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Appendix I

Supplementary Material for **3**·$2CH_3OH \cdot CH_2Cl_2$ ($C_{60}H_{70}N_{10}O_4 \cdot 2CH_3OH \cdot CH_2Cl_2$).

X-ray Experimental for $C_{60}H_{70}N_{10}O_4 \cdot 2CH_3OH \cdot CH_2Cl_2$.

Supplementary Table I-1. Crystallographic Data for $C_{60}H_{70}N_{10}O_4 \cdot 2CH_3OH \cdot CH_2Cl_2$.

Supplementary Table I-2. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of $(C_{60}H_{70}N_{10}O_4) \cdot 2CH_3OH \cdot CH_2Cl_2$.

Supplementary Table I-3. Anisotropic thermal parameters for the non-hydrogen atoms of $(C_{60}H_{70}N_{10}O_4) \cdot 2CH_3OH \cdot CH_2Cl_2$.

Supplementary Table I-4. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of $(C_{60}H_{70}N_{10}O_4) \cdot 2CH_3OH \cdot CH_2Cl_2$.

Supplementary Table I-5. Bond Lengths (Å) and Angles (^{o}) for the non-hydrogen atoms of $(C_{60}H_{70}N_{10}O_4) \cdot 2CH_3OH \cdot CH_2Cl_2$.

Supplementary Table I-6. Bond Lengths (Å) and Angles (^{o}) for the hydrogen atoms of $(C_{60}H_{70}N_{10}O_4) \cdot 2CH_3OH \cdot CH_2Cl_2$.

Supplementary Table I-7. Torsion angles (^{o}) for the non-hydrogen atoms of $(C_{60}H_{70}N_{10}O_4) \cdot 2CH_3OH \cdot CH_2Cl_2$.

Supplementary Table I-8. Observed and calculated structure factor amplitudes for $(C_{60}H_{70}N_{10}O_4) \cdot 2CH_3OH \cdot CH_2Cl_2$. Values for Fo, Fc and σ(Fo) have been multiplied by 10. Note: This table is available only in electronic form; see any current masthead page for internet access instructions.

Supplementary Figure I-1. View of $(C_{60}H_{70}N_{10}O_4) \cdot 2CH_3OH$ showing the atom labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. Hydrogen atoms shown are drawn to an arbitrary scale. The molecule lies around a crystallographic two-fold rotation axis passing through 0,y, 1/4. Atoms labeled by ' are related by -x, y, 1/2-z. Two symmetry related molecules of methanol are within H-bonding distance to the macrocycle. The relevant geometry of this interaction is: N2-H2N···O1a, N···O 3.006(12)Å, H···O 2.131(12)Å, N-H···O 163.8(3)°; N5-H5N···O1a, N···O 2.949(12)Å, H···O 2.117(12)Å, N-H···O 153.3(3)°; O1A···N3 2.967(13)Å, O1A···N4 2.870(12)Å. The position of the hydrogen atom on the methanol oxygen atom could not be located in a ΔF map and was not calculated in the refinement model.

Supplementary Figure I-2. Unit cell packing diagram for $(C_{60}H_{70}N_{10}O_4) \cdot 2CH_3OH \cdot CH_2Cl_2$. The view is approximately down the **b** axis. The methylene chloride solvate molecules reside in cavities of the macrocycles and are represented in wireframe fashion.

Supplementary Material for $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$ (continued).

Supplementary Figure I-3.
Top: View down the crystallographic two-fold rotation axis. Thermal ellipsoids are scaled to the 30% probability level. The molecule lies around a crystallographic two-fold rotation axis passing through 0,y, 1/4. Atoms labeled by ' are related by -x, y, 1/2-z. Two symmetry related molecules of methanol are within H-bonding distance to the macrocycle. The relevant geometry of this interaction is: N2-H2N···O1a, N···O 3.006(12)Å, H···O 2.131(12)Å, N-H···O 163.8(3)°; N5-H5N···O1a, N···O 2.949(12)Å, H···O 2.117(12)Å, N-H···O 153.3(3)°; O1A···N3 2.967(13)Å, O1A···N4 2.870(12)Å.
Bottom: Side view of the macrocycle showing the dome shape of the macrocycle. The disordered solvate molecule resides on the opposite side from the H-bound methanol molecules.

Supplementary Figure I-4. Ball and stick view of $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$ down the crystallographic two-fold rotation axis. Hydrogen atoms are not shown. The molecule lies around a crystallographic two-fold rotation axis passing through 0,y, 1/4. Atoms labeled by ' are related by -x, y, 1/2-z. Two symmetry related molecules of methanol are within H-bonding distance to the macrocycle. The H-bonding interaction is illustrated by dashed lines. A poorly defined solvent molecule, modelled as a methylene chloride molecule, lies around the two-fold rotation axis in the depression at the center of the macrocycle.

Supplementary Figure I-5. Side view showing the dome shape of the macrocycle. The disordered solvate molecule resides on the opposite side from the H-bound methanol molecules.

X-ray Experimental for $(C_{60}H_{70}N_{10}O_6)\cdot 2CH_3OH\cdot CH_2Cl_2$: Crystals grew as orange prisms by slow evaporation from CH_3OH-CH_2Cl_2. The data crystal was a prism of approximate dimensions: 0.17 x 0.34 x 0.34 mm. The data were collected at -90 °C on a Siemens P4 diffractometer, equipped with a Nicolet LT-2 low-temperature device and using a graphite monochromator with MoKα radiation (λ = 0.71073 Å). Two reflections (0,2,3; 4,2,-1) were remeasured every 97 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these check reflections was used to scale the data. The scaling factor ranged from 0.992 to 1.01. The data were corrected for Lp effects but not for absorption. Data reduction, decay correction, structure solution and refinement were performed using the SHELXTL/PC software package.[1] The structure was solved by direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms. The anisotropic displacement parameters were restrained to be approximately isotropic throughout the remainder of the structure refinement. One ethyl group was found to be disordered about two orientations of nearly equal occupancy. The site occupancy factors (SOFs) for the two atoms, C31a and C31b, were refined while using a common isotropic displacement parameter. The SOF refined to a value not significantly different to 0.5 for each atom and was fixed at this value for subsequent refinements. The hydrogen atoms were calculated in idealized positions (C-H 0.96Å, N-H 0.90Å) with isotropic temperature factors set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms). The hydrogen atom on the methanolic oxygen atom could not be located in a ΔF map and was not included in the final refinement model. The macrocycle lies around a crystallographic two-fold rotation axis passing through 0,y,1/4. The macrocycle is slightly concave and an indeterminate solvent molecule resides in this depression. The best fit to the data was found using two half-weight chlorine atoms as if one molecule of methylene chloride resided in the concave region. This area of electron density was not well defined and was complicated by its close proximity to the crystallographic two-fold rotation axis. No carbon atom position was included in the model for the methylene chloride molecule. 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 $P = (|F_o|^2 + 2|F_c|^2)/3$. The data were checked for secondary extinction errors but no correction to the data 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).[2] Other computer programs used in this work are listed elsewhere.[3] All figures were generated using SHELXTL/PC.[1] Tables of positional and thermal parameters, bond lengths, angles and torsion angles, and supplementary figures now follow.

References

1) Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

2) 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.

3) Gadol, S. M. and Davis, R. E. (1982). Organometallics 1, 1607-1613.

Supplementary Table I-1. Crystallographic Data[a] for $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$.

Formula	$C_{63}H_{80}N_{10}O_6Cl_2$
fw	1144.27
a, Å	28.375(13)
b, Å	10.231(2)
c, Å	21.248(8)
β, °	102.85(3)
V, Å3	6014(5)
Z	4
F(000)	2440
Crystal System	Monoclinic
Space Group	C2/c
T, °C	-90
2θ range (°)	4 - 50
Scan speed (°/min) (1.5° ω scan)	5 - 10
ρ_{calc}, g/cc	1.26
Reflections measured	5181
Unique reflections	4258
Decay Correction	0.992 - 1.01
R_{int} (F^2)	0.040

Supplementary Table I-1. Crystallographic Data (continued).

μ, cm-1	1.68
Crystal size, mm	0.17 x 0.34 x 0.34
Transmission factor range	n/a
$R_W(F^2)$[b]	0.232
R(F)[c]	0.132
Goodness of fit, S[d]	1.26
Parameters	379
Max $\lvert\Delta/\sigma\rvert$	<0.1
Min, max peaks ($e^-/Å^3$)	-0.40, 0.48

[a] Data were collected on a Siemens P4 diffractometer, equipped with a Nicolet LT-2 low-temperature device and using graphite monochromatized Mo Kα radiation (λ = 0.71073 Å). Data were collected using ω scans with a scan range of 1.5° in ω. Lattice parameters were obtained from the least-squares refinement of 24 reflections with $4.8 < 2\theta < 14.2°$.

[b] $R_w = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ and where the weight, w, is defined as follows: $w = 1/\{\sigma^2(|F_o|^2) + (0.02*P)^2\}$; $P = [1/3*(\text{Maximum of } (0 \text{ or } |F_o|^2) + 2/3*|F_c|^2]$.

[c] The conventional R index based on F where the 1132 observed reflections have $F_o > 4(\sigma(F_o))$.

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

Supplementary Table I-2. Fractional coordinates and equivalent isotropic thermal parameters (Å^2) for the non-hydrogen atoms of $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$.

