)	49(1)	8(1)
Cl2B	91(4)	94(4)	205(10)	39(5)	57(6)	50(3)
C1A	57(2)	67(2)	117(3)	35(2)	47(2)	33(2)
N1	29(1)	25(1)	24(1)	8(1)	9(1)	14(1)
N2	29(1)	20(1)	34(1)	0(1)	13(1)	10(1)
N3	26(1)	25(1)	31(1)	5(1)	10(1)	14(1)
N4	28(1)	24(1)	30(1)	5(1)	10(1)	17(1)
N5	23(1)	29(1)	21(1)	3(1)	6(1)	12(1)
N6	26(1)	25(1)	25(1)	8(1)	9(1)	13(1)
C1	31(1)	24(1)	28(1)	11(1)	12(1)	14(1)
C2	42(1)	32(1)	27(1)	13(1)	12(1)	19(1)
C3	41(1)	31(1)	25(1)	5(1)	12(1)	17(1)
C4	23(1)	25(1)	29(1)	5(1)	13(1)	10(1)
C5	27(1)	23(1)	29(1)	6(1)	12(1)	13(1)
C6	28(1)	23(1)	29(1)	7(1)	13(1)	14(1)
C7	34(1)	29(1)	36(1)	1(1)	14(1)	16(1)
C8	28(1)	24(1)	47(1)	-2(1)	14(1)	9(1)
C9	26(1)	21(1)	39(1)	4(1)	12(1)	10(1)
C10	29(1)	23(1)	41(1)	4(1)	16(1)	11(1)
C11	22(1)	27(1)	34(1)	2(1)	11(1)	10(1)
C12	37(1)	39(1)	31(1)	0(1)	8(1)	19(1)
C13	38(1)	40(1)	29(1)	7(1)	9(1)	23(1)
C14	25(1)	26(1)	32(1)	7(1)	10(1)	13(1)
C15	24(1)	25(1)	30(1)	8(1)	8(1)	13(1)
C16	25(1)	25(1)	33(1)	7(1)	9(1)	15(1)
C17	32(1)	28(1)	36(1)	5(1)	9(1)	19(1)
C18	33(1)	30(1)	31(1)	0(1)	8(1)	18(1)
C19	22(1)	29(1)	28(1)	5(1)	9(1)	12(1)
C20	26(1)	33(1)	27(1)	8(1)	11(1)	15(1)
C21	28(1)	27(1)	26(1)	8(1)	11(1)	17(1)
C22	33(1)	31(1)	25(1)	2(1)	10(1)	18(1)
C23	27(1)	27(1)	26(1)	1(1)	5(1)	12(1)
C24	22(1)	25(1)	25(1)	7(1)	6(1)	12(1)
C25	21(1)	27(1)	25(1)	8(1)	8(1)	12(1)
C26	20(1)	24(1)	27(1)	6(1)	10(1)	9(1)
C27	37(1)	26(1)	30(1)	4(1)	11(1)	15(1)
C28	37(1)	25(1)	35(1)	9(1)	14(1)	18(1)
C29	26(1)	23(1)	29(1)	8(1)	9(1)	11(1)
C30	28(1)	27(1)	29(1)	12(1)	13(1)	15(1)
C31	35(1)	33(1)	42(1)	4(1)	19(1)	17(1)
C32	30(1)	28(1)	36(1)	8(1)	10(1)	17(1)
C33	33(1)	29(1)	64(2)	-1(1)	23(1)	9(1)
C34	40(1)	31(1)	52(1)	16(1)	26(1)	22(1)
C35	34(1)	28(1)	25(1)	3(1)	6(1)	18(1)
C36	38(1)	31(1)	28(1)	3(1)	6(1)	19(1)
C37	59(2)	26(1)	31(1)	5(1)	6(1)	19(1)

C38	65(2)	40(1)	33(1)	6(1)	-2(1)	36(1)
C39	37(1)	53(2)	35(1)	2(1)	-5(1)	30(1)
C40	35(1)	32(1)	33(1)	7(1)	6(1)	17(1)
C41	36(1)	57(1)	33(1)	16(1)	17(1)	27(1)
C42	29(1)	33(1)	38(1)	14(1)	12(1)	15(1)
C43	33(1)	38(1)	31(1)	13(1)	14(1)	23(1)
C44	26(1)	42(1)	33(1)	6(1)	7(1)	14(1)
C45	36(1)	27(1)	24(1)	6(1)	10(1)	18(1)
C46	33(1)	29(1)	26(1)	7(1)	7(1)	18(1)
C47	48(1)	23(1)	26(1)	7(1)	13(1)	15(1)
C48	59(2)	31(1)	25(1)	7(1)	7(1)	31(1)
C49	41(1)	43(1)	28(1)	3(1)	1(1)	30(1)
C50	34(1)	31(1)	32(1)	5(1)	10(1)	16(1)
F36	38(1)	35(1)	52(1)	9(1)	13(1)	13(1)
F37	81(1)	29(1)	54(1)	15(1)	12(1)	20(1)
F38	93(1)	53(1)	56(1)	9(1)	-12(1)	53(1)
F39	48(1)	74(1)	66(1)	6(1)	-8(1)	41(1)
F40	32(1)	43(1)	57(1)	13(1)	8(1)	15(1)
F46	34(1)	39(1)	51(1)	19(1)	14(1)	16(1)
F47	63(1)	31(1)	45(1)	19(1)	20(1)	18(1)
F48	84(1)	45(1)	42(1)	16(1)	7(1)	45(1)
F49	52(1)	67(1)	52(1)	8(1)	2(1)	44(1)
F50	35(1)	47(1)	56(1)	14(1)	16(1)	18(1)
O1W	27(1)	33(1)	37(1)	4(1)	10(1)	11(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **1**.

	x	y	z	U(eq)
H1AA	2624	1279	2189	90
H1AB	2392	888	1150	90
H2	6337	2843	4768	40
H3	6517	4726	4997	39
H7	7628	7694	4388	39
H8	9637	8368	4225	42
H12	10639	6133	4513	44
H13	10606	4340	4216	42
H17	9152	1694	810	37
H18	8206	1859	-867	38
H22	5546	2085	-2658	34
H23	3587	1251	-2374	34
H27	3800	25	-200	38
H28	4613	-140	1483	37
H31A	5750	6061	4479	53
H31B	5282	6589	3654	53
H31C	4459	5372	3720	53
H32A	5240	4996	1791	47
H32B	4142	4713	2072	47
H32C	4966	5930	2006	47
H33A	11161	7899	3960	65
H33B	11409	7871	3030	65
H33C	10815	8552	3197	65
H34A	8401	6233	1459	55
H34B	9115	7526	1654	55
H34C	9709	6846	1487	55
H41A	7693	2806	-2120	58
H41B	7558	3828	-2419	58
H41C	8799	3996	-1756	58
H42A	7884	4989	-191	50
H42B	8915	5339	-571	50
H42C	7674	5171	-1234	50
H43A	4735	3745	161	46
H43B	3678	3507	-781	46
H43C	3423	3149	127	46
H44A	2448	596	-1282	53
H44B	2015	1209	-764	53
H44C	2269	1566	-1672	53
H1N	4993(18)	3437(16)	2255(15)	37(6)
H2N	7296(17)	5490(16)	2516(13)	32(6)
H4N	8754(16)	4298(16)	882(12)	24(5)
H5N	6187(18)	3376(17)	-52(15)	42(6)
H1WA	6310(30)	3530(30)	1461(19)	86(10)
H1WB	7500(30)	4190(20)	1700(19)	81(10)

Figure 1. View of **1** showing the atom labeling scheme. Thermal ellipsoids are scaled to the 50% probability level. Most hydrogen atoms have been removed for clarity.

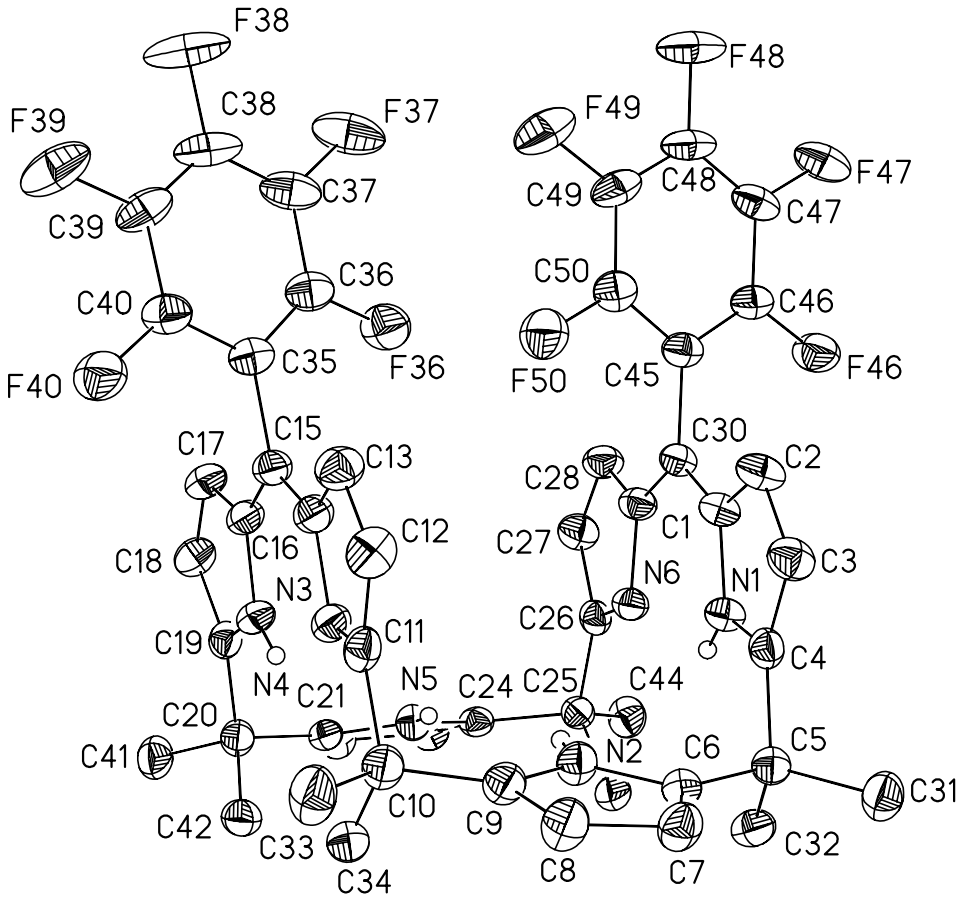


Figure 2. View of **1** showing the hydrogen bonding interactions between the macrocycle and the water molecule. Thermal ellipsoids are scaled to the 50% probability level. Most hydrogen atoms have been removed for clarity. The relevant H-bonding interactions are: N2-H2N···O1W, N···O 3.072(2)Å, H···O 2.22(2)Å, N-H···O 167(2)°; N5-H5N···O1W, N···O 3.056(2)Å, H···O 2.14(2)Å, N-H···O 172(2)°; O1W-H1WA···N6, O···N 2.917(2)Å, H···N 2.07(3)Å, O-H···N 156(3)°; O1W-H1WB···N3, O···N 2.916(2)Å, H···N 2.12(3)Å, O-H···N 150(3)°.

