Submitted to *J. Org. Chem.*
Version 1 of September 23, 2002

Supporting Information

Molecular Tectonics.
Construction of Porous Hydrogen-Bonded Networks
from Bisketals of Pentaerythritol

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Contents

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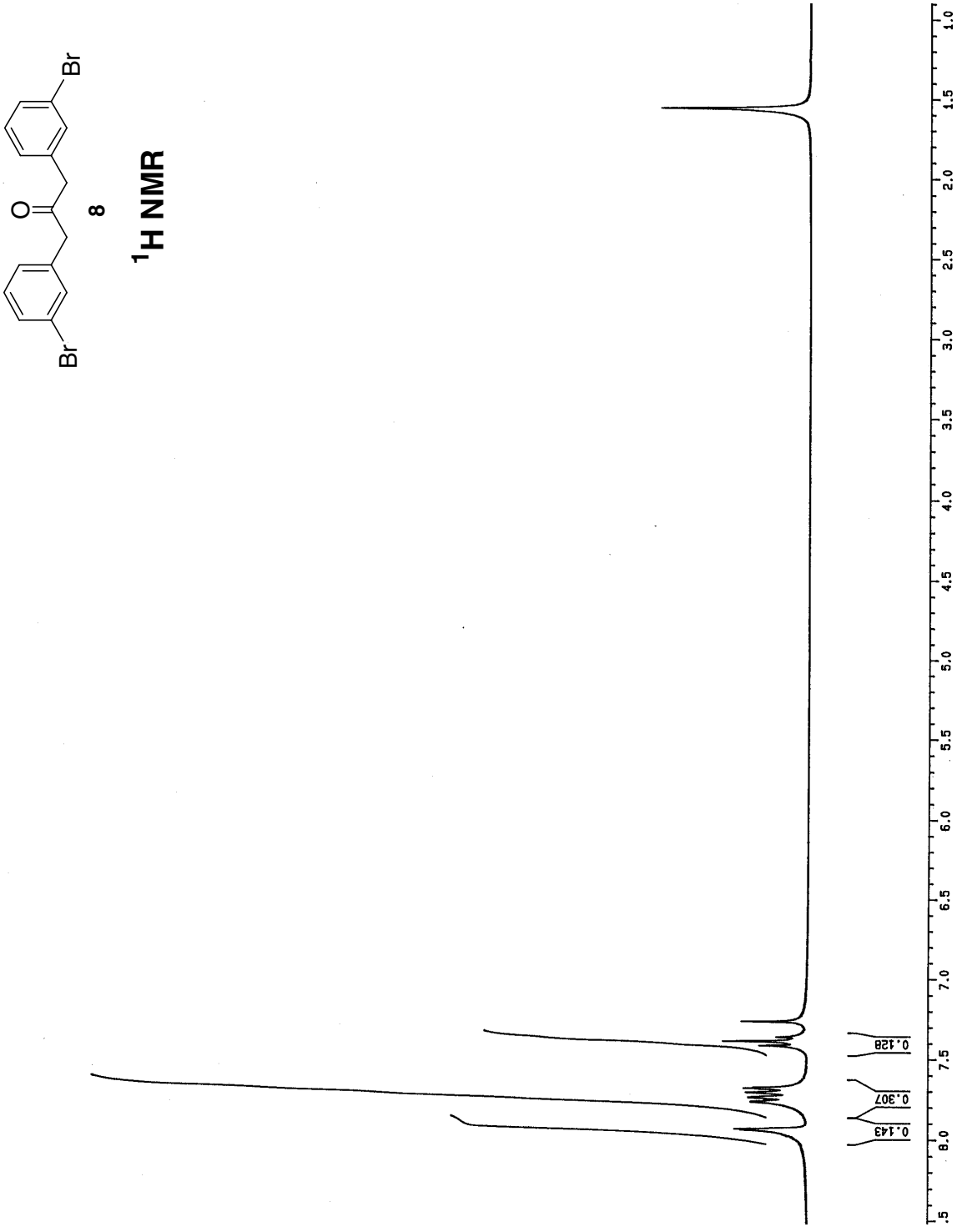
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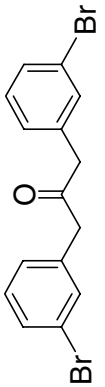