Atom	x	y	z	U
N1	0.1144(3)	0.2277(10)	0.3942(4)	0.030(4)
N2	0.0101(3)	0.2497(10)	0.4635(4)	0.028(4)
N3	-0.0833(3)	0.3145(11)	0.4880(5)	0.036(4)
N4	-0.1614(3)	0.3055(10)	0.3853(5)	0.034(4)
N5	-0.1505(3)	0.2275(10)	0.2578(5)	0.034(4)
C1	0.1237(4)	0.1631(14)	0.3419(6)	0.040(4)
C2	0.0931(4)	0.0580(14)	0.3323(6)	0.031(4)
C3	0.0639(4)	0.0624(14)	0.3793(6)	0.031(4)
C4	0.0765(4)	0.1738(14)	0.4151(6)	0.034(4)
C5	0.0597(4)	0.2324(12)	0.4680(5)	0.021(4)
C6	0.0831(4)	0.3008(13)	0.5199(5)	0.028(4)
C7	0.0490(4)	0.3562(12)	0.5507(5)	0.026(4)
C8	0.0042(4)	0.3207(13)	0.5171(6)	0.027(4)
C9	-0.0445(4)	0.3571(12)	0.5207(5)	0.029(4)
C10	-0.1297(4)	0.3499(13)	0.4969(5)	0.027(4)
C11	-0.1339(4)	0.3852(13)	0.5611(5)	0.031(4)
C12	-0.1801(5)	0.4203(15)	0.5684(6)	0.045(5)
C13	-0.2187(5)	0.4141(14)	0.5182(6)	0.038(4)
C14	-0.2144(4)	0.3805(12)	0.4574(5)	0.027(4)
C15	-0.1678(4)	0.3448(12)	0.4495(6)	0.024(4)
C16	-0.1938(4)	0.3142(14)	0.3378(6)	0.044(4)
C17	-0.1914(4)	0.2842(15)	0.2719(6)	0.045(5)
C18	-0.2254(4)	0.297(2)	0.2189(6)	0.044(5)
C19	-0.2083(4)	0.2521(14)	0.1637(6)	0.044(4)
C20	-0.1603(4)	0.2084(13)	0.1921(6)	0.033(4)
C21	0.0906(4)	-0.0499(13)	0.2823(5)	0.053(5)
C22	0.0266(4)	-0.0400(13)	0.3867(6)	0.048(5)
C23	0.1388(3)	0.3098(13)	0.5453(5)	0.039(4)
C24	0.0589(4)	0.4278(13)	0.6116(5)	0.042(4)
C25	0.0619(4)	0.3530(14)	0.6757(6)	0.069(5)
O26	-0.1910(4)	0.4388(12)	0.6284(5)	0.089(5)
C27	-0.1528(5)	0.442(2)	0.6830(7)	0.104(6)
O28	-0.2641(3)	0.4434(10)	0.5322(4)	0.058(4)
C29	-0.3070(4)	0.4431(14)	0.4813(6)	0.057(5)
C30	-0.2777(5)	0.356(2)	0.2095(8)	0.077(6)
C31A	-0.3064(8)	0.286(3)	0.2188(11)	0.050(7)
C31B	-0.2760(8)	0.480(3)	0.2022(13)	0.053(7)
C32	-0.2324(4)	0.2494(13)	0.0937(6)	0.067(5)
O1A	-0.0801(3)	0.1464(8)	0.3753(4)	0.039(3)
C2A	-0.0933(4)	0.0338(13)	0.4030(6)	0.047(5)
Cl1A	-0.0286(5)	0.407(2)	0.1966(7)	0.218(6)
Cl2A	0.0192(6)	0.305(2)	0.2152(8)	0.233(6)

For anisotropic atoms, the U value is U_{eq}, calculated as $U_{eq} = 1/3\ \Sigma_i\Sigma_j\ U_{ij}\ a_i^*\ a_j^*\ A_{ij}$ where A_{ij} is the dot product of the ith and j^{th} direct space unit cell vectors.

Supplementary Table I-3. Anisotropic thermal parameters for the non-hydrogen atoms of

$(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$.

Atom	U11	U22	U33	U12	U13	U23
N1	0.027(5)	0.015(7)	0.048(6)	-0.013(5)	0.008(5)	0.003(5)
N2	0.028(5)	0.019(6)	0.039(6)	-0.003(5)	0.009(4)	0.014(5)
N3	0.028(5)	0.031(7)	0.044(6)	-0.001(5)	-0.003(5)	0.021(6)
N4	0.039(5)	0.015(6)	0.044(6)	0.003(5)	0.004(5)	-0.002(5)
N5	0.030(5)	0.021(7)	0.050(6)	0.008(5)	0.008(5)	-0.005(5)
C1	0.038(7)	0.035(8)	0.049(7)	-0.005(6)	0.013(6)	0.007(7)
C2	0.044(7)	0.020(8)	0.031(6)	-0.013(6)	0.013(5)	0.002(6)
C3	0.025(6)	0.025(8)	0.047(7)	-0.002(6)	0.015(5)	-0.006(6)
C4	0.029(6)	0.030(8)	0.043(7)	-0.002(6)	0.010(5)	-0.001(6)
C5	0.021(6)	0.015(7)	0.027(6)	-0.008(5)	0.004(5)	0.013(6)
C6	0.026(6)	0.015(7)	0.036(6)	-0.004(5)	-0.007(5)	0.016(6)
C7	0.039(6)	0.018(7)	0.023(6)	0.004(6)	0.011(5)	0.016(6)
C8	0.031(6)	0.019(7)	0.031(6)	0.001(6)	0.003(5)	0.014(6)
C9	0.035(6)	0.017(7)	0.033(6)	0.002(6)	0.003(5)	0.000(6)
C10	0.032(6)	0.017(7)	0.033(6)	0.006(5)	0.009(5)	0.004(6)
C11	0.044(7)	0.017(7)	0.033(6)	0.016(6)	0.011(5)	0.000(6)
C12	0.058(7)	0.042(9)	0.035(7)	0.018(6)	0.012(6)	0.005(7)
C13	0.050(7)	0.019(8)	0.046(7)	0.010(6)	0.011(6)	0.009(6)
C14	0.033(6)	0.013(7)	0.034(6)	-0.001(5)	0.008(5)	0.014(6)
C15	0.022(6)	0.010(7)	0.040(6)	0.000(5)	0.009(5)	-0.001(6)
C16	0.029(6)	0.046(8)	0.051(7)	0.005(6)	-0.002(6)	-0.019(7)
C17	0.039(7)	0.042(8)	0.057(7)	0.005(6)	0.019(6)	-0.009(7)
C18	0.039(7)	0.048(9)	0.043(7)	0.015(6)	0.006(6)	-0.010(7)
C19	0.036(6)	0.042(9)	0.049(7)	-0.000(6)	-0.002(6)	-0.013(7)
C20	0.032(6)	0.018(7)	0.047(7)	0.007(6)	0.006(5)	-0.001(6)
C21	0.055(7)	0.042(9)	0.066(8)	-0.005(6)	0.022(6)	-0.006(7)
C22	0.047(7)	0.029(8)	0.070(8)	-0.005(6)	0.015(6)	0.002(7)
C23	0.035(6)	0.029(8)	0.049(7)	-0.009(6)	0.001(5)	0.001(6)
C24	0.047(7)	0.029(8)	0.043(7)	-0.002(6)	-0.005(6)	0.015(7)
C25	0.080(8)	0.061(9)	0.065(8)	-0.014(7)	0.011(7)	-0.002(8)
O26	0.092(7)	0.096(9)	0.082(7)	0.057(6)	0.025(6)	-0.013(6)
C27	0.117(10)	0.100(11)	0.092(10)	0.045(8)	0.022(8)	-0.013(8)
O28	0.057(5)	0.052(7)	0.071(6)	0.032(5)	0.026(5)	0.008(5)
C29	0.052(7)	0.048(9)	0.076(8)	0.012(7)	0.022(6)	-0.004(7)
C30	0.050(8)	0.097(11)	0.093(9)	0.010(8)	0.035(7)	0.004(8)
C31A	0.048(11)	0.054(13)	0.053(11)	-0.008(9)	0.019(9)	-0.008(9)
C31B	0.047(11)	0.045(13)	0.068(12)	0.006(9)	0.013(9)	0.002(9)
C32	0.070(8)	0.068(10)	0.066(8)	0.012(7)	0.019(7)	-0.001(7)

Supplementary Table I-3. (continued).

Atom	U11	U22	U33	U12	U13	U23
O1A	0.041(5)	0.016(6)	0.058(5)	-0.002(4)	0.009(4)	0.011(5)
C2A	0.045(7)	0.041(9)	0.060(8)	0.004(6)	0.025(6)	-0.005(7)
Cl1A	0.214(10)	0.249(12)	0.197(9)	-0.039(8)	0.062(8)	-0.027(9)
Cl2A	0.236(10)	0.229(11)	0.223(11)	0.021(9)	0.023(8)	-0.016(9)

The Uij are the mean-square amplitudes of vibration in $Å^2$ from the general temperature factor expression

$$\exp[-2\pi^2(h^2a^{*2}U11 + k^2b^{*2}U22 + l^2c^{*2}U33 + 2hka^*b^*U12 + 2hla^*c^*U13 + 2klb^*c^*U23)]$$

Supplementary Table I-4. Fractional coordinates and isotropic thermal parameters (Å^2) for the hydrogen atoms of $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$.

Atom	x	y	z	U
H1N	0.1322	0.2946	0.4143	0.036
H2N	-0.0136	0.2205	0.4309	0.034
H5N	-0.1223	0.2103	0.2855	0.040
H9	-0.0468	0.4148	0.5555	0.035
H11	-0.1065	0.3884	0.5969	0.037
H14	-0.2416	0.3837	0.4214	0.032
H16	-0.2244	0.3461	0.3436	0.052
H21A	0.1133	-0.0348	0.2557	0.079
H21B	0.0981	-0.1311	0.3051	0.079
H21C	0.0585	-0.0540	0.2558	0.079
H22A	0.0108	-0.0146	0.4203	0.072
H22B	0.0027	-0.0541	0.3474	0.072
H22C	0.0443	-0.1193	0.3988	0.072
H23A	0.1537	0.2641	0.5154	0.058
H23B	0.1490	0.3995	0.5480	0.058
H23C	0.1481	0.2700	0.5871	0.058
H24A	0.0329	0.4893	0.6089	0.051
H24B	0.0880	0.4776	0.6150	0.051
H25A	0.0674	0.4126	0.7115	0.104
H25B	0.0326	0.3051	0.6745	0.104
H25C	0.0885	0.2931	0.6807	0.104
H27A	-0.1644	0.455	0.7217	0.155
H27B	-0.1330	0.365	0.6873	0.155
H27C	-0.1340	0.517	0.6761	0.155
H29A	-0.3349	0.465	0.4979	0.086
H29B	-0.3033	0.506	0.4493	0.086
H29C	-0.3113	0.358	0.4620	0.086
H31A	-0.3376	0.327	0.2108	0.075
H31B	-0.3072	0.214	0.1901	0.075
H31C	-0.2980	0.256	0.2627	0.075
H31D	-0.3075	0.519	0.1946	0.080
H31E	-0.2561	0.515	0.2409	0.080
H31F	-0.2615	0.498	0.1664	0.080
H32A	-0.2108	0.2156	0.0686	0.101
H32B	-0.2603	0.1942	0.0883	0.101
H32C	-0.2422	0.3363	0.0794	0.101
H2AA	-0.0825	-0.0405	0.3826	0.070
H2AB	-0.1278	0.0302	0.3975	0.070
H2AC	-0.0791	0.0322	0.4485	0.070

Supplementary Table I-5. Bond Lengths (Å) and Angles (°) for the non-hydrogen atoms of $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$.