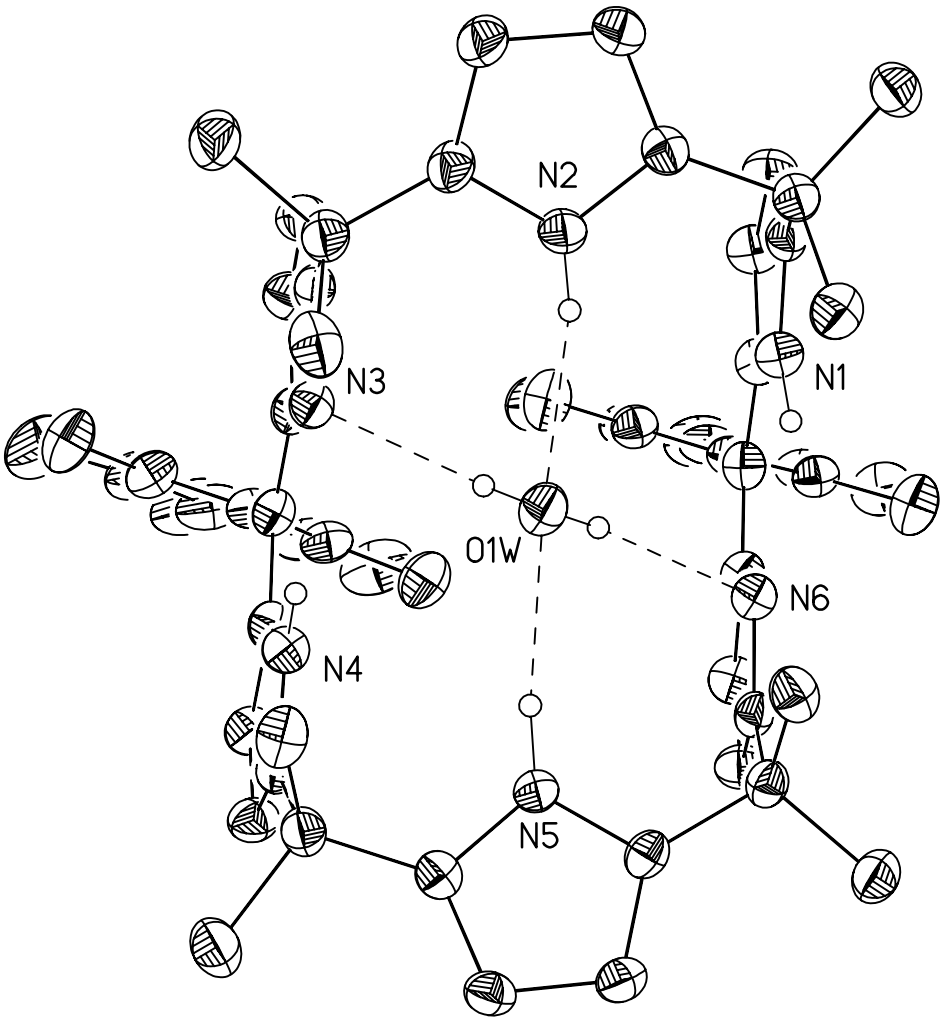
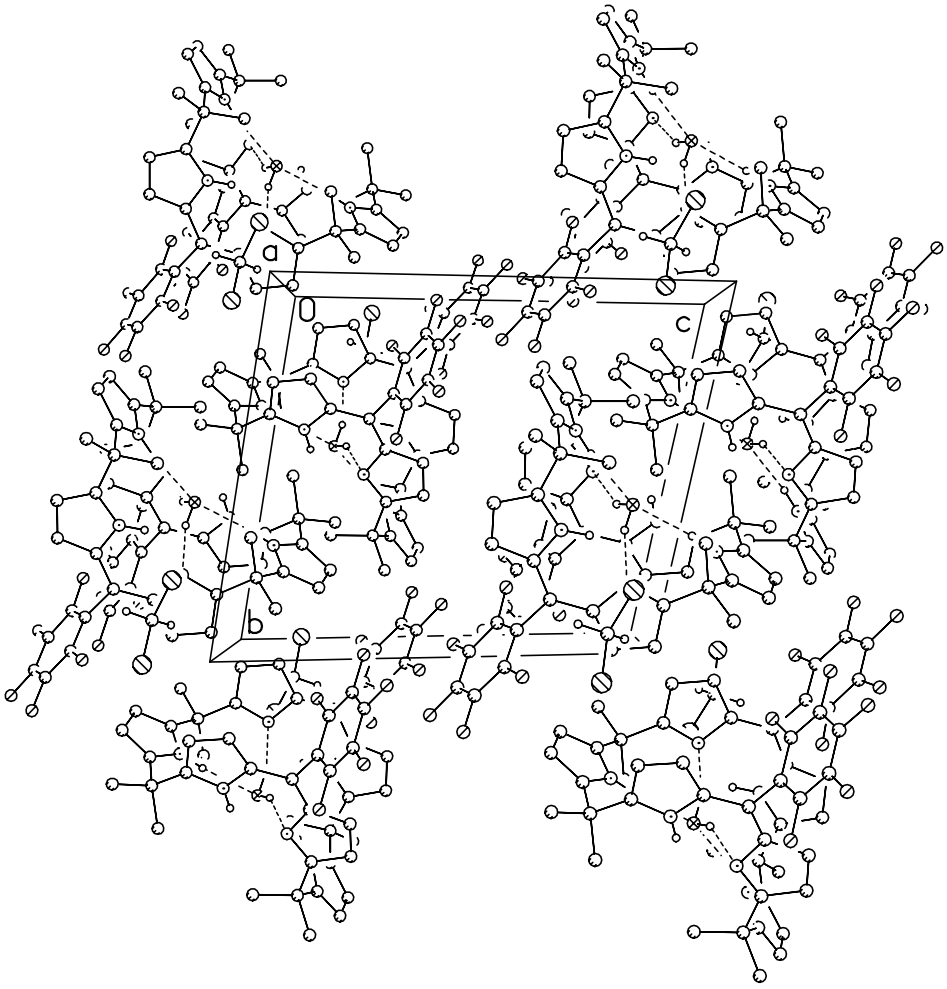


Figure 3. Unit cell packing diagram for **1**. The view is approximately down the **a** axis



Crystallographic Material for $(\mathbf{1} \bullet H^+)Cl^-$

X-ray Experimental.

Table 1. Crystallographic Data for $(\mathbf{1} \bullet H^+)Cl^-$.

Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of $(\mathbf{1} \cdot H^+)Cl^-$.

Table 3. Bond Lengths (Å) and Angles (°) for the non-hydrogen atoms of $(\mathbf{1} \bullet H^+)Cl^-$.

Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of $(\mathbf{1} \cdot H^+)Cl^-$.

Table 5. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of $(\mathbf{1} \cdot H^+)Cl^-$.

Figure 1. View of $(\mathbf{1} \cdot H^+)Cl^-$ showing the atom labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. The fluorine atoms and most hydrogen atoms have been removed for clarity.

Figure 2. View of $(\mathbf{1} \cdot H^+)Cl^-$ showing a partial atom labeling scheme. Thermal ellipsoids are scaled to the 30% probability level.

Figure 3. View of $(\mathbf{1} \cdot H^+)Cl^-$ showing the H-bonding interactions and the disordered Cl^- ion. Thermal ellipsoids are scaled to the 30% probability level. The geometry of the H-bonding interactions are: N1-H1N···Cl1, N···Cl 3.184(3)Å, H···Cl 2.30(4)Å, N-H···Cl 165(4)°; N3-H3N···Cl1, N···Cl 3.105(3)Å, H···Cl 2.27(5)Å, N-H···Cl 158(4)°; N4-H4N···Cl1, N···Cl 3.229(4)Å, H···Cl 2.41(3)Å, N-H···Cl 173(3)°; N1-H1N···Cl1a, N···Cl 3.267(8)Å, H···Cl 2.41(4)Å, N-H···Cl 157(3)°; N4-H4N···Cl1a, N···Cl 3.204(8)Å, H···Cl 2.41(3)Å, N-H···Cl 161(3)°; N6-H6N···Cl1a, N···Cl 3.121(8)Å, H···Cl 2.35(4)Å, N-H···Cl 148(3)°.

Figure 4. Unit cell packing diagram for $(\mathbf{1} \cdot H^+)Cl^-$. The view is approximately down the **a** axis. Dashed lines are indicative of a NH···Cl hydrogen bonding interaction.

X-ray Experimental for $C_{28}H_{44}N_4O_4Cl$: Crystals grew as very dark green plates by slow evaporation of a MeOH-dichloromethane mixture. The data crystal was a plate that had approximate dimensions; 0.22 x 0.17 x 0.04mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoKα radiation (λ = 0.71073Å). A total of 328 frames of data were collected using ω-scans with a scan range of 1.2° and a counting time of 241 seconds per frame. The data were collected at –120 °C using a 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.[2] The structure was solved by direct methods using SIR92[3] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[4] 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). The hydrogen atoms on the pyrrole nitrogen atoms were observed in a ΔF map and refined with isotropic displacement parameters. A fairly large peak less than 1Å from Cl1 persisted throughout the refinement. It was concluded to be due to a positional disorder of the chloride ion in the cavity of the macrocycle (Figure 3). The site occupancy factors for the two atoms were refined while refining a common isotropic displacement parameter while all other atoms were refined normally. Upon convergence of the site occupancy factors at 84(2)% for Cl1, the isotropic displacement parameters for the Cl^- ions were refined independently. Finally, the Cl^- ions were refined anisotropically. The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0757*P)^2 + (6.4763*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.166, with R(F) equal to 0.0757 and a goodness of fit, S, = 1.021. Definitions used for calculating R(F),$R_w(F^2)$ and the goodness of fit, S, are given below.[5] Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[6] All figures were generated using SHELXTL/PC.[7]

Table 1. Crystal data and structure refinement for $(\mathbf{1}\cdot H^+)Cl^-$.

Empirical formula	C50 H41 Cl F10 N6	
Formula weight	951.34	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P21/c	
Unit cell dimensions	a = 10.4868(2) Å	α= 90°.
	b = 23.5023(4) Å	β= 99.287(1)°.
	c = 18.3880(4) Å	γ = 90°.
Volume	4472.57(15) $Å^3$	
Z	4	
Density (calculated)	1.413 Mg/m^3	
Absorption coefficient	0.171 mm^{-1}	
F(000)	1960	
Crystal size	0.22 x 0.17 x 0.04 mm	
Theta range for data collection	2.98 to 27.50°.	
Index ranges	-13<=h<=13, -30<=k<=26, -23<=l<=23	
Reflections collected	18696	
Independent reflections	10159 [R(int) = 0.0729]	
Completeness to theta = 27.50°	98.8 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10159 / 0 / 633	
Goodness-of-fit on F^2	1.021	
Final R indices [I>2sigma(I)]	R1 = 0.0757, wR2 = 0.1312	
R indices (all data)	R1 = 0.1786, wR2 = 0.1658	
Largest diff. peak and hole	0.311 and -0.281 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for (**1**·H^+)Cl^-. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl1	3961(2)	6089(1)	112(1)	46(1)
Cl1A	3232(7)	6031(4)	-95(4)	52(2)
N1	2913(3)	6058(1)	1643(2)	31(1)
N2	5893(3)	6655(1)	1412(2)	36(1)
N3	6559(3)	6629(1)	-135(2)	33(1)
N4	4493(3)	6071(1)	-1570(2)	31(1)
N5	1627(3)	6835(1)	-1230(2)	34(1)
N6	990(3)	6822(1)	215(2)	35(1)
C1	1650(3)	5976(1)	1745(2)	31(1)
C2	1690(4)	5795(2)	2454(2)	39(1)
C3	3003(4)	5771(2)	2791(2)	42(1)
C4	3744(3)	5934(1)	2285(2)	33(1)
C5	5191(4)	5981(2)	2325(2)	44(1)
C6	5480(3)	6565(2)	2050(2)	36(1)
C7	5321(4)	7098(2)	2365(2)	43(1)
C8	5641(4)	7508(2)	1899(2)	39(1)
C9	6013(3)	7234(1)	1280(2)	32(1)
C10	6451(3)	7469(1)	670(2)	33(1)
C11	6793(3)	7196(2)	52(2)	33(1)
C12	7495(4)	7416(2)	-470(2)	45(1)
C13	7676(4)	6992(2)	-955(2)	50(1)
C14	7060(3)	6501(2)	-747(2)	32(1)
C15	6891(3)	5929(2)	-1128(2)	34(1)
C16	5711(3)	5966(1)	-1722(2)	31(1)
C17	5586(3)	5903(2)	-2466(2)	34(1)
C18	4263(3)	5970(2)	-2770(2)	36(1)
C19	3604(3)	6070(1)	-2205(2)	30(1)
C20	2187(3)	6164(1)	-2182(2)	34(1)
C21	2030(3)	6747(1)	-1869(2)	32(1)
C22	2285(3)	7280(2)	-2209(2)	37(1)
C23	2017(3)	7697(2)	-1760(2)	38(1)
C24	1606(3)	7431(1)	-1133(2)	33(1)
C25	1241(3)	7686(1)	-522(2)	33(1)
C26	900(3)	7404(1)	106(2)	32(1)
C27	398(3)	7626(2)	702(2)	36(1)
C28	189(3)	7177(1)	1158(2)	35(1)
C29	560(3)	6681(1)	848(2)	29(1)
C30	525(3)	6076(1)	1127(2)	35(1)
C31	5846(4)	5931(2)	3141(2)	71(2)
C32	5701(4)	5518(2)	1867(3)	57(1)
C33	6563(3)	8100(2)	671(2)	35(1)
C34	5735(4)	8425(2)	164(2)	42(1)
C35	5749(4)	9005(2)	183(2)	51(1)
C36	6627(4)	9283(2)	694(2)	47(1)
C37	7485(4)	8981(2)	1176(2)	40(1)
C38	7440(3)	8397(2)	1176(2)	34(1)
C39	8074(3)	5807(2)	-1490(2)	47(1)
C40	6721(4)	5454(2)	-576(2)	43(1)
C41	1435(4)	6142(2)	-2971(2)	52(1)

C42	1682(4)	5695(2)	-1715(2)	48(1)
C43	1130(4)	8320(2)	-518(2)	36(1)
C44	187(4)	8609(2)	-993(2)	43(1)
C45	74(4)	9193(2)	-984(2)	51(1)
C46	909(5)	9504(2)	-490(3)	55(1)
C47	1829(5)	9236(2)	0(2)	51(1)
C48	1939(4)	8655(2)	-15(2)	42(1)
C49	587(4)	5650(2)	497(2)	49(1)
C50	-742(4)	5987(2)	1418(3)	56(1)
F34	4858(2)	8165(1)	-337(1)	59(1)
F35	4882(3)	9303(1)	-287(1)	77(1)
F36	6616(3)	9855(1)	729(1)	68(1)
F37	8341(2)	9260(1)	1680(1)	55(1)
F38	8270(2)	8116(1)	1685(1)	45(1)
F44	-655(2)	8315(1)	-1479(1)	58(1)
F45	-843(3)	9457(1)	-1461(2)	77(1)
F46	803(3)	10078(1)	-482(2)	82(1)
F47	2633(3)	9545(1)	495(1)	77(1)
F48	2875(2)	8408(1)	467(1)	54(1)

Table 3. Bond lengths [Å] and angles [°] for $(\mathbf{1}\cdot H^{+})Cl^{-}$.