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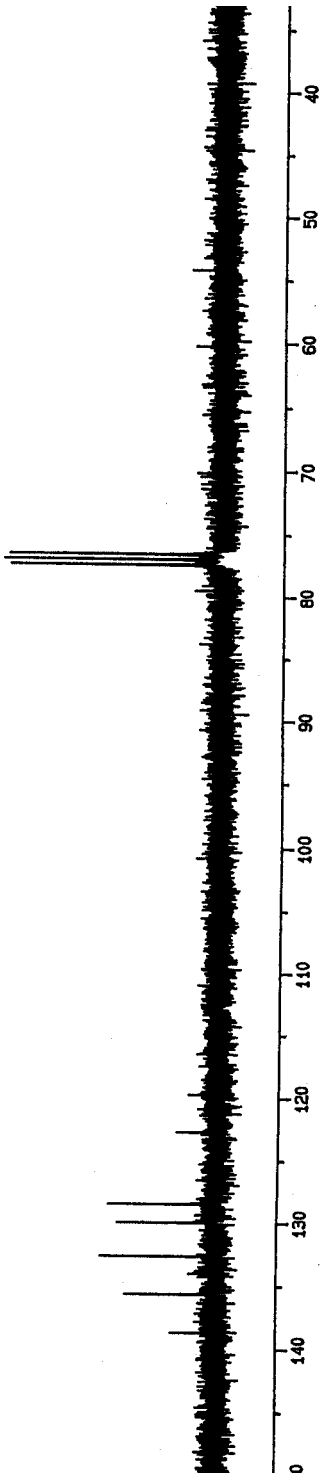

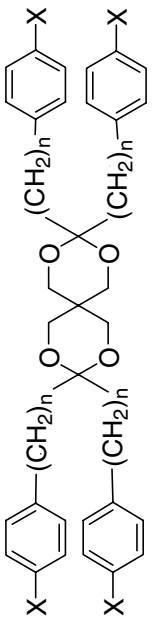

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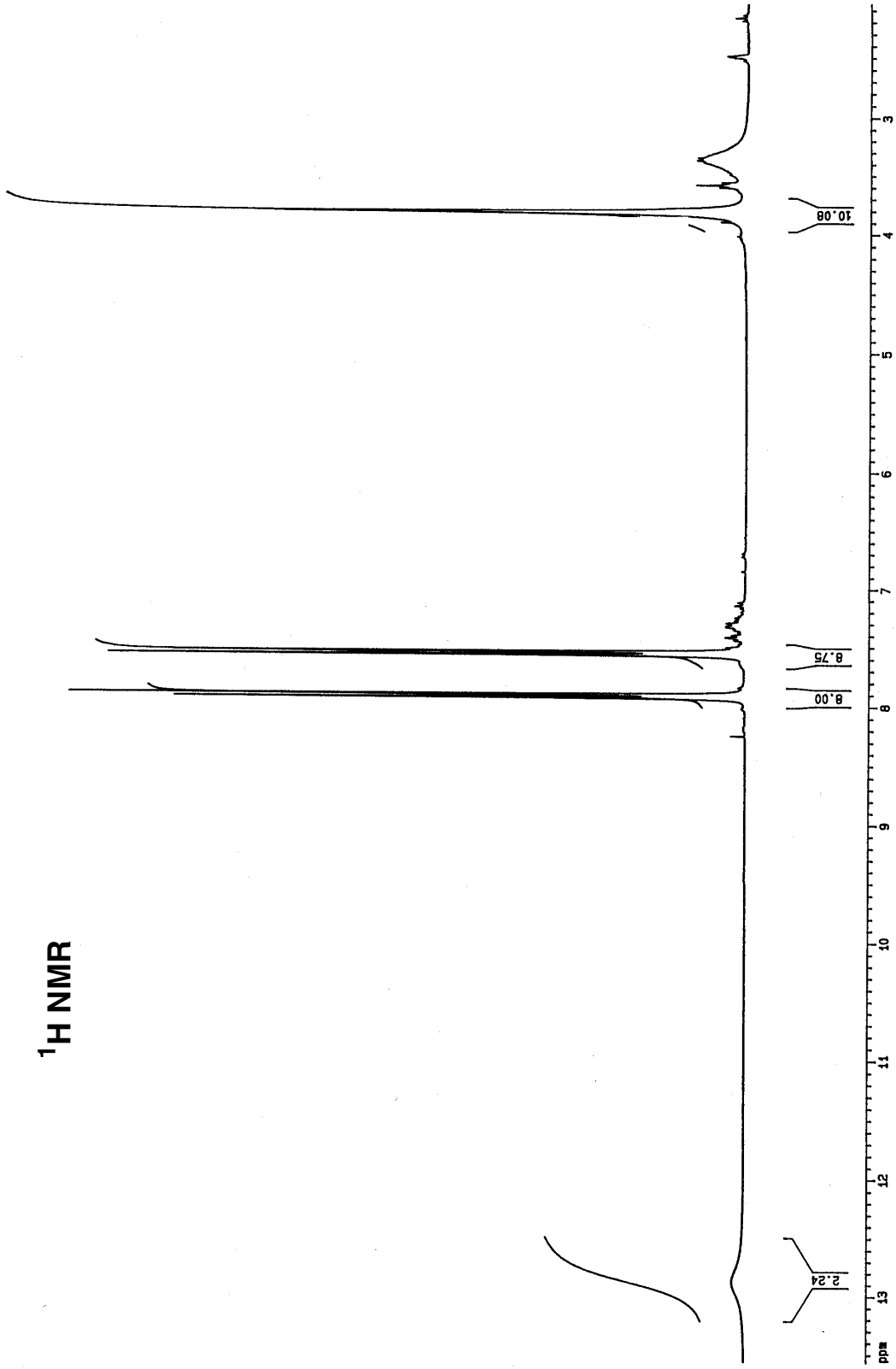