1	2	3	1-2	1-2-3
C1	N1	C4	1.37(2)	112.1(10)
C4	N1		1.37(2)	
C5	N2	C8	1.400(13)	108.1(8)
C8	N2		1.39(2)	
C9	N3	C10	1.243(13)	124.7(11)
C10	N3		1.42(2)	
C15	N4	C16	1.47(2)	121.8(11)
C16	N4		1.208(14)	
C17	N5	C20	1.39(2)	106.8(9)
C20	N5		1.37(2)	
C2	C1	N1	1.37(2)	105.7(11)
C20'	C1	N1	1.466(14)	122.2(12)
C20'	C1	C2		132.0(13)
C3	C2	C21	1.43(2)	124.3(11)
C3	C2	C1		108.5(12)
C21	C2	C1	1.52(2)	127.2(12)
C4	C3	C22	1.37(2)	128.0(12)
C4	C3	C2		107.0(11)
C22	C3	C2	1.52(2)	125.0(11)
C5	C4	N1	1.45(2)	120.4(11)
C5	C4	C3		133.2(12)
N1	C4	C3		106.3(11)
C6	C5	N2	1.35(2)	107.2(10)
C6	C5	C4		131.5(10)
N2	C5	C4		119.9(9)
C7	C6	C23	1.40(2)	124.5(10)
C7	C6	C5		109.0(10)
C23	C6	C5	1.556(14)	126.3(11)
C8	C7	C24	1.36(2)	124.8(12)
C8	C7	C6		107.9(10)
C24	C7	C6	1.46(2)	127.0(10)
C9	C8	N2	1.45(2)	117.8(9)
C9	C8	C7		133.9(12)
N2	C8	C7		107.5(10)
N3	C9	C8		128.1(11)
C11	C10	C15	1.44(2)	120.8(11)
C11	C10	N3		117.7(9)
C15	C10	N3	1.304(14)	121.4(11)
C12	C11	C10	1.40(2)	116.5(10)
C13	C12	O26	1.35(2)	115.0(12)
C13	C12	C11		121.0(13)
O26	C12	C11	1.39(2)	122.9(10)
C14	C13	O28	1.37(2)	121.8(10)
C14	C13	C12		122.0(13)
O28	C13	C12	1.41(2)	116.2(12)

Supplementary Table I-5. (continued).

1	2	3	1-2	1-2-3
C15	C14	C13	1.42(2)	117.3(10)
N4	C15	C10		118.4(10)
N4	C15	C14		119.4(9)
C10	C15	C14		122.1(11)
C17	C16	N4	1.45(2)	127.0(12)
C18	C17	N5	1.32(2)	110.1(12)
C18	C17	C16		128.6(12)
N5	C17	C16		121.2(10)
C19	C18	C30	1.44(2)	119.9(11)
C19	C18	C17		110.6(12)
C30	C18	C17	1.57(2)	129.5(13)
C20	C19	C32	1.43(2)	126.5(12)
C20	C19	C18		102.5(10)
C32	C19	C18	1.49(2)	131.0(11)
N5	C20	C19		110.0(11)
N5	C20	C1'		122.7(10)
C19	C20	C1'		127.0(12)
C25	C24	C7	1.55(2)	119.6(11)
C27	O26	C12	1.40(2)	118.2(11)
C29	O28	C13	1.438(13)	120.0(10)
C31A	C30	C31B	1.14(3)	134.(2)
C31A	C30	C18		115.(2)
C31B	C30	C18	1.27(4)	110.(2)
C2A	O1A		1.38(2)	

Atoms marked by ' are related by -x, y, 1/2-z.

Supplementary Table I-6. Bond Lengths (Å) and Angles (°) for the hydrogen atoms of $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$.

1	2	3	1-2	1-2-3
H1N	N1	C1	0.90	124.3(12)
H1N	N1	C4		123.5(12)
H2N	N2	C5	0.90	125.2(12)
H2N	N2	C8		126.7(11)
H5N	N5	C17	0.90	127.2(13)
H5N	N5	C20		125.9(13)
H9	C9	N3	0.96	116.2(13)
H9	C9	C8		115.4(11)
H11	C11	C12	0.96	121.0(14)
H11	C11	C10		122.4(14)
H14	C14	C15	0.96	121.3(13)
H14	C14	C13		121.4(14)
H16	C16	C17	0.96	115.6(12)
H16	C16	N4		117.4(15)
H21A	C21	H21B	0.96	109.(2)
H21A	C21	H21C		109.5(14)
H21A	C21	C2		111.2(13)
H21B	C21	H21C	0.96	109.(2)
H21B	C21	C2		107.8(12)
H21C	C21	C2	0.96	109.4(13)
H22A	C22	H22B	0.96	109.5(14)
H22A	C22	H22C		109.(2)
H22A	C22	C3		109.9(14)
H22B	C22	H22C	0.96	109.(2)
H22B	C22	C3		112.7(14)
H22C	C22	C3	0.96	105.8(12)
H23A	C23	H23B	0.96	109.(2)
H23A	C23	H23C		109.5(15)
H23A	C23	C6		107.8(11)
H23B	C23	H23C	0.96	109.5(14)
H23B	C23	C6		110.3(11)
H23C	C23	C6	0.96	110.3(12)
H24A	C24	H24B	0.96	107.(2)
H24A	C24	C25		105.6(13)
H24A	C24	C7		106.4(11)
H24B	C24	C25	0.96	108.6(11)
H24B	C24	C7		108.9(14)
H25A	C25	H25B	0.96	109.(2)
H25A	C25	H25C		109.5(15)
H25A	C25	C24		110.4(15)
H25B	C25	H25C	0.96	109.(2)
H25B	C25	C24		110.5(12)
H25C	C25	C24	0.96	107.5(14)
H27A	C27	H27B	0.96	109.(2)
H27A	C27	H27C		109.(2)
H27A	C27	O26		111.(2)

Supplementary Table I-6. (continued).

1	2	3	1-2	1-2-3
H27B	C27	H27C	0.96	109.(2)
H27B	C27	O26		113.(2)
H27C	C27	O26	0.96	104.(2)
H29A	C29	H29B	0.96	109.(2)
H29A	C29	H29C		109.(2)
H29A	C29	O28		110.6(12)
H29B	C29	H29C	0.96	109.(2)
H29B	C29	O28		108.7(12)
H29C	C29	O28	0.96	109.1(13)
H31A	C31A	H31B	0.96	109.(3)
H31A	C31A	H31C		109.(3)
H31A	C31A	C30		111.(3)
H31B	C31A	H31C	0.96	109.(3)
H31B	C31A	C30		107.(3)
H31C	C31A	C30	0.96	110.(3)
H31D	C31B	H31E	0.96	109.(4)
H31D	C31B	H31F		109.(3)
H31D	C31B	C30		112.(3)
H31E	C31B	H31F	0.96	109.(3)
H31E	C31B	C30		108.(3)
H31F	C31B	C30	0.96	109.(3)
H32A	C32	H32B	0.96	109.(2)
H32A	C32	H32C		109.(2)
H32A	C32	C19		110.7(12)
H32B	C32	H32C	0.96	109.(2)
H32B	C32	C19		108.1(14)
H32C	C32	C19	0.96	109.5(14)
H2AA	C2A	H2AB	0.96	110.(2)
H2AA	C2A	H2AC		109.6(15)
H2AA	C2A	O1A		108.7(13)
H2AB	C2A	H2AC	0.96	108.(2)
H2AB	C2A	O1A		110.2(13)
H2AC	C2A	O1A	0.96	110.4(13)

Supplementary Table I-7. Torsion Angles (°) for the non-hydrogen atoms of $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$.

1	2	3	4	1-2-3-4
C1	N1	C4	C3	6.3(13)
C1	N1	C4	C5	-177.0(10)
C4	N1	C1	C2	-4.9(13)
C4	N1	C1	C20'	175.3(11)
C5	N2	C8	C7	5.3(13)
C5	N2	C8	C9	176.5(11)
C8	N2	C5	C4	-173.3(11)
C8	N2	C5	C6	-5.2(13)
C9	N3	C10	C11	-30.(2)
C9	N3	C10	C15	153.5(13)
C10	N3	C9	C8	176.3(12)
C15	N4	C16	C17	177.1(13)
C16	N4	C15	C10	-170.7(13)
C16	N4	C15	C14	7.(2)
C17	N5	C20	C19	-0.7(14)
C17	N5	C20	C1'	173.3(13)
C20	N5	C17	C16	178.3(13)
C20	N5	C17	C18	2.(2)
N1	C1	C2	C3	1.6(13)
N1	C1	C2	C21	-176.4(10)
C20'	C1	C2	C3	-178.6(13)
C20'	C1	C2	C3	3(3)
C21	C2	C3	C4	-178.8(12)
C21	C2	C3	C22	1.(2)
C1	C2	C3	C4	2.1(13)
C1	C2	C3	C22	-177.5(11)
C22	C3	C4	N1	174.7(10)
C22	C3	C4	C5	-1.(2)
C2	C3	C4	N1	-4.9(12)
C2	C3	C4	C5	179.0(12)
N1	C4	C5	N2	134.7(11)
N1	C4	C5	C6	-30.(2)
C3	C4	C5	N2	-50.(2)
C3	C4	C5	C6	145.5(14)
N2	C5	C6	C7	3.1(14)
N2	C5	C6	C23	178.9(11)
C4	C5	C6	C7	169.4(13)
C4	C5	C6	C23	-15.(2)
C23	C6	C7	C8	-175.8(11)
C23	C6	C7	C24	-2.(2)
C5	C6	C7	C8	0.1(14)
C5	C6	C7	C24	174.1(12)
C8	C7	C24	C25	83.(2)
C24	C7	C8	N2	-177.5(11)
C24	C7	C8	C9	13.(2)
C6	C7	C8	N2	-3.3(14)

Supplementary Table I-7. (continued).