N1-C1	1.381(4)	C24-C25	1.380(5)
N1-C4	1.382(4)	C25-C26	1.426(5)
N1-H1N	0.91(4)	C25-C43	1.494(5)
N2-C6	1.332(5)	C26-C27	1.392(5)
N2-C9	1.390(5)	C27-C28	1.386(5)
N2-H2N	0.73(4)	C27-H27	0.96
N3-C14	1.349(4)	C28-C29	1.382(5)
N3-C11	1.388(4)	C28-H28	0.96
N3-H3N	0.88(5)	C29-C30	1.514(5)
N4-C19	1.372(4)	C30-C50	1.524(5)
N4-C16	1.373(4)	C30-C49	1.542(5)
N4-H4N	0.82(3)	C31-H31A	0.96
N5-C21	1.328(4)	C31-H31B	0.96
N5-C24	1.413(4)	C31-H31C	0.96
N6-C29	1.357(5)	C32-H32A	0.96
N6-C26	1.382(4)	C32-H32B	0.96
N6-H6N	0.87(4)	C32-H32C	0.96
C1-C2	1.365(5)	C33-C38	1.385(5)
C1-C30	1.519(5)	C33-C34	1.395(5)
C2-C3	1.416(5)	C34-F34	1.338(4)
C2-H2	0.96	C34-C35	1.365(5)
C3-C4	1.360(5)	C35-F35	1.347(4)
C3-H3	0.96	C35-C36	1.369(6)
C4-C5	1.511(5)	C36-F36	1.346(4)
C5-C6	1.511(5)	C36-C37	1.358(5)
C5-C32	1.524(6)	C37-F37	1.352(4)
C5-C31	1.551(5)	C37-C38	1.373(5)
C6-C7	1.401(5)	C38-F38	1.344(4)
C7-C8	1.367(5)	C39-H39A	0.96
C7-H7	0.96	C39-H39B	0.96
C8-C9	1.418(5)	C39-H39C	0.96
C8-H8	0.96	C40-H40A	0.96
C9-C10	1.393(5)	C40-H40B	0.96
C10-C11	1.403(5)	C40-H40C	0.96
C10-C33	1.488(5)	C41-H41A	0.96
C11-C12	1.399(5)	C41-H41B	0.96
C12-C13	1.372(5)	C41-H41C	0.96
C12-H12	0.96	C42-H42A	0.96
C13-C14	1.405(5)	C42-H42B	0.96
C13-H13	0.96	C42-H42C	0.96
C14-C15	1.513(5)	C43-C44	1.387(5)
C15-C16	1.515(5)	C43-C48	1.393(5)
C15-C39	1.526(5)	C44-F44	1.342(4)
C15-C40	1.540(5)	C44-C45	1.378(5)
C16-C17	1.362(5)	C45-F45	1.343(5)
C17-C18	1.418(5)	C45-C46	1.367(6)
C17-H17	0.96	C46-F46	1.353(4)
C18-C19	1.358(5)	C46-C47	1.363(6)
C18-H18	0.96	C47-F47	1.348(5)
C19-C20	1.510(5)	C47-C48	1.371(5)
C20-C21	1.505(5)	C48-F48	1.344(4)
C20-C41	1.536(5)	C49-H49A	0.96
C20-C42	1.544(5)	C49-H49B	0.96
C21-C22	1.443(5)	C49-H49C	0.96
C22-C23	1.341(5)	C50-H50A	0.96
C22-H22	0.96	C50-H50B	0.96
C23-C24	1.438(5)	C50-H50C	0.96
C23-H23	0.96		
C1-N1-C4	109.9(3)	C4-N1-H1N	125(3)
C1-N1-H1N	125(3)	C6-N2-C9	111.1(3)

C6-N2-H2N	128(4)	C39-C15-C40	109.9(3)
C9-N2-H2N	120(3)	C17-C16-N4	106.4(3)
C14-N3-C11	110.0(3)	C17-C16-C15	130.7(3)
C14-N3-H3N	122(3)	N4-C16-C15	122.9(3)
C11-N3-H3N	125(3)	C16-C17-C18	108.3(3)
C19-N4-C16	110.7(3)	C16-C17-H17	125.4
C19-N4-H4N	126(2)	C18-C17-H17	126.3
C16-N4-H4N	123(2)	C19-C18-C17	107.7(3)
C21-N5-C24	106.3(3)	C19-C18-H18	126.4
C29-N6-C26	110.0(3)	C17-C18-H18	125.9
C29-N6-H6N	121(3)	C18-C19-N4	107.0(3)
C26-N6-H6N	128(3)	C18-C19-C20	132.2(3)
C2-C1-N1	106.9(3)	N4-C19-C20	120.8(3)
C2-C1-C30	131.5(3)	C21-C20-C19	108.2(3)
N1-C1-C30	121.5(3)	C21-C20-C41	108.7(3)
C1-C2-C3	107.9(3)	C19-C20-C41	109.1(3)
C1-C2-H2	126.0	C21-C20-C42	111.6(3)
C3-C2-H2	126.1	C19-C20-C42	109.8(3)
C4-C3-C2	108.2(3)	C41-C20-C42	109.4(3)
C4-C3-H3	126.2	N5-C21-C22	110.8(3)
C2-C3-H3	125.6	N5-C21-C20	123.4(3)
C3-C4-N1	107.0(3)	C22-C21-C20	125.7(3)
C3-C4-C5	131.9(3)	C23-C22-C21	107.3(3)
N1-C4-C5	121.1(3)	C23-C22-H22	126.5
C6-C5-C4	107.7(3)	C21-C22-H22	126.2
C6-C5-C32	110.9(3)	C22-C23-C24	107.2(3)
C4-C5-C32	111.1(3)	C22-C23-H23	126.8
C6-C5-C31	108.1(3)	C24-C23-H23	126.1
C4-C5-C31	109.0(3)	C25-C24-N5	123.0(3)
C32-C5-C31	109.9(4)	C25-C24-C23	128.5(3)
N2-C6-C7	107.5(3)	N5-C24-C23	108.5(3)
N2-C6-C5	123.4(3)	C24-C25-C26	126.5(3)
C7-C6-C5	129.1(4)	C24-C25-C43	117.9(3)
C8-C7-C6	108.2(3)	C26-C25-C43	115.5(3)
C8-C7-H7	126.5	N6-C26-C27	106.4(3)
C6-C7-H7	125.3	N6-C26-C25	123.9(3)
C7-C8-C9	108.0(3)	C27-C26-C25	129.7(3)
C7-C8-H8	126.3	C28-C27-C26	107.9(3)
C9-C8-H8	125.7	C28-C27-H27	126.9
N2-C9-C10	125.5(3)	C26-C27-H27	125.2
N2-C9-C8	105.1(3)	C29-C28-C27	108.1(3)
C10-C9-C8	129.4(3)	C29-C28-H28	126.1
C9-C10-C11	129.1(3)	C27-C28-H28	125.8
C9-C10-C33	115.6(3)	N6-C29-C28	107.6(3)
C11-C10-C33	115.3(3)	N6-C29-C30	123.4(3)
N3-C11-C12	106.1(3)	C28-C29-C30	129.0(3)
N3-C11-C10	125.6(3)	C29-C30-C1	109.9(3)
C12-C11-C10	128.2(3)	C29-C30-C50	108.5(3)
C13-C12-C11	108.6(3)	C1-C30-C50	109.4(3)
C13-C12-H12	124.8	C29-C30-C49	110.4(3)
C11-C12-H12	126.5	C1-C30-C49	109.9(3)
C12-C13-C14	107.5(4)	C50-C30-C49	108.7(3)
C12-C13-H13	126.9	C5-C31-H31A	109.4
C14-C13-H13	125.5	C5-C31-H31B	109.1
N3-C14-C13	107.7(3)	H31A-C31-H31B	109.5
N3-C14-C15	123.5(3)	C5-C31-H31C	109.9
C13-C14-C15	128.8(3)	H31A-C31-H31C	109.5
C14-C15-C16	107.7(3)	H31B-C31-H31C	109.5
C14-C15-C39	109.1(3)	C5-C32-H32A	111.1
C16-C15-C39	108.9(3)	C5-C32-H32B	109.2
C14-C15-C40	110.8(3)	H32A-C32-H32B	109.5
C16-C15-C40	110.4(3)	C5-C32-H32C	108.1

H32A-C32-H32C	109.5	H41B-C41-H41C	109.5
H32B-C32-H32C	109.5	C20-C42-H42A	109.4
C38-C33-C34	116.6(3)	C20-C42-H42B	109.5
C38-C33-C10	123.1(3)	H42A-C42-H42B	109.5
C34-C33-C10	120.3(3)	C20-C42-H42C	109.6
F34-C34-C35	118.6(3)	H42A-C42-H42C	109.5
F34-C34-C33	119.6(3)	H42B-C42-H42C	109.5
C35-C34-C33	121.7(4)	C44-C43-C48	115.9(3)
F35-C35-C34	120.0(4)	C44-C43-C25	122.3(3)
F35-C35-C36	120.1(4)	C48-C43-C25	121.8(3)
C34-C35-C36	119.9(4)	F44-C44-C45	118.2(4)
F36-C36-C37	120.1(4)	F44-C44-C43	119.5(3)
F36-C36-C35	119.9(4)	C45-C44-C43	122.3(4)
C37-C36-C35	120.0(3)	F45-C45-C46	120.1(4)
F37-C37-C36	119.4(3)	F45-C45-C44	120.4(4)
F37-C37-C38	120.3(3)	C46-C45-C44	119.6(4)
C36-C37-C38	120.2(4)	F46-C46-C47	120.3(4)
F38-C38-C37	118.2(3)	F46-C46-C45	119.6(4)
F38-C38-C33	120.3(3)	C47-C46-C45	120.1(4)
C37-C38-C33	121.5(3)	F47-C47-C46	119.8(4)
C15-C39-H39A	109.5	F47-C47-C48	120.3(4)
C15-C39-H39B	109.9	C46-C47-C48	120.0(4)
H39A-C39-H39B	109.5	F48-C48-C47	118.1(4)
C15-C39-H39C	108.9	F48-C48-C43	119.7(3)
H39A-C39-H39C	109.5	C47-C48-C43	122.2(4)
H39B-C39-H39C	109.5	C30-C49-H49A	109.8
C15-C40-H40A	110.5	C30-C49-H49B	109.3
C15-C40-H40B	108.7	H49A-C49-H49B	109.5
H40A-C40-H40B	109.5	C30-C49-H49C	109.3
C15-C40-H40C	109.3	H49A-C49-H49C	109.5
H40A-C40-H40C	109.5	H49B-C49-H49C	109.5
H40B-C40-H40C	109.5	C30-C50-H50A	109.1
C20-C41-H41A	109.0	C30-C50-H50B	109.6
C20-C41-H41B	109.7	H50A-C50-H50B	109.5
H41A-C41-H41B	109.5	C30-C50-H50C	109.7
C20-C41-H41C	109.8	H50A-C50-H50C	109.5
H41A-C41-H41C	109.5	H50B-C50-H50C	109.5

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for (**1**·H^+)Cl^-. 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	47(1)	63(1)	31(1)	-4(1)	15(1)	-11(1)
Cl1A	34(4)	76(5)	45(5)	11(4)	-1(3)	16(4)
N1	35(2)	36(2)	23(2)	7(1)	5(1)	-4(1)
N2	43(2)	29(2)	33(2)	-3(2)	2(2)	1(1)
N3	36(2)	32(2)	35(2)	0(1)	15(2)	-4(1)
N4	35(2)	35(2)	22(2)	-4(1)	7(1)	0(1)
N5	38(2)	28(2)	34(2)	1(1)	4(1)	2(1)
N6	39(2)	27(2)	42(2)	-5(2)	14(2)	1(1)
C1	36(2)	26(2)	31(2)	0(2)	10(2)	-3(2)
C2	47(2)	36(2)	38(2)	4(2)	18(2)	-1(2)
C3	65(3)	35(2)	26(2)	7(2)	3(2)	-6(2)
C4	39(2)	31(2)	28(2)	4(2)	-2(2)	-3(2)
C5	44(2)	38(2)	46(2)	11(2)	-7(2)	-6(2)
C6	34(2)	37(2)	34(2)	4(2)	-2(2)	-4(2)
C7	49(2)	45(2)	39(2)	-5(2)	18(2)	-4(2)
C8	50(2)	32(2)	39(2)	-5(2)	17(2)	-3(2)
C9	37(2)	25(2)	32(2)	-3(2)	7(2)	-2(2)
C10	30(2)	33(2)	38(2)	1(2)	6(2)	-3(2)
C11	35(2)	31(2)	33(2)	-2(2)	6(2)	-2(2)
C12	53(3)	40(2)	45(3)	-5(2)	17(2)	-16(2)
C13	62(3)	53(3)	41(2)	-6(2)	29(2)	-16(2)
C14	32(2)	37(2)	29(2)	1(2)	5(2)	-1(2)
C15	28(2)	38(2)	35(2)	-3(2)	5(2)	1(2)
C16	30(2)	30(2)	33(2)	-2(2)	7(2)	-1(2)
C17	40(2)	37(2)	28(2)	0(2)	15(2)	-2(2)
C18	37(2)	43(2)	26(2)	3(2)	3(2)	-2(2)
C19	37(2)	26(2)	26(2)	0(2)	3(2)	1(2)
C20	32(2)	32(2)	35(2)	-2(2)	0(2)	4(2)
C21	27(2)	35(2)	33(2)	1(2)	1(2)	5(2)
C22	46(2)	36(2)	30(2)	4(2)	11(2)	2(2)
C23	45(2)	31(2)	38(2)	3(2)	10(2)	3(2)
C24	36(2)	32(2)	32(2)	1(2)	6(2)	3(2)
C25	33(2)	32(2)	34(2)	-2(2)	4(2)	-3(2)
C26	36(2)	29(2)	33(2)	-4(2)	8(2)	2(2)
C27	39(2)	32(2)	37(2)	-1(2)	8(2)	5(2)
C28	44(2)	33(2)	29(2)	-1(2)	10(2)	4(2)
C29	23(2)	32(2)	32(2)	0(2)	3(2)	1(1)
C30	32(2)	28(2)	47(2)	2(2)	8(2)	0(2)
C31	61(3)	82(3)	57(3)	34(3)	-23(2)	-19(3)
C32	36(2)	30(2)	105(4)	16(2)	11(2)	3(2)
C33	40(2)	36(2)	30(2)	0(2)	10(2)	0(2)
C34	51(2)	38(2)	35(2)	-2(2)	4(2)	-3(2)
C35	76(3)	38(2)	36(2)	10(2)	3(2)	14(2)
C36	77(3)	26(2)	42(2)	2(2)	23(2)	0(2)
C37	46(2)	37(2)	41(2)	-9(2)	15(2)	-9(2)
C38	35(2)	35(2)	34(2)	2(2)	11(2)	2(2)
C39	34(2)	58(3)	50(3)	-11(2)	4(2)	8(2)
C40	44(2)	34(2)	48(2)	-2(2)	-1(2)	2(2)
C41	41(2)	57(3)	51(3)	-18(2)	-11(2)	8(2)
C42	33(2)	37(2)	76(3)	-3(2)	16(2)	-1(2)