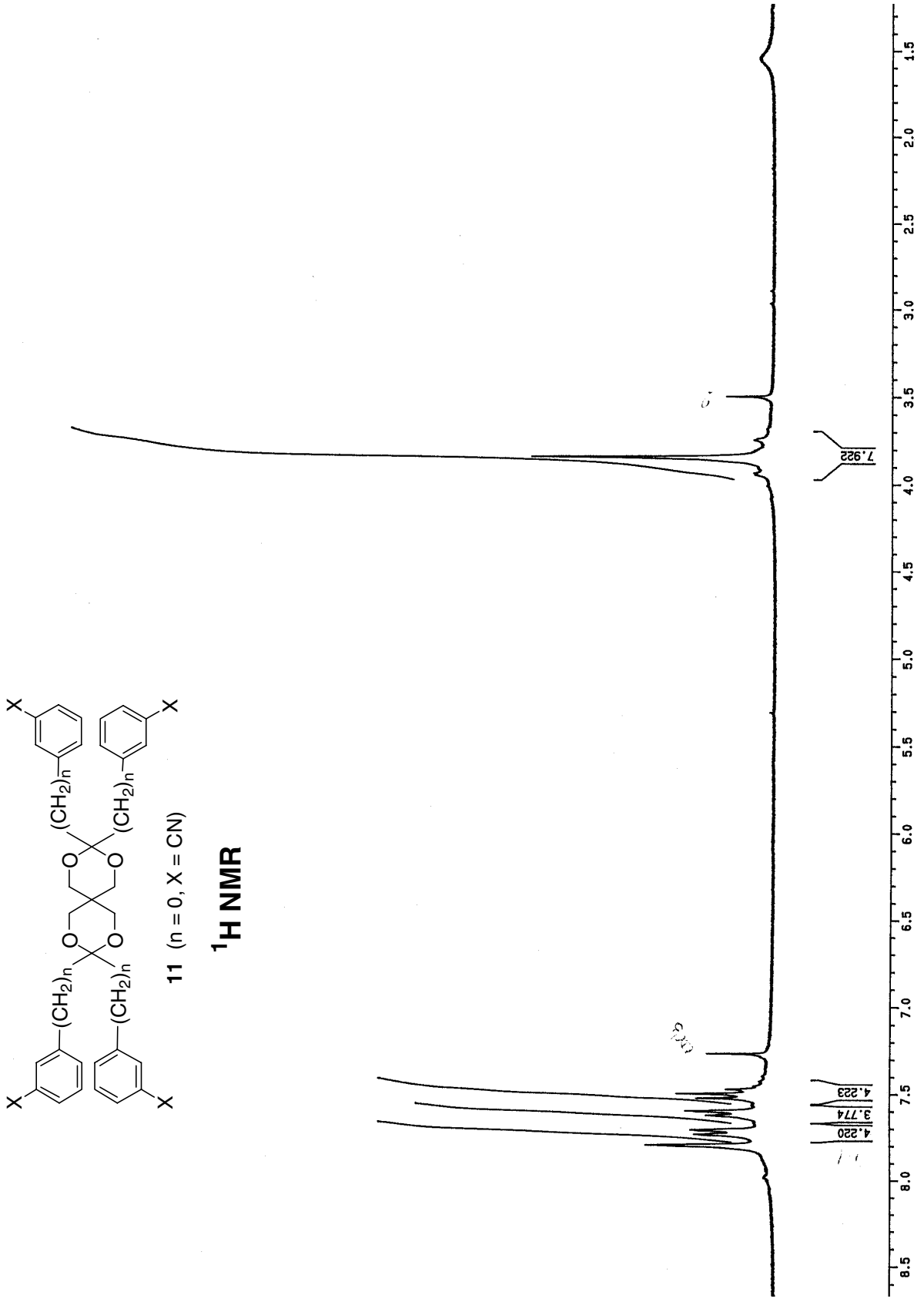

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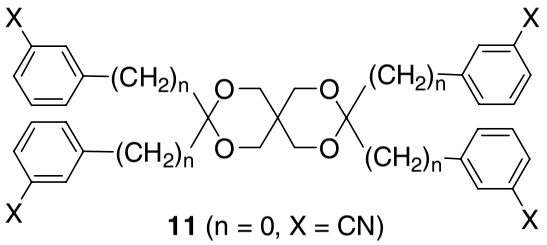
^{1}H NMR