1	2	3	4	1-2-3-4
C6	C7	C8	C9	-172.5(13)
C6	C7	C24	C25	-90.(2)
N2	C8	C9	N3	14.(2)
C7	C8	C9	N3	-177.7(14)
C11	C10	C15	N4	-178.7(11)
C11	C10	C15	C14	4.(2)
C15	C10	C11	C12	-3.(2)
N3	C10	C11	C12	179.7(11)
N3	C10	C15	N4	-2.(2)
N3	C10	C15	C14	-179.7(12)
C10	C11	C12	C13	3.(2)
C10	C11	C12	O26	170.7(13)
C13	C12	O26	C27	178.2(14)
O26	C12	C13	C14	-171.9(13)
O26	C12	C13	O28	8.(2)
C11	C12	C13	C14	-4.(2)
C11	C12	C13	O28	175.9(12)
C11	C12	O26	C27	10.(2)
C14	C13	O28	C29	-2.(2)
O28	C13	C14	C15	-176.0(12)
C12	C13	C14	C15	3.(2)
C12	C13	O28	C29	178.1(12)
C13	C14	C15	N4	178.8(12)
C13	C14	C15	C10	-4.(2)
N4	C16	C17	N5	7.(2)
N4	C16	C17	C18	-177.(2)
N5	C17	C18	C19	-2.(2)
N5	C17	C18	C30	-179.3(15)
C16	C17	C18	C19	-178.3(14)
C16	C17	C18	C30	4.(3)
C19	C18	C30	C31A	98.(2)
C19	C18	C30	C31B	-91.(2)
C30	C18	C19	C20	179.1(14)
C30	C18	C19	C32	-1.(2)
C17	C18	C19	C20	1.(2)
C17	C18	C19	C32	-178.3(15)
C17	C18	C30	C31A	-85.(3)
C17	C18	C30	C31B	86.(2)
C32	C19	C20	N5	179.3(12)
C32	C19	C20	C1'	6(2)
C18	C19	C20	N5	-0.5(15)
N1	C1	C20'	N5'	-128(2)
N1	C1	C20'	C19'	45(2)
C2	C1	C20'	N5'	52(2)
C2	C1	C20'	C19'	135(2)

Atoms marked by ' are related by -x, y, 1/2-z.

Supplementary Figure I-1. View of $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH$ showing the atom labeling scheme. Thermal ellipsoids are scaled to the 30% probability level. Hydrogen atoms shown are drawn to an arbitrary scale. The molecule lies around a crystallographic two-fold rotation axis passing through 0,y, 1/4. Atoms labeled by ' are related by -x, y, 1/2-z. Two symmetry related molecules of methanol are within H-bonding distance to the macrocycle. The relevant geometry of this interaction is: N2-H2N···O1a, N···O 3.006(12)Å, H···O 2.131(12)Å, N-H···O 163.8(3)°; N5-H5N···O1a, N···O 2.949(12)Å, H···O 2.117(12)Å, N-H···O 153.3(3)°; O1A···N3 2.967(13)Å, O1A···N4 2.870(12)Å. The position of the hydrogen atom on the methanol oxygen atom could not be located in a ΔF map and was not calculated in the refinement model.

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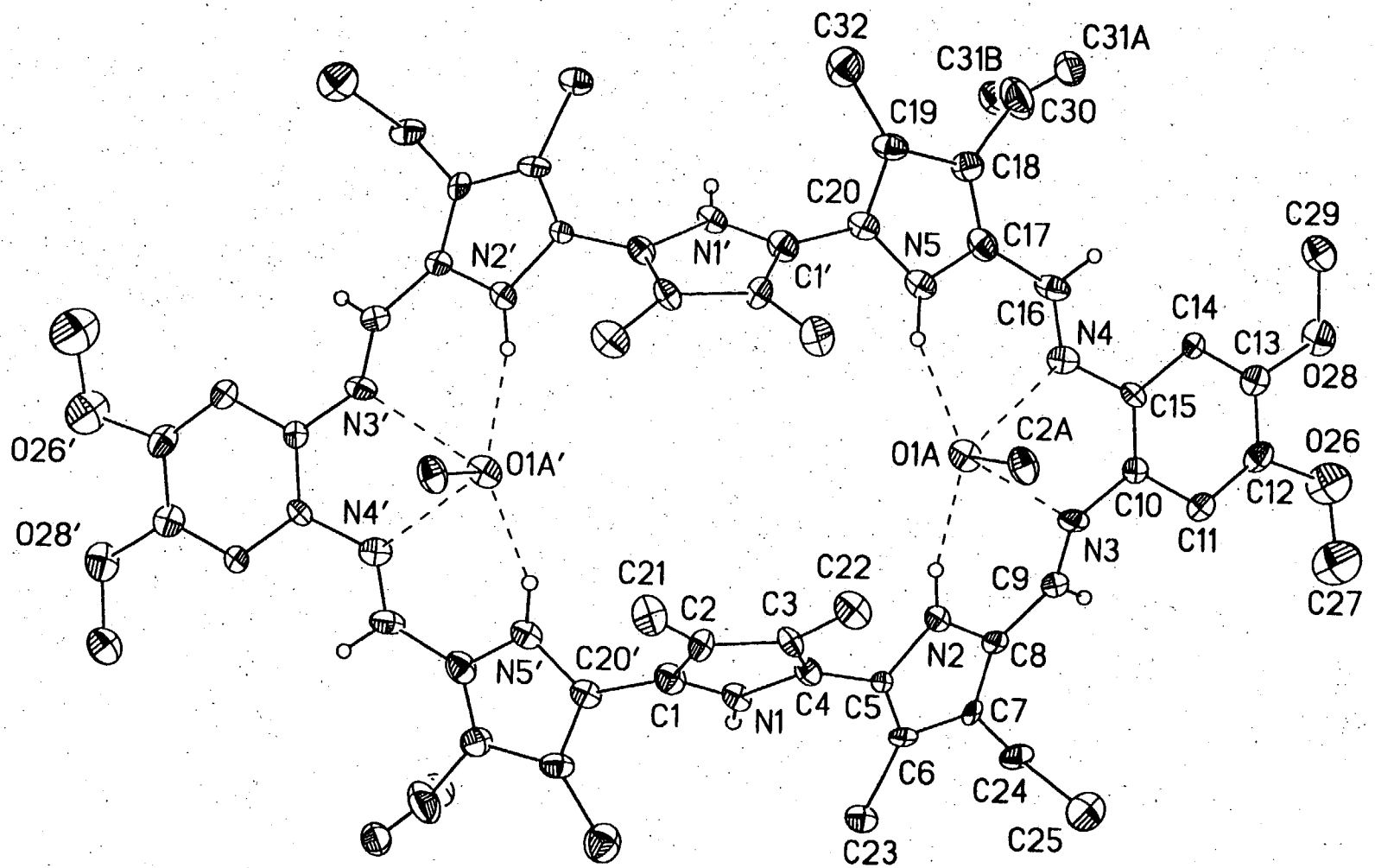
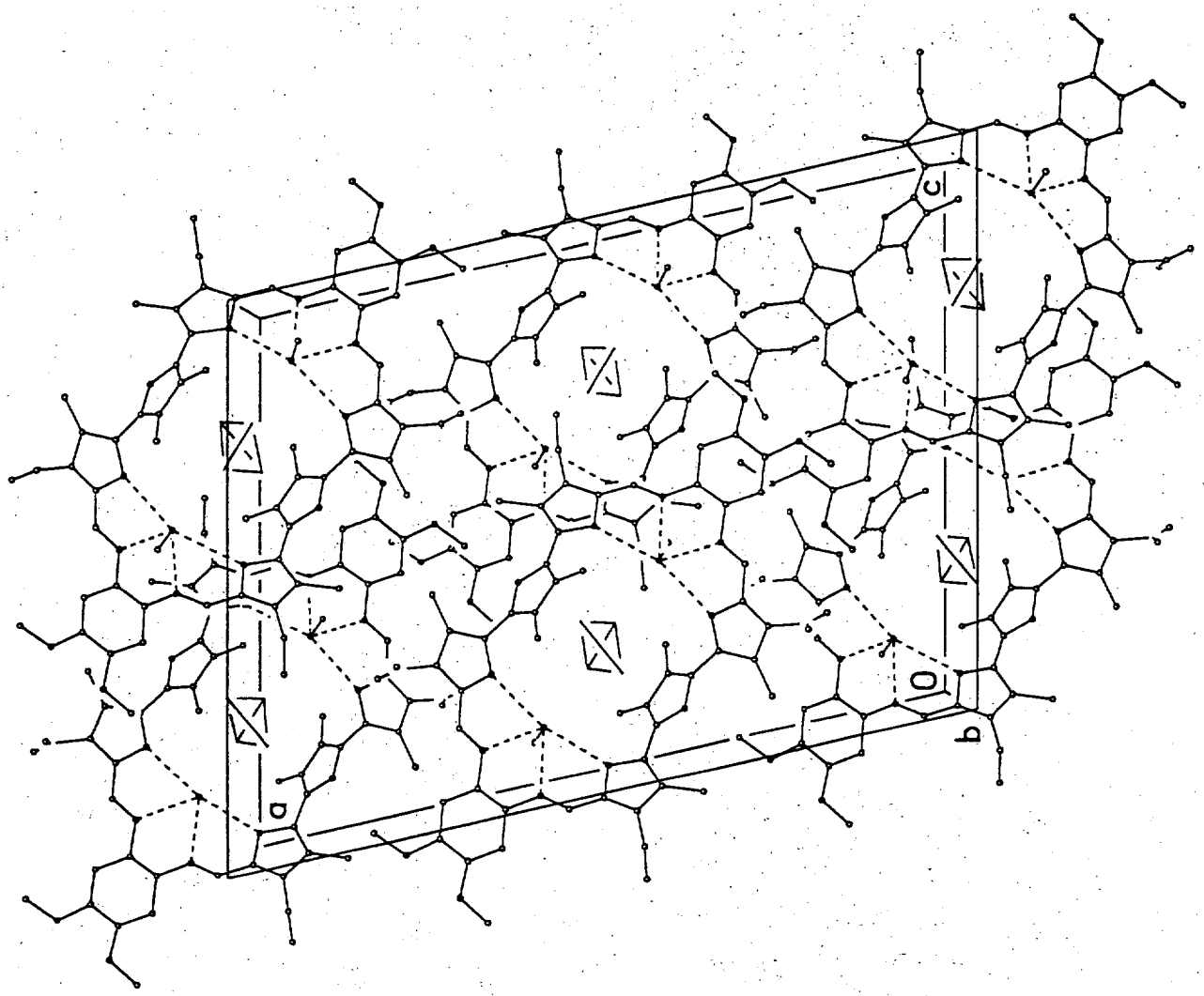
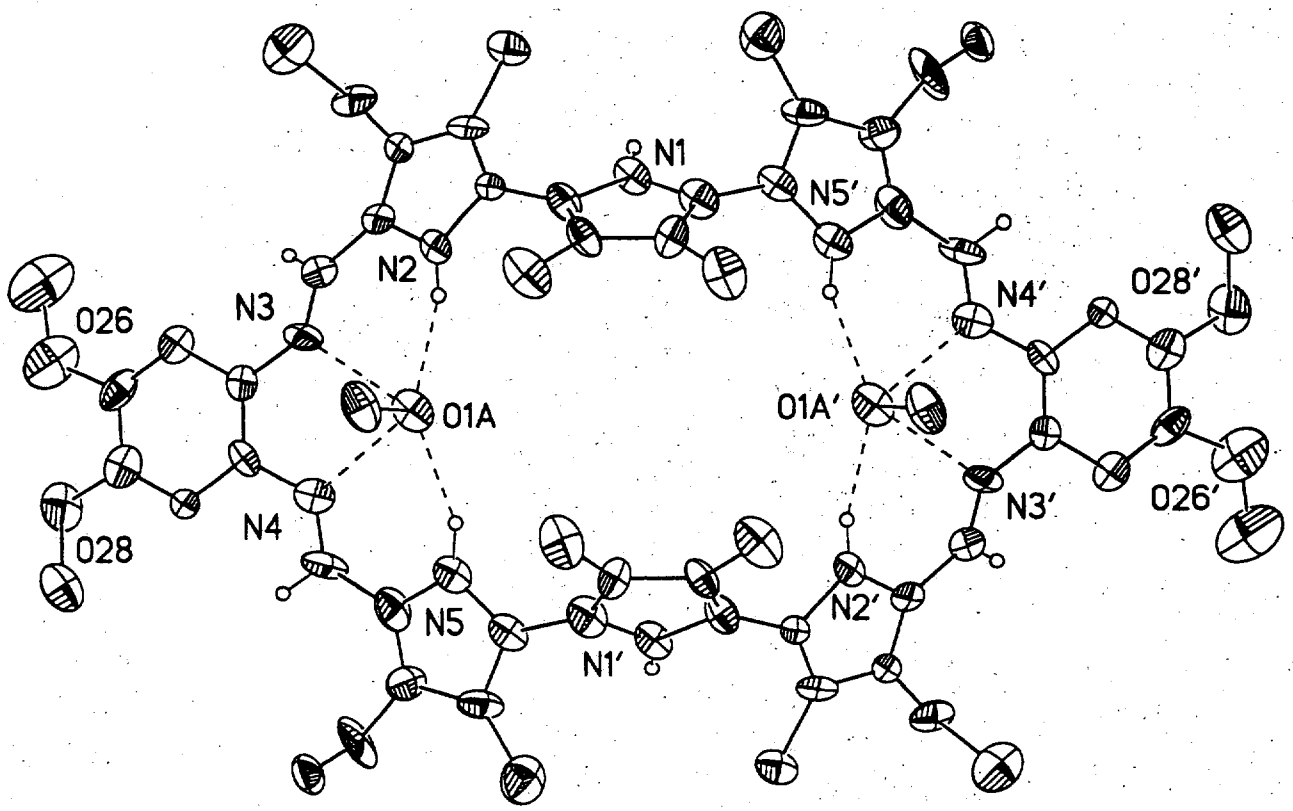
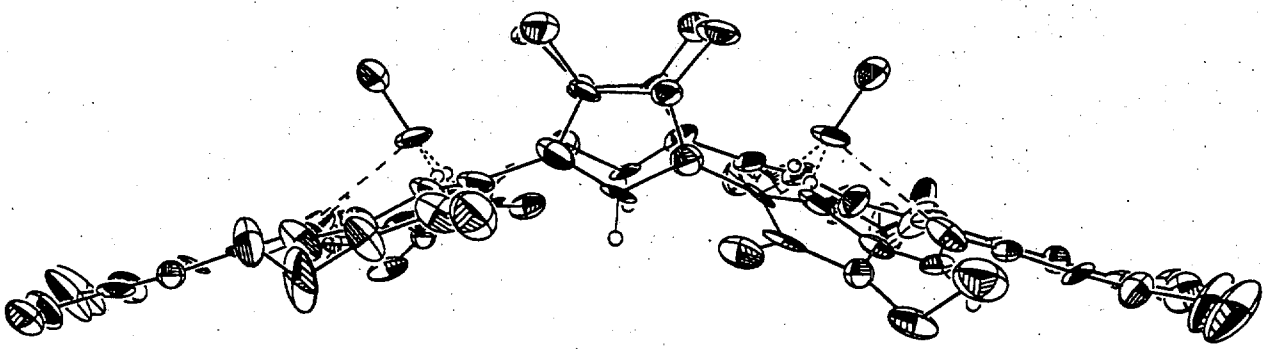