C43	44(2)	32(2)	36(2)	0(2)	16(2)	3(2)
C44	45(2)	42(2)	45(3)	2(2)	14(2)	8(2)
C45	57(3)	46(3)	55(3)	17(2)	26(2)	21(2)
C46	87(4)	30(2)	58(3)	5(2)	44(3)	10(2)
C47	83(3)	38(3)	36(2)	-5(2)	21(2)	-8(2)
C48	61(3)	35(2)	34(2)	3(2)	18(2)	0(2)
C49	48(2)	31(2)	58(3)	-4(2)	-15(2)	0(2)
C50	37(2)	52(3)	80(3)	18(2)	19(2)	-2(2)
F34	68(2)	56(2)	47(2)	-3(1)	-11(1)	6(1)
F35	116(2)	56(2)	54(2)	11(1)	-2(2)	27(2)
F36	108(2)	32(1)	67(2)	2(1)	27(2)	3(1)
F37	60(2)	45(1)	63(2)	-16(1)	16(1)	-14(1)
F38	43(1)	42(1)	47(1)	-7(1)	1(1)	1(1)
F44	47(1)	62(2)	62(2)	12(1)	0(1)	5(1)
F45	71(2)	66(2)	100(2)	37(2)	30(2)	34(1)
F46	133(3)	33(1)	98(2)	11(1)	69(2)	15(1)
F47	140(3)	44(2)	50(2)	-9(1)	26(2)	-28(2)
F48	68(2)	48(1)	41(1)	3(1)	-2(1)	-12(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for (**1**·H^+)Cl^-

	x	y	z	U(eq)
H1N	3160(40)	6137(17)	1200(20)	59(13)
H3N	5970(50)	6410(20)	20(30)	84(17)
H4N	4330(30)	6105(12)	-1149(17)	9(8)
H6N	1370(40)	6574(17)	-30(20)	48(12)
H2	958	5697	2682	47
H3	3310	5660	3291	51
H7	5042	7160	2831	52
H8	5610	7912	1972	47
H12	7784	7802	-500	54
H13	8159	7016	-1355	60
H17	6279	5826	-2735	41
H18	3904	5951	-3284	43
H22	2579	7325	-2673	44
H23	2088	8099	-1838	45
H27	218	8021	772	43
H28	-144	7208	1613	42
H31A	5496	6215	3429	106
H31B	6759	5991	3172	106
H31C	5694	5560	3328	106
H32A	5533	5147	2046	85
H32B	6615	5567	1888	85
H32C	5276	5558	1366	85
H39A	8164	6101	-1842	71
H39B	7978	5445	-1736	71
H39C	8828	5801	-1117	71
H40A	6617	5092	-820	65
H40B	5968	5535	-358	65
H40C	7467	5443	-197	65
H41A	1754	6435	-3258	78
H41B	533	6204	-2962	78
H41C	1552	5778	-3186	78
H42A	2152	5709	-1222	72
H42B	1803	5329	-1927	72
H42C	780	5754	-1704	72
H49A	540	5267	676	73
H49B	1385	5700	312	73
H49C	-125	5717	108	73
H50A	-782	6247	1816	83
H50B	-783	5604	1593	83
H50C	-1457	6056	1031	83
H2N	5960(40)	6446(18)	1120(20)	48(15)

Figure 1. View of molecule of $(\mathbf{1}\cdot H^{+})Cl^{-}$ showing the atom labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. The fluorine, chloride atoms and most hydrogen atoms have been removed for clarity.

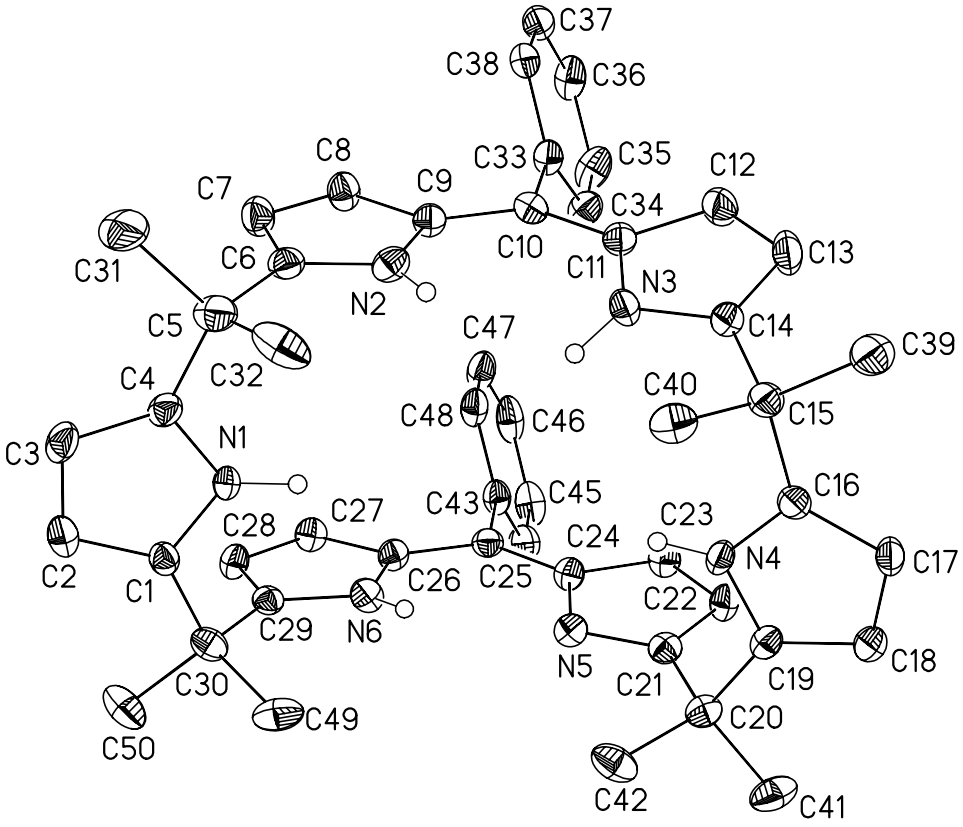


Figure 2. View of (**1**·H^+)Cl^- showing a partial atom labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. The pyrrole rings containing nitrogen atoms, N2 and N3, have a dihedral angle of 16.5(2)° while the pyrrole rings containing nitrogen atoms, N5 and N6, have a dihedral angle of 8.1(2)°.

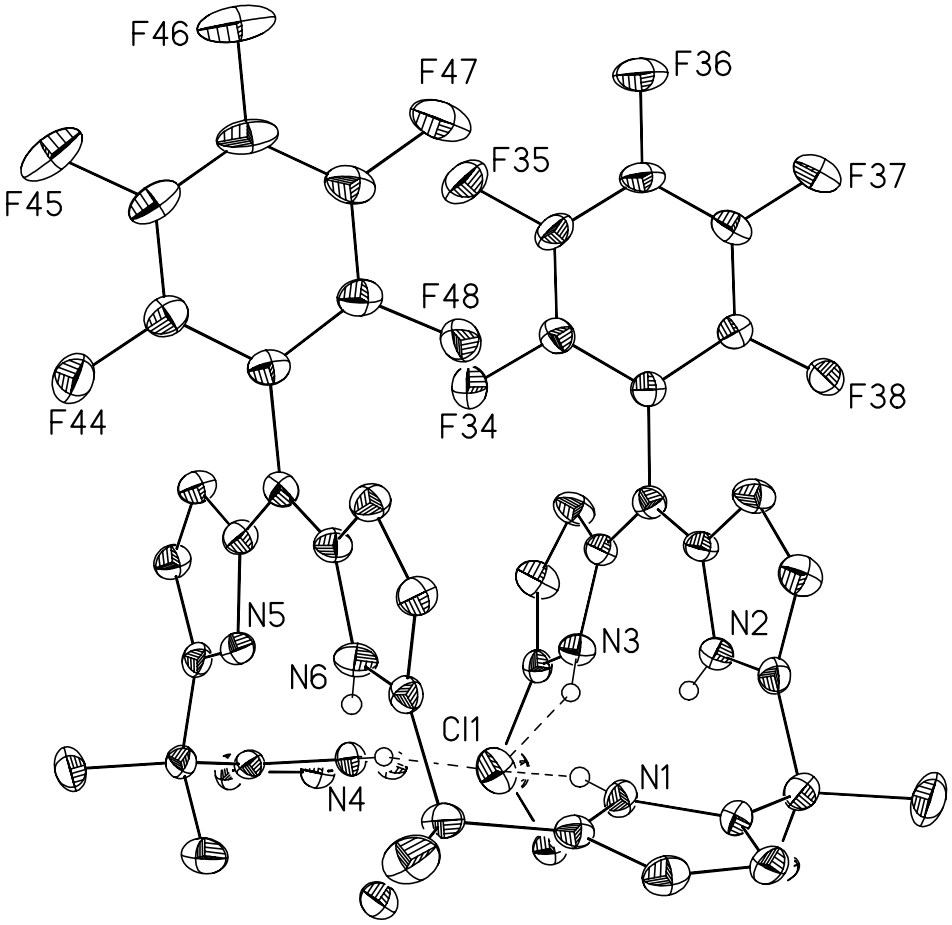


Figure 3. View of (**1**·H^+)Cl^- showing the H-bonding interactions and the disordered Cl^- ion. Thermal ellipsoids are scaled to the 30% probability level. The geometry of the H-bonding interactions are: N1-H1N···Cl1, N···Cl 3.184(3)Å, H···Cl 2.30(4)Å, N-H···Cl 165(4)°; N3-H3N···Cl1, N···Cl 3.105(3)Å, H···Cl 2.27(5)Å, N-H···Cl 158(4)°; N4-H4N···Cl1, N···Cl 3.229(4)Å, H···Cl 2.41(3)Å, N-H···Cl 173(3)°; N1-H1N···Cl1a, N···Cl 3.267(8)Å, H···Cl 2.41(4)Å, N-H···Cl 157(3)°; N4-H4N···Cl1a, N···Cl 3.204(8)Å, H···Cl 2.41(3)Å, N-H···Cl 161(3)°; N6-H6N···Cl1a, N···Cl 3.121(8)Å, H···Cl 2.35(4)Å, N-H···Cl 148(3)°.