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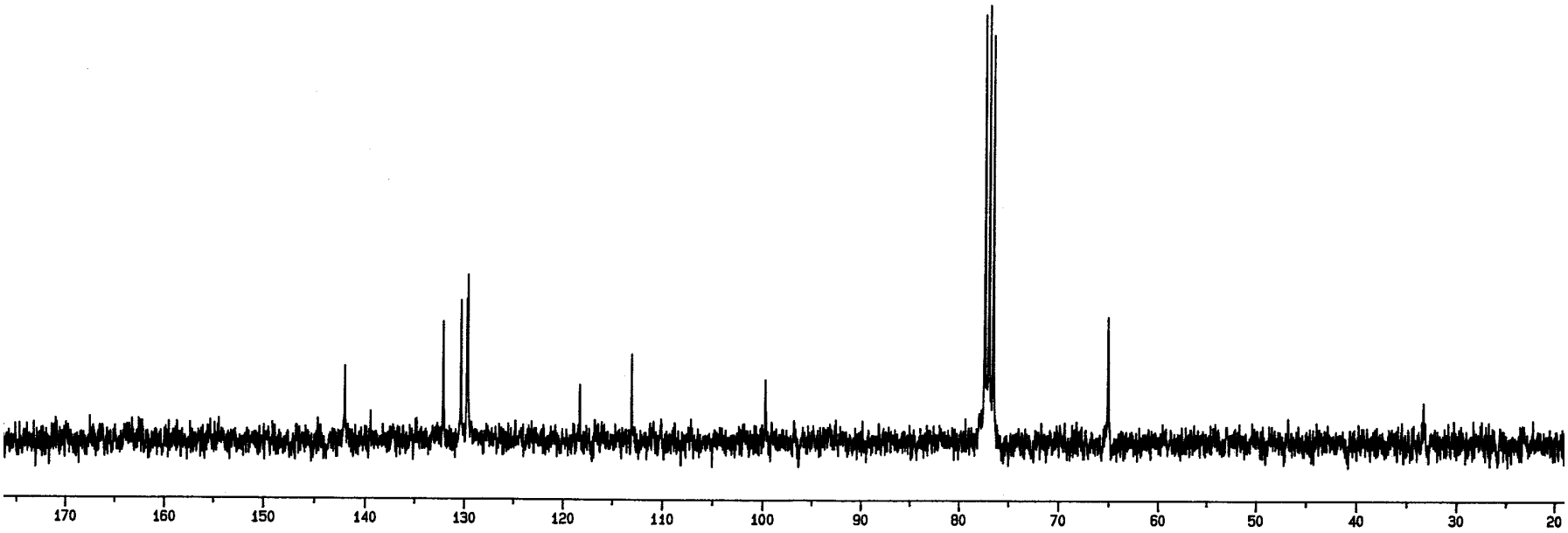

Université de Montréal

CRYSTAL AND MOLECULAR STRUCTURE OF

C83 H132 N32 O16 COMPOUND (JIW257)