Supplementary Figure I-2. Unit cell packing diagram for $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$. The view is approximately down the **b** axis. The methylene chloride solvate molecules reside in cavities of the macrocycles and are represented in wireframe fashion.


a
c
b

Supplementary Figure I-3.
Top: View down the crystallographic two-fold rotation axis. Thermal ellipsoids are scaled to the 50% probability level. The molecule lies around a crystallographic two-fold rotation axis passing through 0,y, 1/4. Atoms labeled by ' are related by -x, y, 1/2-z. Two symmetry related molecules of methanol are within H-bonding distance to the macrocycle. The relevant geometry of this interaction is: N2-H2N···O1a, N···O 3.006(12)Å, H···O 2.131(12)Å, N-H···O 163.8(3)°; N5-H5N···O1a, N···O 2.949(12)Å, H···O 2.117(12)Å, N-H···O 153.3(3)°; O1A···N3 2.967(13)Å, O1A···N4 2.870(12)Å.
Bottom: Side view showing the dome shape of the macrocycle. The disordered solvate molecule resides on the opposite side from the H-bound methanol molecules.


N1
N5′
N2
N3
N4′
O28′
O26
O1A
O1A′
N3′
O26′
N4
O28
N5
N2′
N1′



Supplementary Figure I-4. Ball and stick view of $(C_{60}H_{70}N_{10}O_4)\cdot 2CH_3OH\cdot CH_2Cl_2$ down the crystallographic two-fold rotation axis. Hydrogen atoms are not shown. The molecule lies around a crystallographic two-fold rotation axis passing through 0,y, 1/4. Atoms labeled by ' are related by -x, y, 1/2-z. Two symmetry related molecules of methanol are within H-bonding distance to the macrocycle. The H-bonding interaction is illustrated by dashed lines. A poorly defined solvent molecule, modelled as a methylene chloride molecule, lies around the two-fold rotation axis in the depression at the center of the macrocycle.