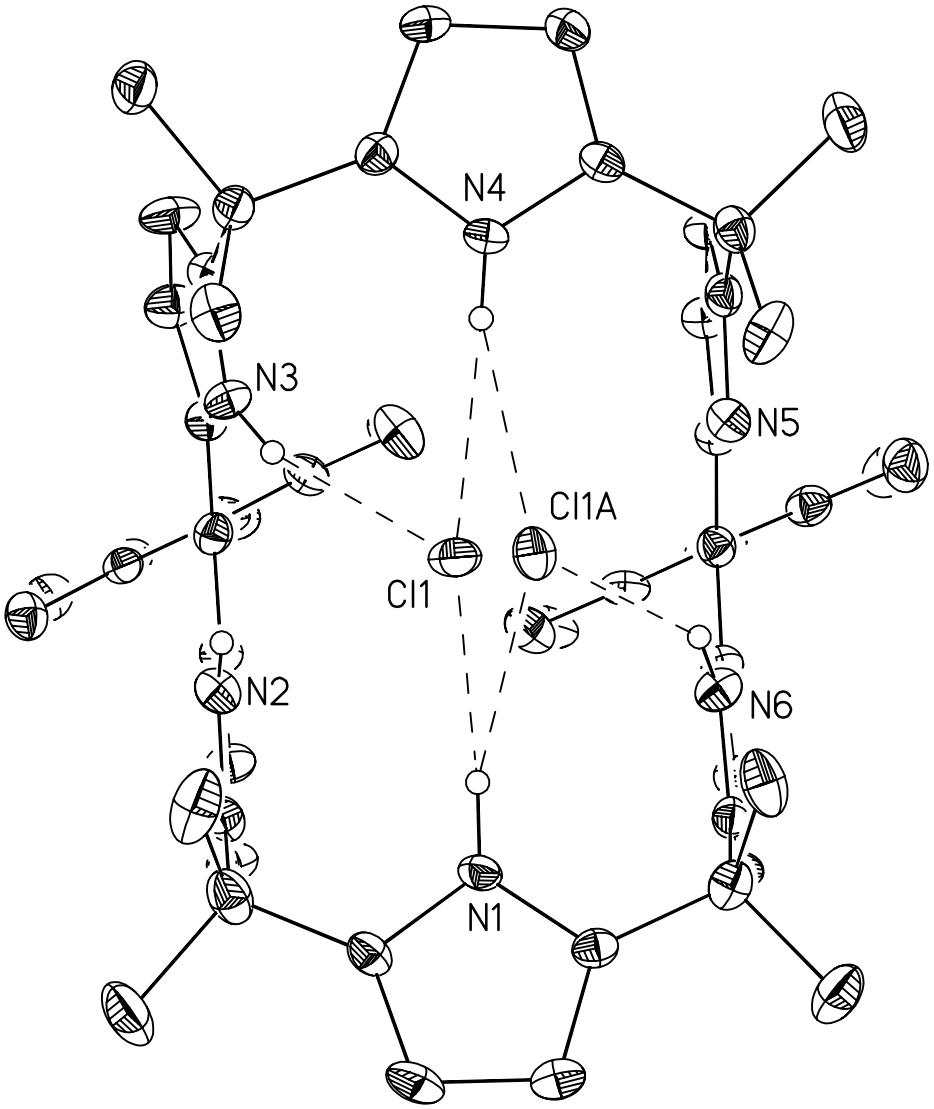
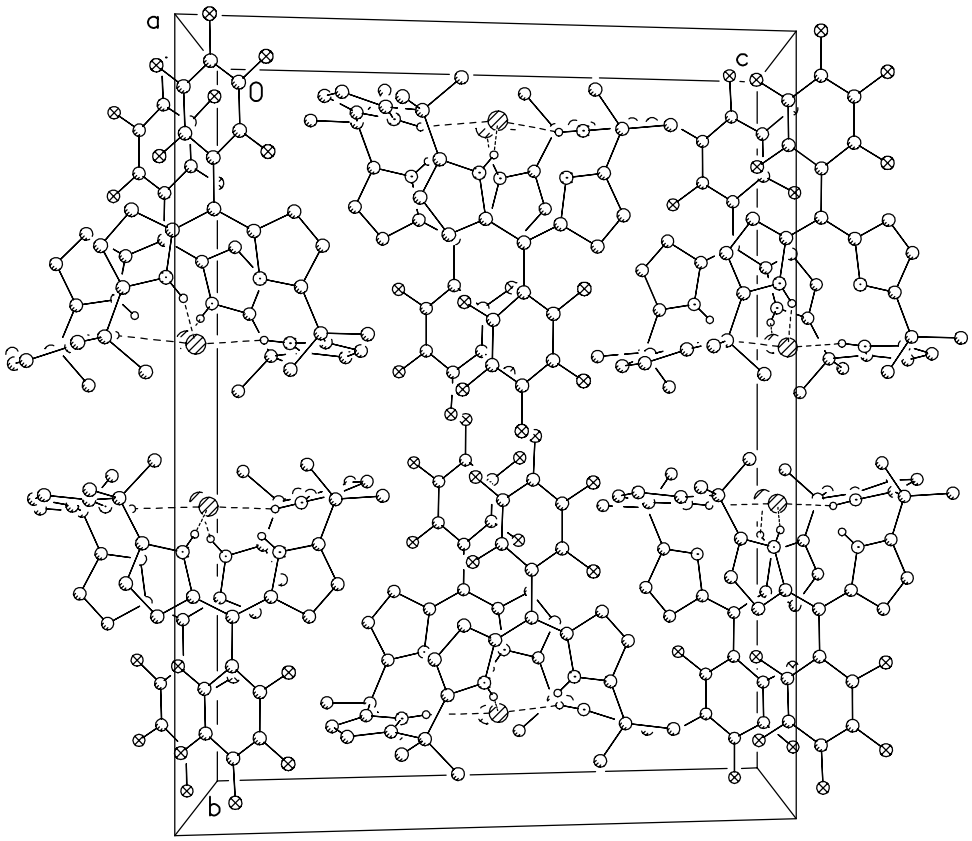


Figure 4. Unit cell packing diagram for (**1**·H^+)Cl^-. The view is approximately down the **a** axis. Dashed lines are indicative of a NH···Cl hydrogen bonding interaction.



Crystallographic Material for 5.

X-ray Experimental.

Table 1. Crystallographic Data for **5**.

Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of **5**.

Table 3. Bond Lengths (Å) and Angles (^{o}) for the non-hydrogen atoms of **5**.

Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of **5**.

Table 5. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of **5**.

Figure 1. View of **5** showing the atom labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. Most hydrogen atoms have been removed for clarity.

Figure 2. View of **5** showing the close packing of the perfluorophenyl rings between adjacent molecules. The molecules are related by an inversion center at ½ , ½ , 1.

Figure 3. View of the packing in **5** between adjacent dimers.

Figure 4. Unit cell packing diagram for **5**.

X-ray Experimental for ($C_{75}H_{60}N_9F_{15}$) – $2CHCl_3$ – CH_3OH: Crystals grew as orange plates by slow evaporation of a dichloromethane-hexane mixture. The data crystal was a plate with approximate dimensions; 0.20 x 0.17 x 0.04 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoKα radiation (λ = 0.71073Å). A total of 296 frames of data were collected using ω-scans with a scan range of 2° and a counting time of 196 seconds per frame. The data were collected at –120 °C using a 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.[2] The structure was solved by direct methods using SIR92[3] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[4] At the base of the macrocycle in the area encompassed by the pyrrole groups, there were two regions of electron density that appeared to due to the presence of a molecule of chloroform and methanol. The molecule of chloroform was at the larger end of the macrocycle near pyrrole rings containing N1 and N4. The molecule of methanol was at the narrower end of the macrocycle near the pyrrole ring containing N7. The two disordered molecules were within H-bonding distance to the pyrroles. Attempts to refine these molecules were unsuccessful. The contribution to the F^2_{obs} of these disordered solvent molecules was removed by use of the routine SQUEEZE in PLATON98.[8] 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). The hydrogen atoms on the pyrrole nitrogen atoms were observed in a ΔF map and refined with isotropic displacement parameters. The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0472*P)^2 + (3.274*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.160, with R(F) equal to 0.0706 and a goodness of fit, S, = 1.162. Definitions used for calculating R(F),$R_w(F^2)$ and the goodness of fit, S, are given below.[5] Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[6] All figures were generated using SHELXTL/PC.[7]

Table 1. Crystal data and structure refinement for **5**.

Empirical formula	C78 H66 Cl6 F15 N9 O	
Formula weight	1643.10	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 15.3277(2) Å	α= 107.008(1)°.
	b = 15.3634(2) Å	β= 108.942(1)°.
	c = 18.9058(2) Å	γ = 96.758(1)°.
Volume	3913.93(8) $Å^3$	
Z	2	
Density (calculated)	1.394 Mg/m^3	
Absorption coefficient	0.306 mm^{-1}	
F(000)	1684	
Crystal size	0.20 x 0.17 x 0.14 mm	
Theta range for data collection	2.92 to 27.47°.	
Index ranges	0<=h<=19, -19<=k<=19, -24<=l<=22	
Reflections collected	17780	
Completeness to theta = 27.47°	99.2 %	
Absorption correction	None	
Refinement method	Full-matrix-block least-squares on F^2	
Data / restraints / parameters	17780 / 0 / 942	
Goodness-of-fit on F^2	1.162	
Final R indices [I>2sigma(I)]	R1 = 0.0706, wR2 = 0.1457	
R indices (all data)	R1 = 0.1199, wR2 = 0.1601	
Largest diff. peak and hole	0.66 and -0.59 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl1A	822(1)	-2140(1)	5467(1)	88(1)
Cl2A	-255(1)	-3465(1)	3878(1)	88(1)
Cl3A	-195(1)	-3923(1)	5265(1)	116(1)
C1A	-189(2)	-3037(2)	4862(2)	48(1)
N1	5506(2)	-1601(2)	6971(1)	23(1)
N2	3026(2)	-1091(1)	6825(1)	25(1)
N3	2145(2)	105(2)	6195(1)	25(1)
N4	2861(2)	1898(2)	5155(1)	26(1)
N5	4867(2)	4334(2)	6716(1)	22(1)
N6	6786(1)	4981(1)	7474(1)	23(1)
N7	9020(2)	3942(2)	7723(1)	23(1)
N8	9090(2)	2279(1)	8924(1)	23(1)
N9	7923(2)	580(2)	8680(1)	24(1)
C1	6388(2)	-1771(2)	7280(1)	22(1)
C2	6238(2)	-2686(2)	7224(2)	27(1)
C3	5249(2)	-3072(2)	6880(2)	26(1)
C4	4805(2)	-2389(2)	6729(1)	23(1)
C5	3766(2)	-2398(2)	6359(2)	25(1)
C6	3489(2)	-1730(2)	6976(2)	25(1)
C7	3638(2)	-1716(2)	7770(2)	32(1)
C8	3223(2)	-1051(2)	8085(2)	28(1)
C9	2837(2)	-641(2)	7503(2)	25(1)
C10	2364(2)	72(2)	7535(1)	24(1)
C11	2062(2)	460(2)	6934(2)	25(1)
C12	1681(2)	1249(2)	6944(2)	33(1)
C13	1548(2)	1366(2)	6220(2)	37(1)
C14	1834(2)	636(2)	5765(2)	28(1)
C15	1750(2)	402(2)	4902(2)	31(1)
C16	1976(2)	1307(2)	4756(1)	26(1)
C17	1437(2)	1694(2)	4262(2)	28(1)
C18	2012(2)	2557(2)	4370(1)	24(1)
C19	2891(2)	2668(2)	4927(1)	22(1)
C20	3777(2)	3448(2)	5290(1)	26(1)
C21	3988(2)	3902(2)	6172(1)	25(1)
C22	3366(2)	4019(2)	6572(2)	38(1)
C23	3903(2)	4536(2)	7379(2)	35(1)
C24	4847(2)	4735(2)	7466(1)	24(1)
C25	5676(2)	5277(2)	8148(1)	22(1)
C26	6579(2)	5423(2)	8148(1)	22(1)
C27	7436(2)	6048(2)	8774(2)	29(1)
C28	8127(2)	5993(2)	8477(2)	33(1)
C29	7698(2)	5315(2)	7675(1)	23(1)
C30	8214(2)	5036(2)	7114(2)	25(1)
C31	9082(2)	4726(2)	7518(1)	21(1)
C32	10020(2)	5087(2)	7720(2)	25(1)
C33	10545(2)	4511(2)	8057(2)	25(1)
C34	9911(2)	3799(2)	8051(1)	21(1)
C35	10061(2)	2959(2)	8294(1)	24(1)
C36	9688(2)	2983(2)	8950(1)	21(1)
C37	9974(2)	3774(2)	9685(2)	27(1)
C38	9514(2)	3527(2)	10121(2)	25(1)
C39	8956(2)	2581(2)	9658(1)	23(1)
C40	8396(2)	2023(2)	9866(1)	23(1)
C41	7873(2)	1082(2)	9402(1)	26(1)
C42	7224(2)	495(2)	9529(2)	35(1)
C43	6895(2)	-356(2)	8886(2)	35(1)

C44	7340(2)	-287(2)	8366(2)	26(1)
C45	7285(2)	-1015(2)	7601(2)	25(1)
C46	3156(2)	-3389(2)	6109(2)	37(1)
C47	3569(2)	-2112(2)	5616(2)	33(1)
C48	2140(2)	464(2)	8263(2)	24(1)
C49	1221(2)	276(2)	8247(2)	26(1)
C50	1002(2)	621(2)	8910(2)	29(1)
C51	1707(2)	1191(2)	9620(2)	29(1)
C52	2621(2)	1408(2)	9661(2)	29(1)
C53	2831(2)	1042(2)	8990(2)	27(1)
C54	2415(3)	-219(2)	4724(2)	46(1)
C55	728(2)	-121(2)	4352(2)	43(1)
C56	4613(2)	3054(2)	5156(2)	32(1)
C57	3621(2)	4186(2)	4897(2)	34(1)
C58	5545(2)	5734(2)	8910(1)	25(1)
C59	5953(2)	5515(2)	9586(2)	34(1)
C60	5844(2)	5944(2)	10292(2)	44(1)
C61	5303(2)	6597(2)	10333(2)	46(1)
C62	4892(2)	6834(2)	9673(2)	38(1)
C63	5016(2)	6400(2)	8976(2)	29(1)
C64	8533(2)	5911(2)	6935(2)	38(1)
C65	7565(2)	4249(2)	6325(2)	34(1)
C66	9567(2)	2048(2)	7566(2)	31(1)
C67	11134(2)	3020(2)	8639(2)	32(1)
C68	8328(2)	2412(2)	10666(2)	27(1)
C69	8783(2)	2128(2)	11291(2)	33(1)
C70	8737(2)	2482(2)	12035(2)	44(1)
C71	8217(2)	3140(2)	12166(2)	45(1)
C72	7752(2)	3441(2)	11560(2)	42(1)
C73	7813(2)	3081(2)	10821(2)	32(1)
C74	8147(2)	-1455(2)	7792(2)	36(1)
C75	7301(2)	-557(2)	6980(2)	34(1)
F49	511(1)	-269(1)	7561(1)	38(1)
F50	113(1)	419(1)	8870(1)	43(1)
F51	1500(1)	1551(1)	10265(1)	45(1)
F52	3316(1)	1977(1)	10351(1)	44(1)
F53	3735(1)	1267(1)	9055(1)	40(1)
F59	6462(1)	4862(1)	9570(1)	51(1)
F60	6251(2)	5716(2)	10936(1)	71(1)
F61	5186(2)	7009(2)	11015(1)	73(1)
F62	4394(1)	7495(1)	9713(1)	57(1)
F63	4640(1)	6676(1)	8358(1)	39(1)
F69	9309(1)	1499(1)	11188(1)	49(1)
F70	9205(2)	2199(2)	12625(1)	70(1)
F71	8185(2)	3512(2)	12890(1)	69(1)
F72	7261(1)	4098(1)	11687(1)	63(1)
F73	7364(1)	3400(1)	10248(1)	47(1)

Table 3. Bond lengths [Å] and angles [°] for **5**.