8 janvier 2001

Equipe WUEST

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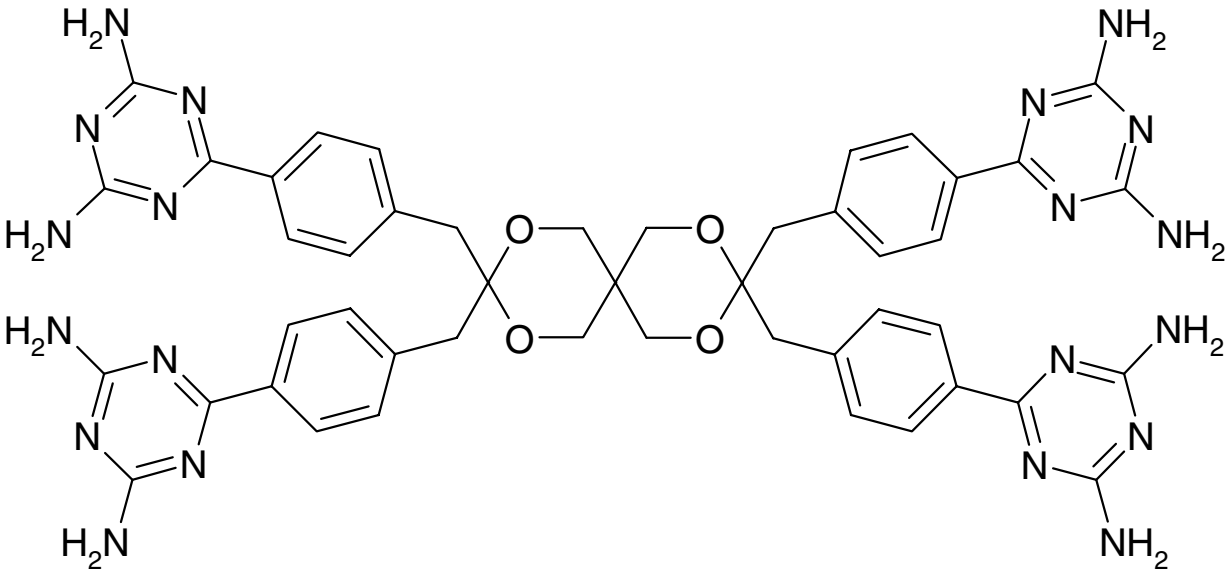


Solvant : DMF.
Formule estimée avec une molécule tectonique pour 12 molécules de DMF, donnant une densité de 1.3.
C47 H48 N20 O4 12 (C3H7NO)

Structure mesurée et résolue à Ottawa par le Pr Gary Enright, affinée au laboratoire de diffraction des rayons X de l'Université de Montréal par le Dr. Thierry Maris.

Table 1. Crystal data and structure refinement for C83 H132 N32 O16.

Identification code	JIW257
Empirical formula	C83 H132 N32 O16
Formula weight	957.05
Temperature	220(2)K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	Pbcn
Unit cell dimensions	a = 23.6973(17) Å α = 90° b = 19.9952(15) Å β = 90° c = 20.2313(15) Å γ = 90°
Volume	9586.2(12) $Å^3$
Z	4
Density (calculated)	1.287 Mg/m^3
Absorption coefficient	0.085 mm^{-1}
F(000)	2008
Crystal size	0.9 x 0.15 x 0.20 mm
Theta range for data collection	1.33 to 22.50°
Index ranges	$-25 \leq h \leq 25$, $-21 \leq k \leq 21$, $-21 \leq \ell \leq 21$
Reflections collected	58495
Independent reflections	6277 [R_{int} = 0.1099]
Absorption correction	Semi-empirical from equivalents
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6277 / 0 / 321
Goodness-of-fit on F^2	0.969
Final R indices [I>2sigma(I)]	R_1 = 0.0836, wR_2 = 0.1886
R indices (all data)	R_1 = 0.1831, wR_2 = 0.2203
Largest diff. peak and hole	0.173 and -0.099 e/$Å^3$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for C83 H132 N32 O16.