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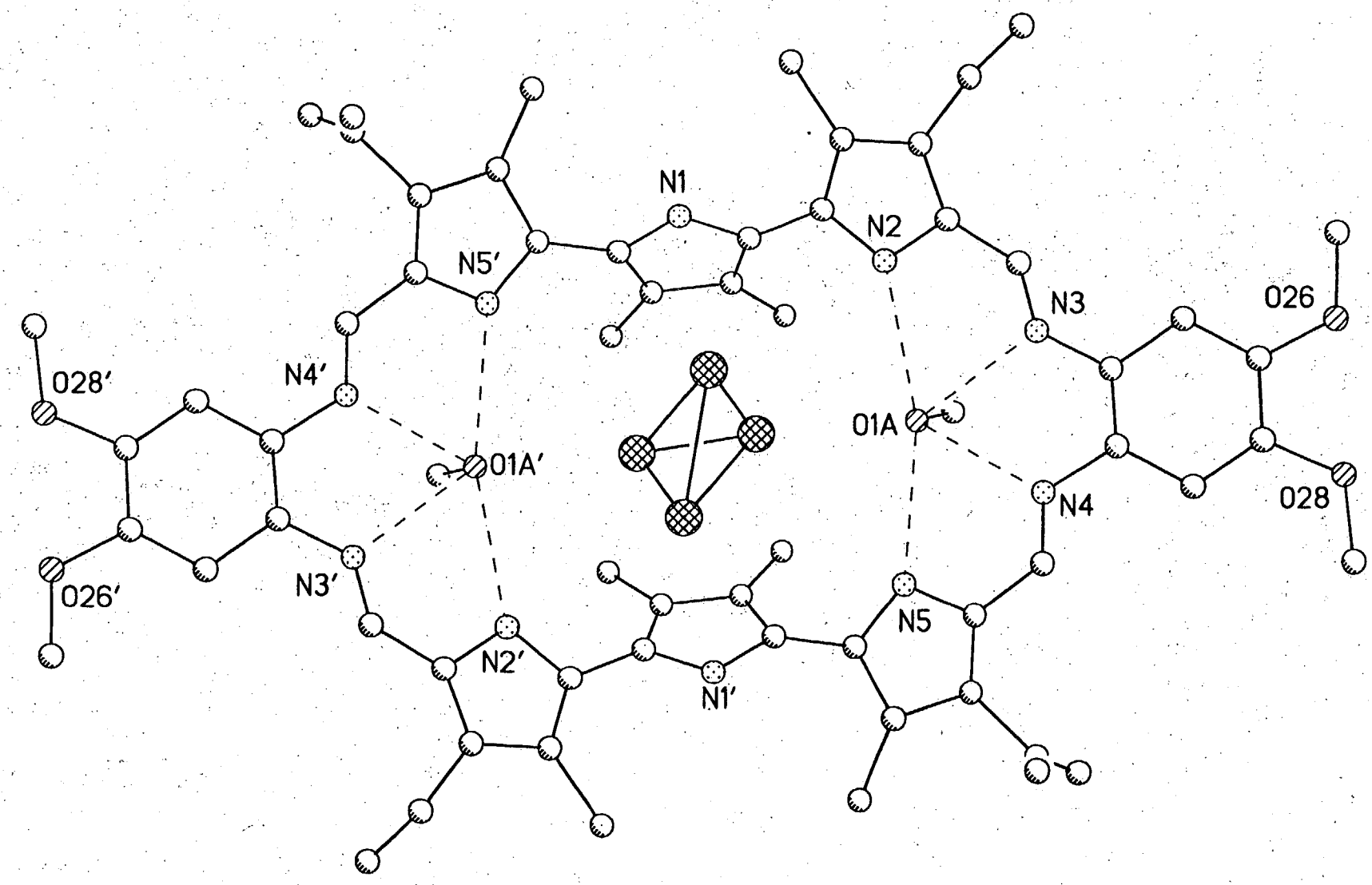
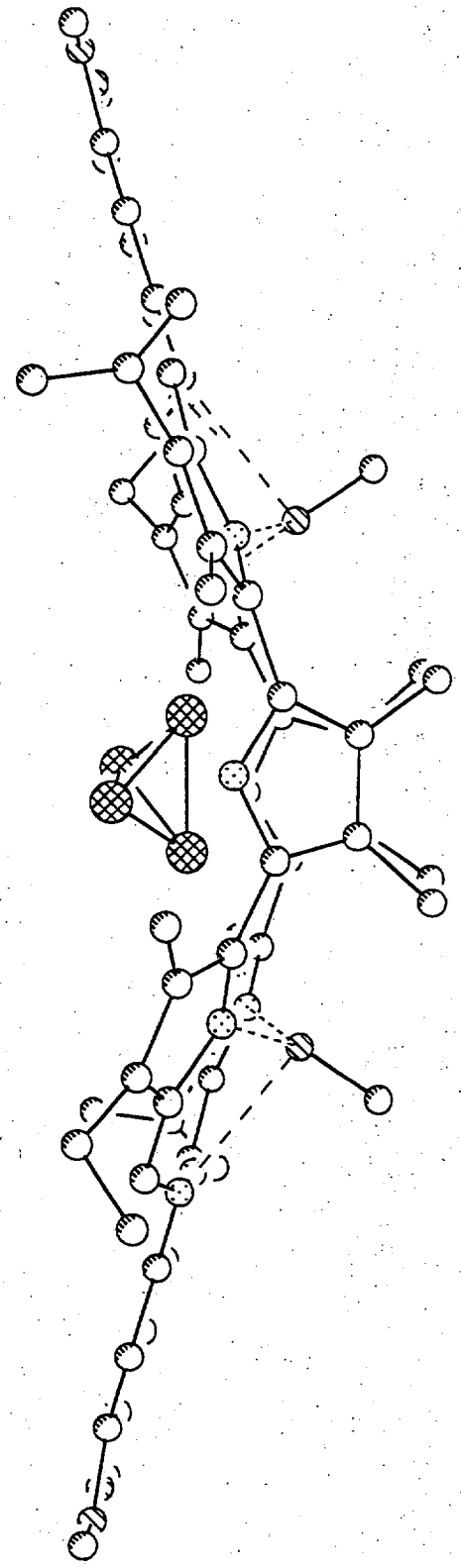


Supplementary Figure I-5. Side view showing the dome shape of the macrocycle. The disordered solvate molecule resides on the opposite side from the H-bound methanol molecules.



Appendix II

Supplementary Material for **5**·H_2O ($C_{22}H_{27}N_3O_2 \cdot H_2O$).

X-ray Experimental for $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Supplementary Table II-1. Crystallographic Data for $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Supplementary Table II-2. Fractional coordinates and equivalent isotropic thermal parameters ($Å^2$) for the non-hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Supplementary Table II-3. Anisotropic thermal parameters for the non-hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Supplementary Table II-4. Fractional coordinates and isotropic thermal parameters ($Å^2$) for the hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Supplementary Table II-5. Bond Lengths (Å) and Angles (^{o}) for the non-hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Supplementary Table II-6. Bond Lengths (Å) and Angles (^{o}) for the hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Supplementary Table II-7. Torsion angles (^{o}) for the non-hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Supplementary Table II-8. Bond Lengths (Å) and Angles (^{o}) for the hydrogen bonding interactions for $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Supplementary Table II-9. Observed and calculated structure factor amplitudes for $C_{22}H_{27}N_3O_2 \cdot H_2O$. Values for Fo, Fc and σ(Fo) have been multiplied by 10. Note: This table is available only in electronic form; see any current masthead page for internet access instructions.

Supplementary Figure II-1. View of molecule 1 of $C_{22}H_{27}N_3O_2$ showing the atom labelling scheme. Thermal ellipsoids are scaled to the 30% probability level. Hydrogen atoms shown are drawn to an arbitrary scale.

Supplementary Figure II-2. View of molecule 2 of $C_{22}H_{27}N_3O_2$ showing the atom labelling scheme. Thermal ellipsoids are scaled to the 30% probability level. Hydrogen atoms shown are drawn to an arbitrary scale.

Supplementary Figure II-3. Unit cell packing diagram for $C_{22}H_{27}N_3O_2 \cdot H_2O$. The view is approximately down the **a** axis. The molecules parallel to the **ab** plane. Molecules in each layer form a sandwich with the hydrogen bonding, hydrophilic portion of the complex forming the inner core while the hydrophobic portion forms the outer layer. Dashed lines are indicative of H-bonding interactions. The geometry of these interactions are listed in Supplementary Table II-8.

Supplementary Figure II-4. Superposition by least-square of the atoms of the terpyrrole unit of compound **5** (dashed lines) onto the equivalent atoms of compound **1** (solid lines). Only minor differences in the orientation of the ethyl groups are observed between the two molecules.

X-ray Experimental for $C_{22}H_{27}N_3O_2 \cdot H_2O$: Crystals grew pale green plates by slow evaporation from CH_2Cl_2-CH_3OH. The data crystal was a plate of approximate dimensions: 0.07 x 0.14 x 0.42 mm. The data were collected at -85 °C on a Siemens P4 diffractometer, equipped with a Nicolet LT-2 low-temperature device and using a graphite monochromator with MoKα radiation (λ = 0.71073 Å). Three reflections (0,4,5; 3,0,-5; 3,0,3) were remeasured every 97 reflections to monitor instrument and crystal stability. A smoothed curve of the intensities of these check reflections was used to scale the data. The scaling factor ranged from 0.9787 to 1.007. The data were corrected for Lp effects but not for absorption. Data reduction, decay correction, structure solution and refinement were performed using the SHELXTL/PC software package.[1] The structure was solved by direct methods and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms. Most hydrogen atoms were calculated in idealized positions (C-H 0.96Å, N-H 0.90Å) with isotropic temperature factors set to 1.2xUeq of the attached atom (1.5xUeq for methyl hydrogen atoms). The hydrogen atoms attached to the two water molecules were observed in a ΔF map. Attempts at refining these atoms independently failed as either the resulting geometries were unsatisfactory or the Uiso was unreasonable. Therefore, the O-H bond lengths for these atoms were adjusted to 0.80Å and fixed in subsequent refinements with the Uiso set to 1.2 x Ueq for the attached oxygen atom. The function, $\sum w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0369*P)^2 + (5.2432P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. The data were corrected for secondary extinction effects. The correction takes the form: $F_{corr} = kF_c/[1 + 1.4(2)x10^{-6}* F_c^2 \lambda^3/\sin 2\theta)]^{0.25}$ 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).[2] Other computer programs used in this work are listed elsewhere.[3] All figures were generated using SHELXTL/PC.[1] Tables of positional and thermal parameters, bond lengths, angles and torsion angles, and supplementary figures now follow.

References

1) Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

2) 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.

3) Gadol, S. M. and Davis, R. E. (1982). Organometallics 1, 1607-1613.

Supplementary Table II-1. Crystallographic Data[a] for $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Formula	$C_{22}H_{27}N_3O_2 \cdot H_2O$
fw	383.48
a, Å	13.314(3)
b, Å	16.401(4)
c, Å	19.956(5)
β, °	103.07(2)
V, Å3	4245(2)
Z	8
F(000)	1648
Crystal System	Monoclinic
Space Group	$P2_1/n$
T, °C	-85
2θ range (°)	4 - 50
Scan speed (°/min) (1.0° ω scan)	4 - 8
ρ_{calc}, g/cc	1.20
Reflections measured	7778
Unique reflections	7467
Decay Correction	0.9787 - 1.007
R_{int} (F^2)	0.126

Supplementary Table II-1. Crystallographic Data (continued).

μ, cm-1	0.81
Crystal size, mm	0.07x0.14x0.42
Transmission factor range	N/A
$R_W(F^2)$[b]	0.247
R(F)[c]	0.116
Goodness of fit, S[d]	1.074
Parameters	506
Max $\lvert\Delta/\sigma\rvert$	<0.1
Min, max peaks ($e^-/Å^3$)	-0.41, 0.37

[a] Data were collected on a Siemens P4 diffractometer, equipped with a Nicolet LT-2 low-temperature device and using graphite monochromatized Mo Kα radiation (λ = 0.71073 Å). Data were collected using ω scans with a scan range of 1° in ω. Lattice parameters were obtained from the least-squares refinement of 36 reflections with $10.8 < 2\theta < 16.5°$.

[b] $R_w = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ and where the weight, w, is defined as follows: $w = 1/\{\sigma^2(|F_o|^2) + (a*P)^2 + b*P\}$; $P = [1/3*(\text{Maximum of } (0 \text{ or } |F_o|^2) + 2/3*|F_c|^2]$. The parameters a and b were suggested during refinement and are 0.0369 and 5.2432, respectively.

[c] The conventional R index based on F where the 2180 observed reflections have $F_o>4(\sigma(F_o))$.