Cl1A-C1A	1.741(4)	C15-C55	1.529(4)
Cl2A-C1A	1.746(3)	C16-C17	1.360(4)
Cl3A-C1A	1.743(4)	C17-C18	1.424(4)
C1A-H1A	0.96	C17-H17	0.96
N1-C4	1.372(3)	C18-C19	1.368(4)
N1-C1	1.376(3)	C18-H18	0.96
N1-H1N	0.74(3)	C19-C20	1.510(3)
N2-C6	1.322(3)	C20-C21	1.513(3)
N2-C9	1.404(3)	C20-C57	1.528(4)
N3-C14	1.335(3)	C20-C56	1.541(4)
N3-C11	1.397(3)	C21-C22	1.393(4)
N3-H3N	0.95(4)	C22-C23	1.400(4)
N4-C16	1.372(3)	C22-H22	0.96
N4-C19	1.374(3)	C23-C24	1.388(4)
N4-H4N	0.84(3)	C23-H23	0.96
N5-C21	1.343(3)	C24-C25	1.424(3)
N5-C24	1.384(3)	C25-C26	1.376(3)
N5-H5N	0.78(3)	C25-C58	1.495(3)
N6-C29	1.317(3)	C26-C27	1.441(4)
N6-C26	1.411(3)	C27-C28	1.351(4)
N7-C31	1.372(3)	C27-H27	0.96
N7-C34	1.378(3)	C28-C29	1.443(4)
N7-H7N	0.84(3)	C28-H28	0.96
N8-C36	1.312(3)	C29-C30	1.515(3)
N8-C39	1.418(3)	C30-C31	1.510(4)
N9-C44	1.355(3)	C30-C65	1.541(4)
N9-C41	1.389(3)	C30-C64	1.543(4)
N9-H9N	0.89(3)	C31-C32	1.360(3)
C1-C2	1.364(4)	C32-C33	1.415(4)
C1-C45	1.509(3)	C32-H32	0.96
C2-C3	1.413(4)	C33-C34	1.370(3)
C2-H2	0.96	C33-H33	0.96
C3-C4	1.363(4)	C34-C35	1.511(4)
C3-H3	0.96	C35-C36	1.517(3)
C4-C5	1.515(4)	C35-C67	1.540(4)
C5-C6	1.523(3)	C35-C66	1.541(3)
C5-C46	1.540(4)	C36-C37	1.446(3)
C5-C47	1.540(4)	C37-C38	1.349(4)
C6-C7	1.435(4)	C37-H37	0.96
C7-C8	1.360(4)	C38-C39	1.444(3)
C7-H7	0.96	C38-H38	0.96
C8-C9	1.425(4)	C39-C40	1.368(3)
C8-H8	0.96	C40-C41	1.425(3)
C9-C10	1.381(4)	C40-C68	1.498(3)
C10-C11	1.407(4)	C41-C42	1.390(4)
C10-C48	1.497(3)	C42-C43	1.399(4)
C11-C12	1.403(4)	C42-H42	0.96
C12-C13	1.385(4)	C43-C44	1.387(4)
C12-H12	0.96	C43-H43	0.96
C13-C14	1.409(4)	C44-C45	1.517(3)
C13-H13	0.96	C45-C75	1.540(4)
C14-C15	1.522(4)	C45-C74	1.541(4)
C15-C16	1.515(4)	C46-H46A	0.96
C15-C54	1.525(4)	C46-H46B	0.96

C46-H46C	0.96	C61-C62	1.377(5)
C47-H47A	0.96	C62-F62	1.339(4)
C47-H47B	0.96	C62-C63	1.377(4)
C47-H47C	0.96	C63-F63	1.338(3)
C48-C53	1.386(4)	C64-H64A	0.96
C48-C49	1.393(3)	C64-H64B	0.96
C49-F49	1.342(3)	C64-H64C	0.96
C49-C50	1.375(4)	C65-H65A	0.96
C50-F50	1.334(3)	C65-H65B	0.96
C50-C51	1.373(4)	C65-H65C	0.96
C51-F51	1.339(3)	C66-H66A	0.96
C51-C52	1.372(4)	C66-H66B	0.96
C52-F52	1.346(3)	C66-H66C	0.96
C52-C53	1.386(4)	C67-H67A	0.96
C53-F53	1.344(3)	C67-H67B	0.96
C54-H54A	0.96	C67-H67C	0.96
C54-H54B	0.96	C68-C69	1.383(4)
C54-H54C	0.96	C68-C73	1.389(4)
C55-H55A	0.96	C69-F69	1.340(3)
C55-H55B	0.96	C69-C70	1.379(4)
C55-H55C	0.96	C70-F70	1.337(4)
C56-H56A	0.96	C70-C71	1.376(5)
C56-H56B	0.96	C71-F71	1.341(3)
C56-H56C	0.96	C71-C72	1.374(5)
C57-H57A	0.96	C72-F72	1.341(4)
C57-H57B	0.96	C72-C73	1.382(4)
C57-H57C	0.96	C73-F73	1.339(3)
C58-C63	1.381(4)	C74-H74A	0.96
C58-C59	1.386(4)	C74-H74B	0.96
C59-F59	1.341(3)	C74-H74C	0.96
C59-C60	1.378(4)	C75-H75A	0.96
C60-F60	1.343(4)	C75-H75B	0.96
C60-C61	1.375(5)	C75-H75C	0.96
C61-F61	1.341(3)		

Cl1A-C1A-Cl3A	109.3(2)	C31-N7-H7N	128(2)
Cl1A-C1A-Cl2A	111.40(19)	C34-N7-H7N	122(2)
Cl3A-C1A-Cl2A	111.50(19)	C36-N8-C39	105.5(2)
Cl1A-C1A-H1A	108.3	C44-N9-C41	110.4(2)
Cl3A-C1A-H1A	108.2	C44-N9-H9N	127.1(19)
Cl2A-C1A-H1A	108.1	C41-N9-H9N	122(2)
C4-N1-C1	110.7(2)	C2-C1-N1	106.4(2)
C4-N1-H1N	121.5(19)	C2-C1-C45	131.9(2)
C1-N1-H1N	127.6(19)	N1-C1-C45	121.6(2)
C6-N2-C9	107.8(2)	C1-C2-C3	108.1(2)
C14-N3-C11	109.1(2)	C1-C2-H2	125.6
C14-N3-H3N	133(3)	C3-C2-H2	126.3
C11-N3-H3N	118(3)	C4-C3-C2	108.1(2)
C16-N4-C19	110.2(2)	C4-C3-H3	125.4
C16-N4-H4N	127(2)	C2-C3-H3	126.5
C19-N4-H4N	123(2)	C3-C4-N1	106.6(2)
C21-N5-C24	110.8(2)	C3-C4-C5	131.8(2)
C21-N5-H5N	130.7(19)	N1-C4-C5	121.6(2)
C24-N5-H5N	118.5(19)	C4-C5-C6	109.2(2)
C29-N6-C26	105.9(2)	C4-C5-C46	109.6(2)
C31-N7-C34	110.4(2)	C6-C5-C46	108.1(2)

C4-C5-C47	110.8(2)	C22-C21-C20	129.7(2)
C6-C5-C47	110.2(2)	C21-C22-C23	107.9(2)
C46-C5-C47	108.8(2)	C21-C22-H22	126.2
N2-C6-C7	110.3(2)	C23-C22-H22	125.9
N2-C6-C5	122.8(2)	C24-C23-C22	107.5(2)
C7-C6-C5	126.9(2)	C24-C23-H23	125.8
C8-C7-C6	106.7(2)	C22-C23-H23	126.7
C8-C7-H7	126.4	N5-C24-C23	106.6(2)
C6-C7-H7	127.0	N5-C24-C25	122.6(2)
C7-C8-C9	107.7(2)	C23-C24-C25	130.7(2)
C7-C8-H8	126.9	C26-C25-C24	124.6(2)
C9-C8-H8	125.4	C26-C25-C58	118.1(2)
C10-C9-N2	122.0(2)	C24-C25-C58	117.3(2)
C10-C9-C8	130.4(2)	C25-C26-N6	122.5(2)
N2-C9-C8	107.6(2)	C25-C26-C27	128.4(2)
C9-C10-C11	125.4(2)	N6-C26-C27	109.0(2)
C9-C10-C48	117.8(2)	C28-C27-C26	106.6(2)
C11-C10-C48	116.8(2)	C28-C27-H27	126.7
N3-C11-C12	107.3(2)	C26-C27-H27	126.6
N3-C11-C10	122.8(2)	C27-C28-C29	106.7(2)
C12-C11-C10	129.8(2)	C27-C28-H28	127.5
C13-C12-C11	107.3(2)	C29-C28-H28	125.8
C13-C12-H12	126.3	N6-C29-C28	111.8(2)
C11-C12-H12	126.4	N6-C29-C30	123.3(2)
C12-C13-C14	107.5(3)	C28-C29-C30	124.9(2)
C12-C13-H13	126.7	C31-C30-C29	109.6(2)
C14-C13-H13	125.8	C31-C30-C65	110.2(2)
N3-C14-C13	108.8(2)	C29-C30-C65	111.5(2)
N3-C14-C15	123.0(2)	C31-C30-C64	108.9(2)
C13-C14-C15	128.1(3)	C29-C30-C64	107.3(2)
C16-C15-C14	108.9(2)	C65-C30-C64	109.3(2)
C16-C15-C54	110.8(2)	C32-C31-N7	106.9(2)
C14-C15-C54	110.8(2)	C32-C31-C30	130.8(2)
C16-C15-C55	108.9(2)	N7-C31-C30	122.2(2)
C14-C15-C55	108.6(2)	C31-C32-C33	108.3(2)
C54-C15-C55	108.9(2)	C31-C32-H32	126.0
C17-C16-N4	107.5(2)	C33-C32-H32	125.6
C17-C16-C15	131.6(2)	C34-C33-C32	107.6(2)
N4-C16-C15	120.9(2)	C34-C33-H33	126.0
C16-C17-C18	107.6(2)	C32-C33-H33	126.4
C16-C17-H17	126.0	C33-C34-N7	106.8(2)
C18-C17-H17	126.4	C33-C34-C35	130.9(2)
C19-C18-C17	107.7(2)	N7-C34-C35	122.2(2)
C19-C18-H18	125.7	C34-C35-C36	109.3(2)
C17-C18-H18	126.6	C34-C35-C67	108.6(2)
C18-C19-N4	107.0(2)	C36-C35-C67	107.8(2)
C18-C19-C20	131.6(2)	C34-C35-C66	110.3(2)
N4-C19-C20	121.4(2)	C36-C35-C66	111.2(2)
C19-C20-C21	108.6(2)	C67-C35-C66	109.5(2)
C19-C20-C57	110.0(2)	N8-C36-C37	112.3(2)
C21-C20-C57	108.7(2)	N8-C36-C35	123.8(2)
C19-C20-C56	110.0(2)	C37-C36-C35	123.9(2)
C21-C20-C56	111.0(2)	C38-C37-C36	106.6(2)
C57-C20-C56	108.6(2)	C38-C37-H37	127.3
N5-C21-C22	107.3(2)	C36-C37-H37	126.1
N5-C21-C20	122.9(2)	C37-C38-C39	106.6(2)