U_{eq} is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U_{eq}
O(1)	5112(1)	3433(1)	11586(1)	81(1)
O(2)	4528(2)	4350(2)	11467(2)	114(1)
N(1)	6760(2)	2150(2)	8914(2)	120(2)
N(2)	7178(2)	2113(2)	7841(2)	125(2)
N(3)	6678(2)	3077(2)	8193(2)	95(1)
N(4)	7043(2)	3032(2)	7162(2)	127(2)
N(5)	7289(3)	1249(3)	8562(2)	201(3)
N(6)	3359(2)	4486(2)	7962(2)	83(1)
N(7)	2955(2)	5481(2)	7502(2)	96(1)
N(8)	3235(2)	5443(2)	8637(2)	95(1)
N(9)	3065(2)	4544(2)	6861(2)	109(2)
N(10)	2854(2)	6395(2)	8184(2)	158(2)
C(1)	5000	4192(3)	12500	80(2)
C(2)	5403(2)	3770(3)	12108(2)	96(2)
C(3)	4699(3)	4644(3)	11990(2)	141(3)
C(4)	5288(2)	4230(2)	10695(2)	83(1)
C(5)	5591(2)	3822(2)	10199(2)	76(1)
C(6)	5920(2)	3288(3)	10389(2)	99(2)
C(7)	6243(2)	2922(3)	9933(2)	114(2)
C(8)	6253(2)	3093(2)	9266(2)	85(1)
C(9)	5931(2)	3632(2)	9075(2)	87(2)
C(10)	5608(2)	3984(2)	9525(2)	88(1)
C(11)	6591(2)	2747(3)	8749(2)	93(2)
C(12)	7066(3)	1849(3)	8436(3)	140(2)
C(13)	6975(2)	2717(3)	7732(2)	108(2)
C(14)	4436(2)	3454(2)	10736(2)	96(2)
C(15)	4149(2)	3819(3)	10170(2)	93(2)
C(16)	4230(2)	3565(2)	9519(2)	97(2)
C(17)	3969(2)	3887(3)	9003(2)	100(2)
C(18)	3636(2)	4436(3)	9088(2)	82(1)
C(19)	3561(2)	4683(3)	9731(3)	113(2)
C(20)	3799(2)	4348(3)	10253(2)	109(2)
C(21)	3379(2)	4829(2)	8526(2)	69(1)
C(22)	3122(2)	4853(3)	7451(3)	84(1)
C(23)	3027(2)	5772(3)	8102(3)	94(2)
C(30)	4863(2)	3876(2)	11118(2)	78(1)

Table 3. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for C83 H132 N32 O16.

	x	y	z	U_{eq}
H(4A)	7226	2843	6845	153
H(4B)	6904	3426	7108	153
H(5A)	7493	1052	8269	241
H(5B)	7230	1059	8937	241
H(9A)	2923	4757	6531	131
H(9B)	3170	4135	6816	131
H(10A)	2699	6606	7861	189
H(10B)	2895	6589	8560	189
H(2A)	5697	4050	11923	116
H(2B)	5579	3443	12396	116
H(3A)	4377	4852	12203	170
H(3B)	4956	4999	11862	170
H(4C)	5097	4590	10464	100
H(4D)	5566	4433	10983	100
H(6)	5930	3167	10832	118
H(7)	6453	2558	10079	136
H(9A)	5930	3762	8634	105
H(10A)	5393	4342	9375	106
H(14A)	4628	3064	10560	116
H(14B)	4149	3298	11041	116
H(16)	4455	3191	9445	116
H(17)	4021	3722	8577	119
H(19)	3351	5069	9803	136
H(20)	3718	4490	10680	131

Table 4. Anisotropic parameters ($Å^2$ x 10^3) for C83 H132 N32 O16.

The anisotropic displacement factor exponent takes the form:

$$-2\pi^2 [h^2 a^{*2} U_{11} + ... + 2 h k a^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
O(1)	105(2)	94(2)	45(2)	7(2)	3(2)	31(2)
O(2)	167(3)	128(3)	48(2)	-25(2)	-19(2)	58(2)
N(1)	173(4)	115(4)	71(3)	21(3)	50(3)	58(3)
N(2)	188(5)	96(4)	90(3)	29(3)	35(3)	48(3)
N(3)	130(3)	97(3)	58(2)	10(2)	26(2)	20(2)
N(4)	188(5)	119(4)	75(3)	21(3)	51(3)	56(3)
N(5)	332(8)	149(5)	121(4)	54(4)	99(4)	134(5)
N(6)	100(3)	77(3)	71(3)	1(2)	-1(2)	24(2)
N(7)	104(3)	108(4)	76(3)	-17(3)	-34(2)	13(3)
N(8)	129(3)	83(3)	74(3)	-21(2)	-32(2)	32(3)
N(9)	183(4)	98(3)	47(2)	-16(2)	-18(2)	19(3)
N(10)	256(6)	108(4)	110(4)	-28(3)	-87(4)	87(4)
C(1)	79(5)	109(5)	53(4)	0	10(4)	0
C(2)	69(3)	166(5)	54(3)	23(3)	0(3)	-17(3)
C(3)	234(7)	131(5)	59(3)	-9(4)	-27(4)	78(5)
C(4)	124(4)	86(3)	39(2)	4(2)	12(3)	13(3)
C(5)	96(3)	78(3)	54(3)	27(2)	3(2)	32(3)
C(6)	124(4)	136(5)	36(2)	13(3)	26(3)	42(4)
C(7)	159(5)	121(4)	60(3)	43(3)	14(3)	59(4)
C(8)	109(4)	65(3)	81(3)	11(3)	2(3)	37(3)
C(9)	125(4)	97(4)	40(2)	-15(2)	18(3)	32(3)
C(10)	128(4)	77(3)	60(3)	27(3)	-9(3)	30(3)
C(11)	105(4)	100(4)	75(4)	18(3)	18(3)	41(3)
C(12)	208(7)	119(5)	93(4)	50(4)	46(4)	65(5)
C(13)	169(5)	94(4)	59(3)	26(3)	35(3)	25(4)
C(14)	123(4)	95(4)	71(3)	-19(3)	8(3)	3(3)
C(15)	95(4)	136(5)	49(3)	8(3)	-16(3)	35(3)
C(16)	118(4)	101(4)	70(3)	-4(3)	-18(3)	38(3)
C(17)	108(4)	120(5)	71(3)	-12(3)	-15(3)	35(4)
C(18)	63(3)	116(4)	68(3)	-14(3)	-23(2)	10(3)
C(19)	140(5)	109(4)	91(4)	-19(3)	-37(3)	55(3)
C(20)	95(4)	167(5)	66(3)	-11(3)	-6(3)	74(4)
C(21)	72(3)	71(3)	63(3)	-17(3)	-12(2)	17(2)
C(22)	86(4)	70(4)	95(4)	-7(3)	3(3)	19(3)
C(23)	117(4)	78(4)	86(4)	-8(3)	-21(3)	26(3)
C(30)	122(4)	69(3)	42(2)	-4(2)	-9(3)	27(3)