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

Supplementary Table II-2. Fractional coordinates and equivalent isotropic thermal parameters (Å^2) for the non-hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Atom	x	y	z	U
O1	0.4406(5)	0.3374(4)	0.2358(3)	0.026(3)
O2	0.4472(5)	1.0395(4)	0.2416(3)	0.029(3)
N1	0.3440(6)	0.4841(5)	0.1703(4)	0.022(3)
N2	0.2567(5)	0.6893(5)	0.1406(3)	0.023(3)
N3	0.3454(6)	0.8931(5)	0.1753(4)	0.024(3)
C1	0.3460(8)	0.3436(6)	0.2129(5)	0.027(4)
C2	0.2951(7)	0.4112(6)	0.1780(5)	0.020(4)
C3	0.1903(8)	0.4230(6)	0.1481(5)	0.024(4)
C4	0.1768(8)	0.5027(6)	0.1215(5)	0.023(4)
C5	0.2742(7)	0.5403(6)	0.1364(5)	0.021(4)
C6	0.3072(8)	0.6212(6)	0.1243(5)	0.022(4)
C7	0.3878(7)	0.6481(6)	0.0958(5)	0.023(4)
C8	0.3882(8)	0.7342(6)	0.0981(5)	0.025(4)
C9	0.3060(8)	0.7600(6)	0.1252(5)	0.025(4)
C10	0.2763(8)	0.8401(6)	0.1409(5)	0.021(4)
C11	0.1813(8)	0.8783(6)	0.1264(5)	0.025(4)
C12	0.1927(8)	0.9563(6)	0.1531(5)	0.020(4)
C13	0.2978(8)	0.9661(6)	0.1843(5)	0.023(4)
C14	0.3521(8)	1.0327(6)	0.2189(5)	0.026(4)
C15	0.1049(7)	0.3593(6)	0.1411(5)	0.030(4)
C16	0.0855(7)	0.3132(8)	0.0725(5)	0.038(4)
C17	0.0800(7)	0.5433(7)	0.0836(5)	0.032(4)
C18	0.4567(7)	0.5942(6)	0.0630(5)	0.032(4)
C19	0.4597(7)	0.7888(6)	0.0693(5)	0.033(4)
C20	0.0823(7)	0.8400(6)	0.0820(5)	0.038(5)
C21	0.1110(8)	1.0205(6)	0.1499(5)	0.032(4)
C22	0.0945(9)	1.0757(8)	0.0865(6)	0.058(6)
O1W	0.4343(4)	1.1906(4)	0.3042(3)	0.037(2)
O1'	0.5541(5)	0.8354(4)	0.2414(3)	0.031(3)
O2'	0.5516(5)	1.5373(4)	0.2344(3)	0.030(3)
N1'	0.6048(6)	0.9804(5)	0.1725(4)	0.023(3)
N2'	0.6649(5)	1.1844(6)	0.1364(3)	0.027(3)
N3'	0.6002(6)	1.3897(5)	0.1689(4)	0.025(3)
C1'	0.6344(8)	0.8408(6)	0.2199(5)	0.027(4)
C2'	0.6606(8)	0.9093(6)	0.1823(5)	0.028(4)
C3'	0.7439(7)	0.9180(6)	0.1506(5)	0.026(4)
C4'	0.7351(7)	0.9969(6)	0.1204(5)	0.024(4)
C5'	0.6490(8)	1.0342(6)	0.1359(5)	0.027(4)
C6'	0.6053(8)	1.1146(6)	0.1212(5)	0.025(4)
C7'	0.5019(8)	1.1387(6)	0.0953(5)	0.024(4)
C8'	0.5028(7)	1.2246(6)	0.0976(5)	0.020(4)
C9'	0.6021(7)	1.2525(6)	0.1234(5)	0.018(4)
C10'	0.6431(7)	1.3326(6)	0.1348(5)	0.025(4)
C11'	0.7286(7)	1.3693(6)	0.1167(5)	0.025(4)
C12'	0.7356(8)	1.4492(6)	0.1408(5)	0.028(4)

Supplementary Table II-2. (continued).

Atom	x	y	z	U
C13'	0.6559(8)	1.4627(6)	0.1746(5)	0.025(4)
C14'	0.6281(9)	1.5313(6)	0.2090(5)	0.031(4)
C15'	0.8264(7)	0.8546(6)	0.1481(5)	0.034(4)
C16'	0.8035(8)	0.8020(8)	0.0865(5)	0.058(5)
C17'	0.8036(8)	1.0350(7)	0.0771(5)	0.042(5)
C18'	0.4144(7)	1.0835(6)	0.0647(5)	0.032(4)
C19'	0.4087(7)	1.2777(6)	0.0697(5)	0.030(4)
C20'	0.7954(7)	1.3295(7)	0.0748(5)	0.035(4)
C21'	0.8142(8)	1.5130(6)	0.1322(6)	0.039(5)
C22'	0.7801(9)	1.5610(9)	0.0636(6)	0.060(6)
O1W'	0.6093(4)	0.6844(5)	0.3075(3)	0.040(2)

For anisotropic atoms, the U value is U_{eq}, calculated as $U_{eq} = 1/3 \sum_i \sum_j U_{ij} a_i^* a_j^* A_{ij}$ where A_{ij} is the dot product of the ith and j^{th} direct space unit cell vectors.

Supplementary Table II-3. Anisotropic thermal parameters for the non-hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Atom	U11	U22	U33	U12	U13	U23
O1	0.018(4)	0.037(5)	0.022(4)	0.002(3)	0.004(3)	0.007(3)
O2	0.023(4)	0.027(4)	0.038(5)	0.002(4)	0.010(4)	-0.002(4)
N1	0.011(4)	0.026(5)	0.029(5)	0.004(4)	0.006(4)	-0.002(4)
N2	0.022(4)	0.020(4)	0.029(4)	0.006(5)	0.012(3)	0.006(5)
N3	0.026(5)	0.023(5)	0.028(5)	0.007(4)	0.014(4)	0.004(4)
C1	0.033(7)	0.022(6)	0.028(6)	-0.003(5)	0.011(5)	-0.010(5)
C2	0.019(6)	0.016(6)	0.026(6)	0.000(5)	0.009(5)	-0.006(5)
C3	0.027(6)	0.032(7)	0.018(6)	-0.010(5)	0.012(5)	-0.007(5)
C4	0.026(6)	0.024(6)	0.020(6)	0.006(5)	0.007(5)	0.001(5)
C5	0.012(5)	0.027(6)	0.024(6)	0.005(5)	0.004(5)	0.001(5)
C6	0.021(6)	0.022(6)	0.025(6)	-0.008(5)	0.007(5)	-0.006(5)
C7	0.018(6)	0.029(7)	0.019(6)	-0.007(5)	-0.002(5)	0.001(5)
C8	0.033(7)	0.024(7)	0.021(6)	-0.002(5)	0.009(5)	-0.001(5)
C9	0.023(6)	0.030(7)	0.020(6)	-0.013(5)	-0.002(5)	0.002(5)
C10	0.025(6)	0.023(7)	0.013(6)	-0.005(5)	0.003(5)	-0.001(4)
C11	0.026(6)	0.034(7)	0.014(6)	0.005(5)	-0.000(5)	0.014(5)
C12	0.024(6)	0.018(6)	0.021(6)	0.001(5)	0.010(5)	0.016(5)
C13	0.028(6)	0.016(6)	0.025(6)	0.002(5)	0.009(5)	-0.002(5)
C14	0.037(7)	0.020(6)	0.027(6)	0.005(5)	0.017(6)	0.002(5)
C15	0.023(6)	0.022(6)	0.048(8)	-0.004(5)	0.011(6)	-0.004(5)
C16	0.030(6)	0.021(5)	0.063(7)	-0.015(6)	0.011(5)	-0.017(7)
C17	0.022(6)	0.033(8)	0.038(8)	0.005(5)	-0.002(6)	-0.008(6)
C18	0.027(6)	0.045(8)	0.025(6)	0.005(6)	0.009(5)	-0.003(5)
C19	0.033(6)	0.045(8)	0.026(6)	-0.011(5)	0.012(5)	0.005(5)
C20	0.039(7)	0.041(9)	0.033(7)	-0.012(6)	0.005(6)	0.002(6)
C21	0.032(7)	0.039(7)	0.027(6)	0.008(6)	0.011(5)	-0.002(5)
C22	0.064(10)	0.064(10)	0.050(9)	0.029(8)	0.017(8)	0.013(8)
O1W	0.047(4)	0.020(4)	0.049(4)	0.006(4)	0.021(3)	0.001(4)
O1'	0.021(4)	0.034(5)	0.036(4)	-0.002(3)	0.002(3)	0.001(4)
O2'	0.028(4)	0.028(4)	0.035(5)	-0.005(4)	0.009(4)	-0.004(4)
N1'	0.011(4)	0.026(5)	0.031(5)	0.004(4)	0.002(4)	-0.002(4)
N2'	0.022(4)	0.027(5)	0.033(5)	0.006(5)	0.008(4)	0.002(5)
N3'	0.018(5)	0.030(5)	0.027(5)	-0.002(4)	0.005(4)	0.004(4)
C1'	0.025(6)	0.028(7)	0.020(6)	0.003(5)	-0.010(5)	-0.000(5)
C2'	0.026(6)	0.028(7)	0.030(7)	0.005(5)	0.003(5)	0.000(5)
C3'	0.018(6)	0.032(7)	0.026(6)	-0.005(5)	0.001(5)	-0.007(5)
C4'	0.019(6)	0.027(6)	0.028(6)	-0.005(5)	0.005(5)	-0.002(5)
C5'	0.023(6)	0.031(7)	0.031(7)	-0.007(5)	0.011(5)	0.010(5)
C6'	0.025(6)	0.029(7)	0.023(6)	-0.003(5)	0.007(5)	0.011(5)
C7'	0.025(6)	0.032(7)	0.013(6)	-0.003(5)	0.004(5)	0.002(5)
C8'	0.022(6)	0.022(6)	0.019(6)	-0.002(5)	0.009(5)	0.002(5)
C9'	0.013(6)	0.010(5)	0.031(7)	0.002(4)	0.002(5)	0.000(5)
C10'	0.018(6)	0.034(8)	0.021(6)	-0.003(5)	0.006(5)	0.003(5)
C11'	0.019(6)	0.029(6)	0.030(7)	0.008(5)	0.013(5)	0.007(5)
C12'	0.024(6)	0.025(7)	0.033(6)	-0.003(5)	0.002(5)	0.020(5)

Supplementary Table II-3. (continued).