C37-C38-H38	126.7	C15-C54-H54A	108.7
C39-C38-H38	126.7	C15-C54-H54B	109.2
C40-C39-N8	123.0(2)	H54A-C54-H54B	109.5
C40-C39-C38	127.9(2)	C15-C54-H54C	110.5
N8-C39-C38	109.0(2)	H54A-C54-H54C	109.5
C39-C40-C41	126.5(2)	H54B-C54-H54C	109.5
C39-C40-C68	118.2(2)	C15-C55-H55A	109.3
C41-C40-C68	115.3(2)	C15-C55-H55B	109.7
N9-C41-C42	106.3(2)	H55A-C55-H55B	109.5
N9-C41-C40	123.2(2)	C15-C55-H55C	109.4
C42-C41-C40	130.5(2)	H55A-C55-H55C	109.5
C41-C42-C43	108.0(2)	H55B-C55-H55C	109.5
C41-C42-H42	125.8	C20-C56-H56A	109.9
C43-C42-H42	126.1	C20-C56-H56B	108.8
C44-C43-C42	107.8(2)	H56A-C56-H56B	109.5
C44-C43-H43	126.0	C20-C56-H56C	109.7
C42-C43-H43	126.2	H56A-C56-H56C	109.5
N9-C44-C43	107.5(2)	H56B-C56-H56C	109.5
N9-C44-C45	122.4(2)	C20-C57-H57A	109.4
C43-C44-C45	130.0(2)	C20-C57-H57B	109.6
C1-C45-C44	109.1(2)	H57A-C57-H57B	109.5
C1-C45-C75	110.5(2)	C20-C57-H57C	109.4
C44-C45-C75	110.3(2)	H57A-C57-H57C	109.5
C1-C45-C74	109.0(2)	H57B-C57-H57C	109.5
C44-C45-C74	108.8(2)	C63-C58-C59	116.6(2)
C75-C45-C74	109.2(2)	C63-C58-C25	121.6(2)
C5-C46-H46A	109.1	C59-C58-C25	121.8(2)
C5-C46-H46B	109.7	F59-C59-C60	117.6(3)
H46A-C46-H46B	109.5	F59-C59-C58	120.5(2)
C5-C46-H46C	109.7	C60-C59-C58	121.9(3)
H46A-C46-H46C	109.5	F60-C60-C61	119.8(3)
H46B-C46-H46C	109.5	F60-C60-C59	120.3(3)
C5-C47-H47A	108.9	C61-C60-C59	119.9(3)
C5-C47-H47B	109.3	F61-C61-C60	120.1(3)
H47A-C47-H47B	109.5	F61-C61-C62	120.1(3)
C5-C47-H47C	110.2	C60-C61-C62	119.7(3)
H47A-C47-H47C	109.5	F62-C62-C63	120.3(3)
H47B-C47-H47C	109.5	F62-C62-C61	120.4(3)
C53-C48-C49	115.9(2)	C63-C62-C61	119.3(3)
C53-C48-C10	122.2(2)	F63-C63-C62	117.6(3)
C49-C48-C10	121.9(2)	F63-C63-C58	119.7(2)
F49-C49-C50	117.8(2)	C62-C63-C58	122.6(3)
F49-C49-C48	119.4(2)	C30-C64-H64A	109.5
C50-C49-C48	122.9(2)	C30-C64-H64B	109.2
F50-C50-C51	119.5(2)	H64A-C64-H64B	109.5
F50-C50-C49	121.0(2)	C30-C64-H64C	109.7
C51-C50-C49	119.4(2)	H64A-C64-H64C	109.5
F51-C51-C52	120.1(2)	H64B-C64-H64C	109.5
F51-C51-C50	120.1(2)	C30-C65-H65A	110.2
C52-C51-C50	119.7(2)	C30-C65-H65B	108.3
F52-C52-C51	120.2(2)	H65A-C65-H65B	109.5
F52-C52-C53	119.8(2)	C30-C65-H65C	109.9
C51-C52-C53	120.0(2)	H65A-C65-H65C	109.5
F53-C53-C52	118.2(2)	H65B-C65-H65C	109.5
F53-C53-C48	119.7(2)	C35-C66-H66A	110.0
C52-C53-C48	122.0(2)	C35-C66-H66B	110.1

H66A-C66-H66B	109.5
C35-C66-H66C	108.4
H66A-C66-H66C	109.5
H66B-C66-H66C	109.5
C35-C67-H67A	109.8
C35-C67-H67B	108.6
H67A-C67-H67B	109.5
C35-C67-H67C	110.0
H67A-C67-H67C	109.5
H67B-C67-H67C	109.5
C69-C68-C73	116.6(2)
C69-C68-C40	121.5(2)
C73-C68-C40	121.9(2)
F69-C69-C70	117.8(3)
F69-C69-C68	119.7(2)
C70-C69-C68	122.5(3)
F70-C70-C71	120.3(3)
F70-C70-C69	120.4(3)
C71-C70-C69	119.3(3)
F71-C71-C72	119.8(3)
F71-C71-C70	120.2(3)
C72-C71-C70	120.0(3)
F72-C72-C71	120.4(3)
F72-C72-C73	119.8(3)
C71-C72-C73	119.8(3)
F73-C73-C72	118.2(3)
F73-C73-C68	120.0(2)
C72-C73-C68	121.8(3)
C45-C74-H74A	109.1
C45-C74-H74B	110.4
H74A-C74-H74B	109.5
C45-C74-H74C	108.9
H74A-C74-H74C	109.5
H74B-C74-H74C	109.5
C45-C75-H75A	108.8
C45-C75-H75B	109.9
H75A-C75-H75B	109.5
C45-C75-H75C	109.7
H75A-C75-H75C	109.5
H75B-C75-H75C	109.5

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5**. 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}
Cl1A	59(1)	92(1)	83(1)	-6(1)	32(1)	-9(1)
Cl2A	102(1)	108(1)	53(1)	7(1)	42(1)	39(1)
Cl3A	185(2)	76(1)	152(1)	72(1)	105(1)	67(1)
C1A	54(2)	45(2)	49(2)	15(2)	26(2)	19(2)
N1	31(1)	13(1)	27(1)	7(1)	14(1)	8(1)
N2	32(1)	21(1)	23(1)	5(1)	14(1)	9(1)
N3	37(1)	20(1)	24(1)	7(1)	17(1)	9(1)
N4	31(1)	26(1)	20(1)	8(1)	7(1)	6(1)
N5	21(1)	26(1)	20(1)	6(1)	10(1)	4(1)
N6	25(1)	22(1)	23(1)	7(1)	9(1)	6(1)
N7	20(1)	22(1)	28(1)	10(1)	11(1)	1(1)
N8	28(1)	23(1)	19(1)	7(1)	11(1)	5(1)
N9	29(1)	23(1)	21(1)	5(1)	13(1)	3(1)
C1	29(1)	20(1)	18(1)	6(1)	10(1)	8(1)
C2	32(2)	25(1)	29(1)	11(1)	14(1)	12(1)
C3	33(2)	18(1)	31(1)	10(1)	16(1)	5(1)
C4	29(1)	19(1)	20(1)	4(1)	13(1)	4(1)
C5	26(1)	23(1)	24(1)	4(1)	11(1)	7(1)
C6	26(1)	22(1)	27(1)	6(1)	11(1)	5(1)
C7	37(2)	33(2)	30(2)	14(1)	15(1)	15(1)
C8	36(2)	30(2)	24(1)	8(1)	16(1)	11(1)
C9	32(1)	22(1)	23(1)	6(1)	13(1)	6(1)
C10	31(1)	19(1)	23(1)	3(1)	14(1)	2(1)
C11	35(2)	21(1)	24(1)	6(1)	17(1)	7(1)
C12	52(2)	25(1)	27(1)	7(1)	22(1)	14(1)
C13	61(2)	26(2)	34(2)	13(1)	26(1)	19(1)
C14	41(2)	21(1)	25(1)	8(1)	18(1)	3(1)
C15	47(2)	23(1)	25(1)	8(1)	19(1)	5(1)
C16	36(2)	23(1)	19(1)	4(1)	13(1)	3(1)
C17	29(1)	30(1)	23(1)	7(1)	11(1)	2(1)
C18	30(1)	24(1)	22(1)	10(1)	10(1)	8(1)
C19	27(1)	21(1)	20(1)	6(1)	12(1)	5(1)
C20	28(1)	29(1)	19(1)	5(1)	10(1)	3(1)
C21	25(1)	26(1)	22(1)	5(1)	9(1)	2(1)
C22	26(1)	50(2)	26(1)	-1(1)	13(1)	-4(1)
C23	32(2)	42(2)	21(1)	-1(1)	13(1)	0(1)
C24	28(1)	24(1)	20(1)	6(1)	11(1)	5(1)
C25	30(1)	18(1)	20(1)	7(1)	10(1)	6(1)
C26	27(1)	20(1)	19(1)	6(1)	8(1)	8(1)
C27	28(1)	31(2)	20(1)	5(1)	5(1)	9(1)
C28	25(1)	37(2)	27(1)	5(1)	5(1)	4(1)
C29	23(1)	23(1)	24(1)	10(1)	7(1)	7(1)
C30	24(1)	28(1)	24(1)	12(1)	9(1)	7(1)
C31	26(1)	21(1)	18(1)	8(1)	11(1)	5(1)
C32	26(1)	19(1)	29(1)	9(1)	12(1)	2(1)
C33	20(1)	23(1)	29(1)	9(1)	8(1)	3(1)
C34	23(1)	21(1)	19(1)	4(1)	10(1)	6(1)
C35	30(1)	21(1)	23(1)	7(1)	15(1)	7(1)
C36	23(1)	19(1)	24(1)	9(1)	10(1)	8(1)
C37	31(1)	19(1)	29(1)	5(1)	13(1)	3(1)

C38	34(1)	20(1)	23(1)	6(1)	14(1)	8(1)
C39	26(1)	22(1)	20(1)	5(1)	9(1)	7(1)
C40	26(1)	24(1)	20(1)	7(1)	10(1)	6(1)
C41	33(1)	23(1)	20(1)	4(1)	13(1)	3(1)
C42	44(2)	32(2)	25(1)	3(1)	20(1)	-3(1)
C43	46(2)	28(2)	29(2)	5(1)	20(1)	-7(1)
C44	28(1)	22(1)	26(1)	7(1)	10(1)	2(1)
C45	28(1)	23(1)	22(1)	3(1)	12(1)	2(1)
C46	34(2)	28(2)	42(2)	-2(1)	18(1)	2(1)
C47	37(2)	39(2)	24(1)	7(1)	15(1)	18(1)
C50	30(1)	27(1)	36(2)	13(1)	20(1)	9(1)
C51	46(2)	25(1)	25(1)	7(1)	23(1)	10(1)
C52	39(2)	22(1)	21(1)	2(1)	12(1)	3(1)
C53	30(1)	25(1)	27(1)	6(1)	16(1)	3(1)
C54	86(3)	32(2)	41(2)	19(1)	43(2)	23(2)
C55	64(2)	32(2)	26(2)	6(1)	17(1)	-8(2)
C56	28(1)	38(2)	25(1)	3(1)	12(1)	6(1)
C57	42(2)	29(2)	26(1)	8(1)	12(1)	1(1)
C58	27(1)	24(1)	20(1)	4(1)	10(1)	0(1)
C59	40(2)	31(2)	28(2)	9(1)	10(1)	3(1)
C60	56(2)	47(2)	22(2)	13(1)	10(1)	-7(2)
C61	53(2)	48(2)	25(2)	-4(1)	24(1)	-13(2)
C62	31(2)	32(2)	41(2)	-5(1)	21(1)	-1(1)
C63	28(1)	32(2)	21(1)	4(1)	10(1)	3(1)
C64	39(2)	43(2)	45(2)	28(2)	19(1)	15(1)
C65	30(2)	47(2)	21(1)	8(1)	9(1)	9(1)
C66	48(2)	22(1)	27(1)	6(1)	21(1)	7(1)
C67	36(2)	32(2)	41(2)	19(1)	22(1)	19(1)
C68	31(1)	23(1)	24(1)	4(1)	15(1)	-1(1)
C69	44(2)	27(2)	28(2)	8(1)	17(1)	2(1)
C70	61(2)	40(2)	23(2)	8(1)	16(1)	-12(2)
C71	55(2)	42(2)	27(2)	-7(1)	29(2)	-15(2)
C72	31(2)	38(2)	43(2)	-9(1)	24(1)	-5(1)
C73	28(1)	34(2)	30(2)	5(1)	13(1)	3(1)
C74	32(2)	37(2)	34(2)	4(1)	13(1)	7(1)
C75	43(2)	31(2)	27(1)	6(1)	20(1)	-1(1)
F49	32(1)	41(1)	29(1)	2(1)	8(1)	2(1)
F50	35(1)	49(1)	48(1)	11(1)	27(1)	7(1)
F51	62(1)	46(1)	32(1)	4(1)	33(1)	9(1)
F52	49(1)	40(1)	25(1)	-6(1)	14(1)	-8(1)
F53	34(1)	41(1)	35(1)	-1(1)	17(1)	-4(1)
F59	67(1)	52(1)	40(1)	28(1)	14(1)	22(1)
F60	96(2)	83(2)	26(1)	27(1)	16(1)	-5(1)
F61	89(2)	75(2)	37(1)	-15(1)	45(1)	-17(1)
F62	45(1)	47(1)	64(1)	-12(1)	31(1)	9(1)
F63	43(1)	39(1)	37(1)	12(1)	15(1)	20(1)
F69	76(1)	39(1)	38(1)	19(1)	19(1)	24(1)
F70	113(2)	64(1)	29(1)	23(1)	22(1)	3(1)
F71	81(2)	74(1)	36(1)	-10(1)	41(1)	-18(1)
F72	46(1)	58(1)	74(1)	-11(1)	39(1)	10(1)
F73	42(1)	53(1)	48(1)	14(1)	19(1)	23(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **5**.