Atom	U11	U22	U33	U12	U13	U23
C13'	0.017(6)	0.020(6)	0.038(7)	0.007(5)	0.006(5)	0.010(5)
C14'	0.046(8)	0.017(6)	0.034(7)	-0.011(5)	0.014(6)	-0.001(5)
C15'	0.015(6)	0.040(7)	0.047(7)	0.002(5)	0.009(5)	-0.006(6)
C16'	0.038(7)	0.066(10)	0.063(8)	-0.007(8)	-0.002(6)	-0.052(8)
C17'	0.040(7)	0.044(9)	0.046(8)	-0.004(6)	0.019(6)	-0.008(6)
C18'	0.017(6)	0.032(7)	0.042(7)	-0.007(5)	-0.004(5)	0.007(6)
C19'	0.035(6)	0.036(7)	0.023(6)	0.008(5)	0.015(5)	0.003(5)
C20'	0.023(6)	0.041(9)	0.042(7)	-0.010(6)	0.009(5)	0.002(6)
C21'	0.029(7)	0.026(7)	0.064(9)	-0.016(5)	0.015(6)	-0.011(6)
C22'	0.045(8)	0.073(10)	0.071(9)	-0.010(8)	0.032(7)	0.036(8)
O1W'	0.035(4)	0.029(4)	0.052(4)	0.001(4)	-0.000(3)	-0.004(5)

The Uij are the mean-square amplitudes of vibration in $Å^2$ from the general temperature factor expression

$$\exp[-2\pi^2(h^2a^{*2}U11 + k^2b^{*2}U22 + l^2c^{*2}U33 + 2hka^*b^*U12 + 2hla^*c^*U13 + 2klb^*c^*U23)]$$

Supplementary Table II-4. Fractional coordinates and isotropic thermal parameters (Å^2) for the hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

Atom	x	y	z	U
H1N	0.4121	0.4932	0.1850	0.026
H2N	0.2003	0.6881	0.1583	0.027
H3N	0.4131	0.8825	0.1905	0.029
H1	0.3044	0.2980	0.2199	0.032
H14	0.3116	1.0790	0.2260	0.032
H15A	0.0426	0.3867	0.1447	0.037
H15B	0.1219	0.3204	0.1779	0.037
H16A	0.0306	0.2744	0.0691	0.057
H16B	0.0676	0.3521	0.0358	0.057
H16C	0.1476	0.2853	0.0692	0.057
H17A	0.0933	0.5987	0.0726	0.048
H17B	0.0534	0.5136	0.0419	0.048
H17C	0.0305	0.5425	0.1118	0.048
H18A	0.4395	0.5381	0.0684	0.048
H18B	0.4465	0.6067	0.0149	0.048
H18C	0.5276	0.6034	0.0854	0.048
H19A	0.4461	0.8450	0.0774	0.050
H19B	0.5298	0.7761	0.0911	0.050
H19C	0.4486	0.7793	0.0207	0.050
H20A	0.0960	0.7851	0.0701	0.057
H20B	0.0292	0.8399	0.1075	0.057
H20C	0.0600	0.8716	0.0408	0.057
H21A	0.1291	1.0550	0.1898	0.038
H21B	0.0473	0.9938	0.1510	0.038
H22A	0.0411	1.1150	0.0869	0.088
H22B	0.1576	1.1035	0.0856	0.088
H22C	0.0750	1.0418	0.0465	0.088
H1WA	0.4562	1.1523	0.2871	0.045
H1WB	0.4223	1.2344	0.2866	0.045
H1'N	0.5477	0.9902	0.1882	0.028
H2'N	0.7338	1.1850	0.1522	0.032
H3'N	0.5435	1.3822	0.1855	0.030
H1'	0.6827	0.7965	0.2293	0.032
H14'	0.6731	1.5778	0.2144	0.038
H15C	0.8355	0.8208	0.1883	0.040
H15D	0.8902	0.8824	0.1493	0.040
H16D	0.8574	0.7631	0.0867	0.086
H16E	0.7401	0.7738	0.0858	0.086
H16F	0.7953	0.8360	0.0465	0.086
H17D	0.7800	1.0890	0.0627	0.063
H17E	0.8725	1.0378	0.1048	0.063
H17F	0.8029	1.0019	0.0373	0.063
H18D	0.4379	1.0282	0.0727	0.048
H18E	0.3571	1.0924	0.0857	0.048

Supplementary Table II-4. (continued).

Atom	x	y	z	U
H18F	0.3931	1.0930	0.0161	0.048
H19D	0.4266	1.3344	0.0753	0.045
H19E	0.3824	1.2661	0.0218	0.045
H19F	0.3569	1.2653	0.0947	0.045
H20D	0.8488	1.3669	0.0702	0.052
H20E	0.8258	1.2808	0.0974	0.052
H20F	0.7548	1.3159	0.0300	0.052
H21C	0.8794	1.4873	0.1336	0.047
H21D	0.8231	1.5522	0.1689	0.047
H22D	0.8291	1.6015	0.0570	0.090
H22E	0.7717	1.5214	0.0273	0.090
H22F	0.7151	1.5867	0.0628	0.090
H1WC	0.5796	0.6465	0.2869	0.048
H1WD	0.5841	0.7234	0.2860	0.048

Supplementary Table II-5. Bond Lengths (Å) and Angles (°) for the non-hydrogen atoms of $C_{22}H_{27}N_3O_2 \cdot H_2O$.

1	2	3	1-2	1-2-3
C1	O1		1.243(12)	
C14	O2		1.250(12)	
C2	N1	C5	1.387(12)	110.6(7)
C5	N1		1.373(12)	
C6	N2	C9	1.379(13)	109.9(8)
C9	N2		1.401(14)	
C10	N3	C13	1.338(12)	110.3(8)
C13	N3		1.384(13)	
C2	C1	O1	1.399(13)	125.8(9)
C3	C2	N1	1.402(13)	106.3(8)
C3	C2	C1		130.2(9)
N1	C2	C1		123.4(8)
C4	C3	C15	1.407(14)	124.9(8)
C4	C3	C2		108.6(9)
C15	C3	C2	1.526(14)	126.4(9)
C5	C4	C17	1.405(14)	124.1(9)
C5	C4	C3		107.2(8)
C17	C4	C3	1.495(13)	128.7(9)
C6	C5	N1	1.436(14)	120.4(8)
C6	C5	C4		132.2(9)
N1	C5	C4		107.4(8)
C7	C6	N2	1.394(15)	107.5(8)
C7	C6	C5		130.8(10)
N2	C6	C5		121.6(9)
C8	C7	C18	1.413(14)	126.5(10)
C8	C7	C6		107.5(9)
C18	C7	C6	1.525(15)	125.8(9)
C9	C8	C19	1.39(2)	125.8(9)
C9	C8	C7		108.6(9)
C19	C8	C7	1.51(2)	125.3(10)
C10	C9	N2	1.427(15)	123.1(10)
C10	C9	C8		130.3(10)
N2	C9	C8		106.4(9)
C11	C10	N3	1.381(14)	107.7(9)
C11	C10	C9		131.1(9)
N3	C10	C9		121.2(9)
C12	C11	C20	1.382(14)	127.0(9)
C12	C11	C10		108.9(8)
C20	C11	C10	1.546(13)	123.9(9)
C13	C12	C21	1.406(13)	125.2(9)
C13	C12	C11		106.7(9)
C21	C12	C11	1.505(14)	128.1(9)
C14	C13	N3	1.403(13)	122.5(9)
C14	C13	C12		131.0(10)
N3	C13	C12		106.5(8)
O2	C14	C13		127.9(10)
C16	C15	C3	1.534(15)	113.0(9)

Supplementary Table II-5. (continued).

1	2	3	1-2	1-2-3
C22	C21	C12	1.53(2)	114.6(9)
C1'	O1'		1.241(13)	
C14'	O2'		1.241(14)	
C2'	N1'	C5'	1.372(13)	110.0(9)
C5'	N1'		1.361(14)	
C6'	N2'	C9'	1.388(13)	109.5(7)
C9'	N2'		1.383(13)	
C10'	N3'	C13'	1.357(13)	110.9(9)
C13'	N3'		1.399(13)	
C2'	C1'	O1'	1.437(15)	124.4(9)
C3'	C2'	N1'	1.40(2)	107.9(9)
C3'	C2'	C1'		128.7(10)
N1'	C2'	C1'		123.4(10)
C4'	C3'	C15'	1.421(14)	126.9(10)
C4'	C3'	C2'		106.6(9)
C15'	C3'	C2'	1.521(14)	126.5(9)
C5'	C4'	C17'	1.39(2)	125.0(9)
C5'	C4'	C3'		107.4(9)
C17'	C4'	C3'	1.53(2)	127.6(9)
C6'	C5'	N1'	1.444(14)	119.9(10)
C6'	C5'	C4'		132.0(10)
N1'	C5'	C4'		108.2(9)
C7'	C6'	N2'	1.413(14)	108.1(9)
C7'	C6'	C5'		130.1(9)
N2'	C6'	C5'		121.7(9)
C8'	C7'	C18'	1.409(14)	128.2(9)
C8'	C7'	C6'		105.6(8)
C18'	C7'	C6'	1.494(13)	125.8(9)
C9'	C8'	C19'	1.384(13)	125.8(9)
C9'	C8'	C7'		110.0(8)
C19'	C8'	C7'	1.524(13)	124.0(8)
C10'	C9'	N2'	1.421(13)	121.5(8)
C10'	C9'	C8'		131.7(9)
N2'	C9'	C8'		106.8(8)
C11'	C10'	N3'	1.406(15)	106.9(9)
C11'	C10'	C9'		131.2(10)
N3'	C10'	C9'		122.0(9)
C12'	C11'	C20'	1.392(15)	126.9(10)
C12'	C11'	C10'		108.0(9)
C20'	C11'	C10'	1.50(2)	125.0(9)
C13'	C12'	C21'	1.40(2)	124.2(9)
C13'	C12'	C11'		108.5(9)
C21'	C12'	C11'	1.52(2)	127.2(10)
C14'	C13'	N3'	1.409(15)	122.0(10)
C14'	C13'	C12'		132.3(10)
N3'	C13'	C12'		105.7(9)
O2'	C14'	C13'		126.4(9)
C16'	C15'	C3'	1.48(2)	114.3(8)
C22'	C21'	C12'	1.56(2)	112.1(8)