	x	y	z	U(eq)
H1A	-736	-2790	4861	57
H2	6724	-3009	7383	32
H3	4936	-3700	6778	32
H7	3961	-2104	8028	38
H8	3205	-869	8610	34
H12	1530	1630	7370	39
H13	1315	1857	6057	44
H17	784	1434	3909	33
H18	1823	2988	4103	29
H22	2689	3787	6337	46
H23	3668	4714	7801	41
H27	7500	6432	9299	34
H28	8779	6333	8740	39
H32	10282	5630	7636	29
H33	11223	4608	8265	29
H37	10412	4357	9830	32
H38	9545	3907	10636	30
H42	7030	650	9976	42
H43	6444	-894	8818	42
H46A	3272	-3567	6571	56
H46B	2496	-3392	5882	56
H46C	3321	-3824	5721	56
H47A	3948	-1495	5771	49
H47B	3734	-2546	5228	49
H47C	2910	-2115	5389	49
H54A	3056	113	5068	69
H54B	2262	-783	4825	69
H54C	2352	-373	4176	69
H55A	306	270	4456	65
H55B	663	-275	3805	65
H55C	573	-685	4453	65
H56A	5177	3549	5389	48
H56B	4716	2592	5404	48
H56C	4466	2770	4594	48
H57A	3102	4438	4977	50
H57B	4186	4680	5131	50
H57C	3476	3902	4335	50
H64A	8939	6402	7426	57
H64B	8873	5758	6587	57
H64C	7987	6118	6682	57
H65A	7903	4087	5975	51
H65B	7367	3712	6446	51
H65C	7018	4451	6067	51
H66A	8899	2010	7343	47
H66B	9831	2032	7167	47
H66C	9667	1527	7740	47
H67A	11446	3592	9087	48
H67B	11230	2496	8809	48
H67C	11393	3001	8237	48

H74A	8136	-1739	8180	54
H74B	8128	-1921	7317	54
H74C	8718	-971	8007	54
H75A	6757	-293	6858	51
H75B	7869	-70	7192	51
H75C	7279	-1020	6502	51
H5N	5357(19)	4375(18)	6672(15)	24(7)
H4N	3320(20)	1840(20)	5509(18)	40(8)
H9N	8270(20)	830(20)	8460(18)	42(8)
H1N	5394(16)	-1151(18)	6952(14)	14(6)
H3N	2430(30)	-420(30)	6110(20)	89(13)
H7N	8520(20)	3560(20)	7640(18)	43(8)

Figure 1. View of **5** showing the atom labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. Most hydrogen atoms have been removed for clarity.

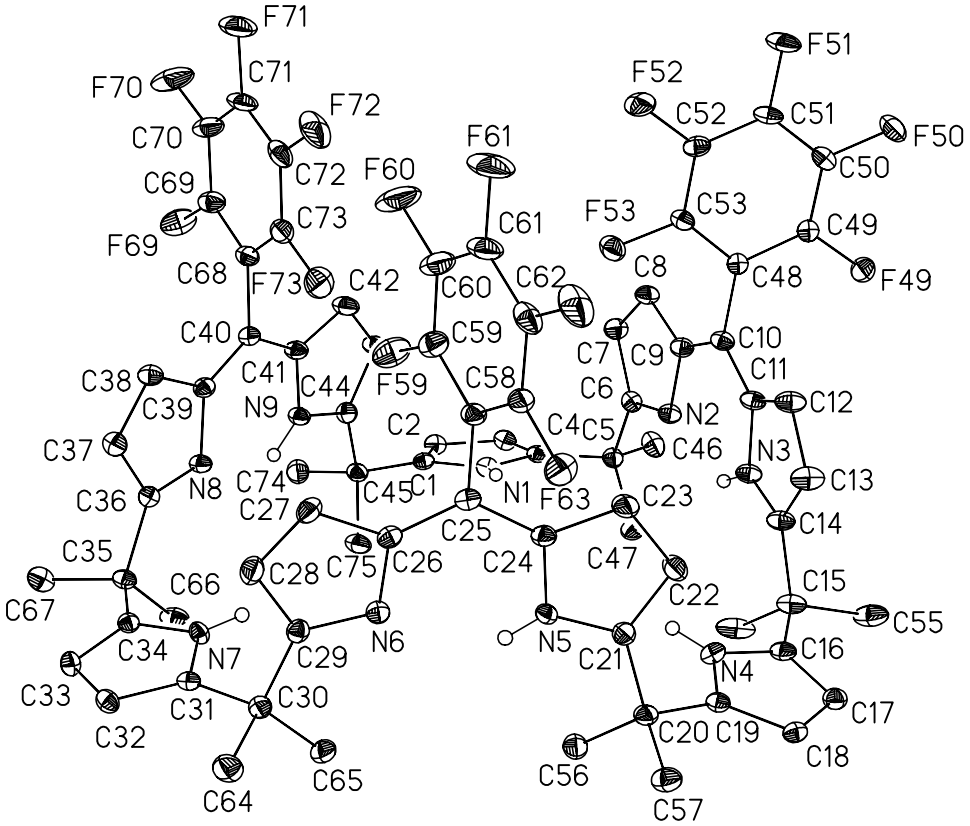


Figure 2. View of **5** showing the close packing of the perfluorophenyl rings between adjacent molecules. The molecules are related by an inversion center at ½ , ½ , 1.

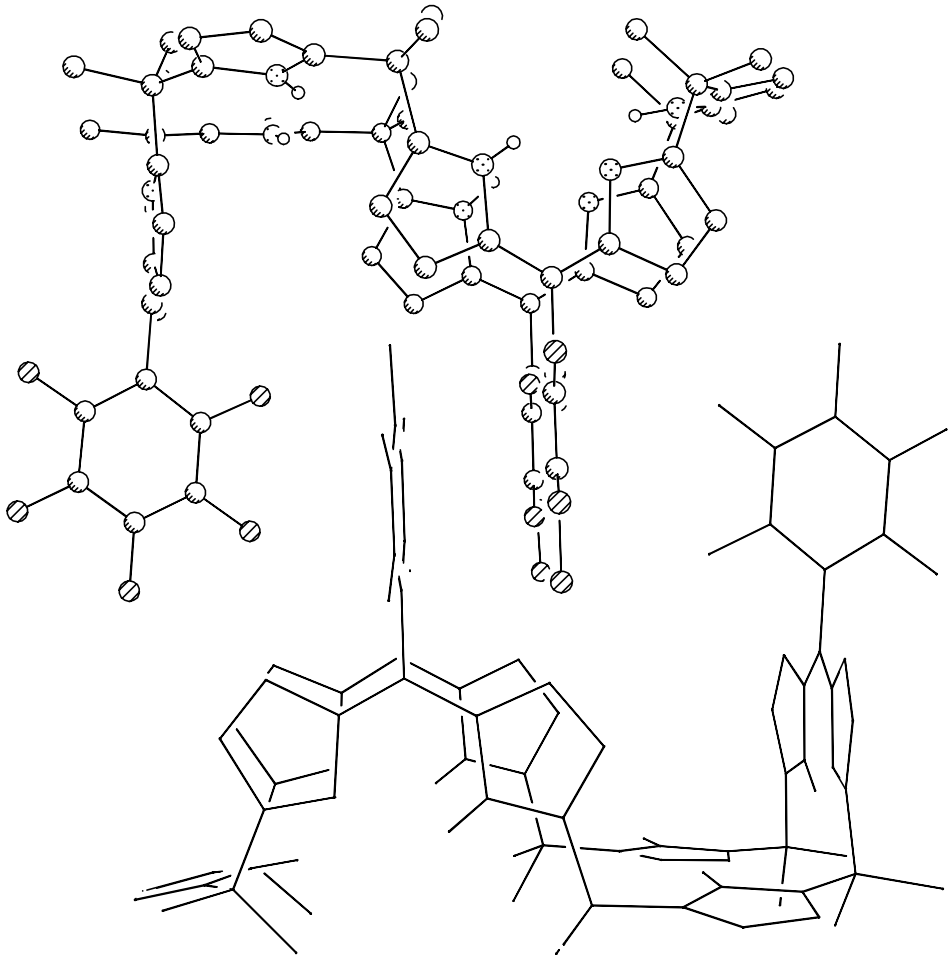


Figure 3. View of the packing in **5** between adjacent dimers.

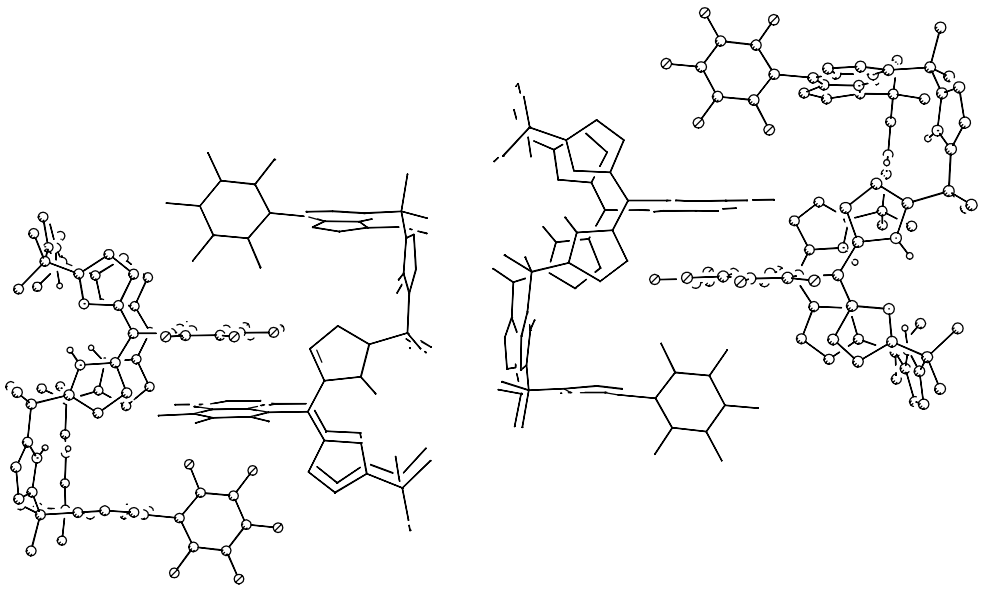
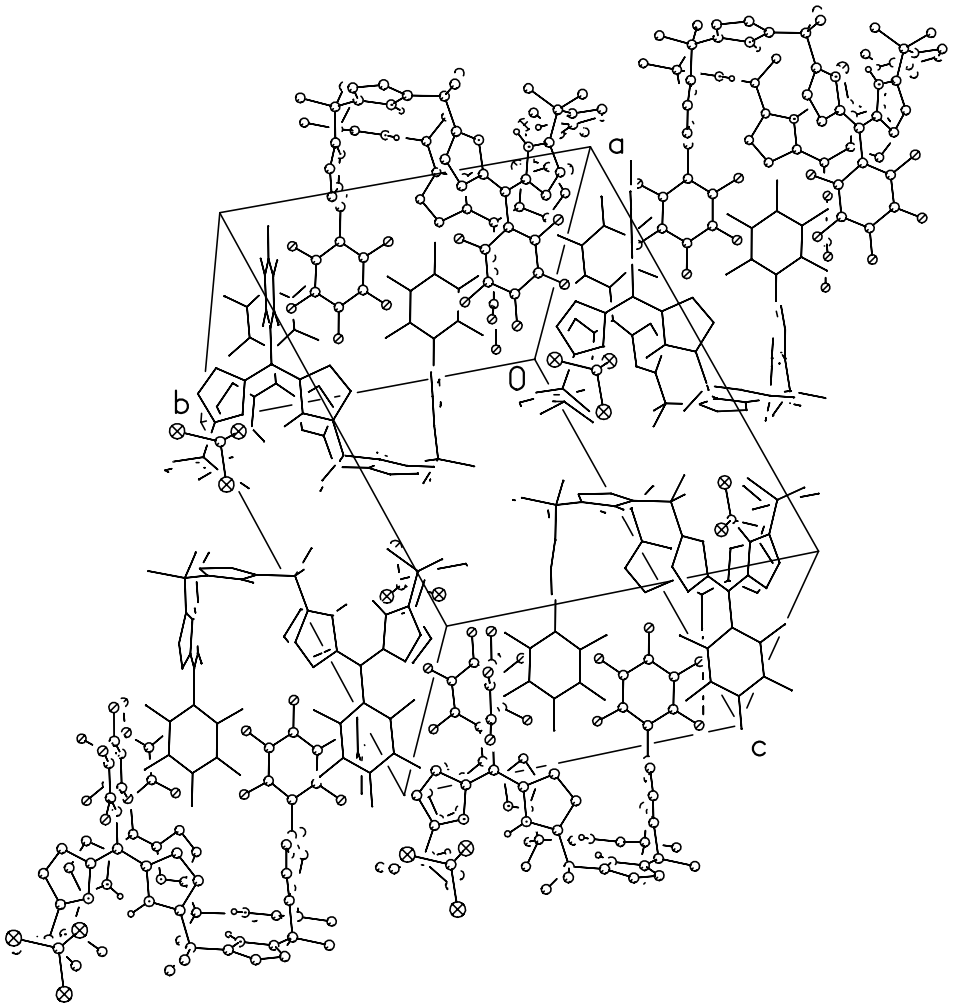


Figure 4. Unit cell packing diagram for **5**.



References

1) Connors K., Binding Constants: The Measurement of Molecular Complex Stability; Wiley: New-York, 1987.

2) 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) SIR92. (1993). A program for crystal structure solution. Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. J. Appl. Cryst. 26, 343-350.

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.

7) Spek, A. L. (1998). PLATON, A Multipurpose Crystallographic Tool. Utrecht University, The Netherlands.

8) Introduction to Organic Chemistry (1985). Appendix IV, A. Streitwieser, Jr. and C. H. Heathcock, New York: Macmillan Publishing Company.