Table 5. Bond lengths [Å] and angles [°] for C83 H132 N32 O16

O(1)-C(30)	1.423(4)	C(2)#1-C(1)-C(3)#1	105.6(3)
O(1)-C(2)	1.430(5)	C(2)-C(1)-C(3)	105.6(3)
O(2)-C(3)	1.277(5)	C(2)#1-C(1)-C(3)	112.8(3)
O(2)-C(30)	1.424(5)	C(3)#1-C(1)-C(3)	108.4(6)
N(1)-C(11)	1.304(5)	O(1)-C(2)-C(1)	110.4(3)
N(1)-C(12)	1.350(6)	O(2)-C(3)-C(1)	115.4(5)
N(2)-C(13)	1.319(6)	C(5)-C(4)-C(30)	116.9(4)
N(2)-C(12)	1.340(6)	C(6)-C(5)-C(10)	115.8(4)
N(3)-C(11)	1.319(5)	C(6)-C(5)-C(4)	120.9(4)
N(3)-C(13)	1.374(6)	C(10)-C(5)-C(4)	123.1(4)
N(4)-C(13)	1.324(5)	C(5)-C(6)-C(7)	122.0(4)
N(5)-C(12)	1.337(6)	C(8)-C(7)-C(6)	121.1(4)
N(6)-C(21)	1.333(5)	C(9)-C(8)-C(7)	117.1(4)
N(6)-C(22)	1.387(5)	C(9)-C(8)-C(11)	117.7(4)
N(7)-C(22)	1.321(5)	C(7)-C(8)-C(11)	125.2(4)
N(7)-C(23)	1.357(5)	C(8)-C(9)-C(10)	121.4(4)
N(8)-C(21)	1.293(5)	C(9)-C(10)-C(5)	122.6(4)
N(8)-C(23)	1.360(5)	N(1)-C(11)-N(3)	128.9(4)
N(9)-C(22)	1.349(5)	N(1)-C(11)-C(8)	114.3(5)
N(10)-C(23)	1.323(5)	N(3)-C(11)-C(8)	116.8(5)
C(1)-C(2)	1.502(6)	N(5)-C(12)-N(2)	116.5(6)
C(1)-C(2)#1	1.502(6)	N(5)-C(12)-N(1)	118.3(5)
C(1)-C(3)#1	1.546(6)	N(2)-C(12)-N(1)	125.1(5)
C(1)-C(3)	1.546(6)	N(2)-C(13)-N(4)	122.5(5)
C(4)-C(5)	1.480(5)	N(2)-C(13)-N(3)	123.6(4)
C(4)-C(30)	1.500(6)	N(4)-C(13)-N(3)	113.9(5)
C(5)-C(6)	1.376(5)	C(15)-C(14)-C(30)	114.4(4)
C(5)-C(10)	1.401(5)	C(20)-C(15)-C(16)	118.5(4)
C(6)-C(7)	1.404(6)	C(20)-C(15)-C(14)	123.8(4)
C(7)-C(8)	1.393(6)	C(16)-C(15)-C(14)	117.6(4)
C(8)-C(9)	1.375(5)	C(17)-C(16)-C(15)	118.2(4)
C(8)-C(11)	1.488(6)	C(18)-C(17)-C(16)	122.9(4)
C(9)-C(10)	1.382(5)	C(17)-C(18)-C(19)	118.2(4)
C(14)-C(15)	1.520(6)	C(17)-C(18)-C(21)	124.0(4)
C(14)-C(30)	1.527(6)	C(19)-C(18)-C(21)	117.6(5)
C(15)-C(20)	1.354(6)	C(20)-C(19)-C(18)	119.3(4)
C(15)-C(16)	1.424(6)	C(15)-C(20)-C(19)	122.6(4)
C(16)-C(17)	1.374(6)	N(8)-C(21)-N(6)	129.0(4)
C(17)-C(18)	1.363(6)	N(8)-C(21)-C(18)	118.0(4)
C(18)-C(19)	1.403(6)	N(6)-C(21)-C(18)	112.9(4)
C(18)-C(21)	1.510(6)	N(7)-C(22)-N(9)	118.3(5)
C(19)-C(20)	1.371(6)	N(7)-C(22)-N(6)	124.5(5)
		N(9)-C(22)-N(6)	117.3(5)
C(30)-O(1)-C(2)	113.4(3)	N(10)-C(23)-N(7)	118.5(5)
C(3)-O(2)-C(30)	122.7(4)	N(10)-C(23)-N(8)	117.9(5)
C(11)-N(1)-C(12)	112.9(4)	N(7)-C(23)-N(8)	123.4(5)
C(13)-N(2)-C(12)	116.0(5)	O(1)-C(30)-O(2)	108.4(3)
C(11)-N(3)-C(13)	113.4(4)	O(1)-C(30)-C(4)	113.3(4)
C(21)-N(6)-C(22)	112.3(4)	O(2)-C(30)-C(4)	110.1(4)
C(22)-N(7)-C(23)	116.1(4)	O(1)-C(30)-C(14)	105.5(4)
C(21)-N(8)-C(23)	114.5(4)	O(2)-C(30)-C(14)	104.4(4)
C(2)-C(1)-C(2)#1	111.5(6)	C(4)-C(30)-C(14)	114.6(4)
C(2)-C(1)-C(3)#1	112.8(3)		

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y,-z+5/2

Table 6. Torsion angles [°] for C83 H132 N32 O16.

C(30)-O(1)-C(2)-C(1)	62.2(5)	C(20)-C(15)-C(16)-C(17)	2.7(8)
C(2)#1-C(1)-C(2)-O(1)	69.2(3)	C(14)-C(15)-C(16)-C(17)	179.4(5)
C(3)#1-C(1)-C(2)-O(1)	-172.0(4)	C(15)-C(16)-C(17)-C(18)	0.0(8)
C(3)-C(1)-C(2)-O(1)	-53.7(5)	C(16)-C(17)-C(18)-C(19)	0.3(8)
C(30)-O(2)-C(3)-C(1)	-43.8(7)	C(16)-C(17)-C(18)-C(21)	175.3(5)
C(2)-C(1)-C(3)-O(2)	45.3(7)	C(17)-C(18)-C(19)-C(20)	-3.1(8)
C(2)#1-C(1)-C(3)-O(2)	-76.8(6)	C(21)-C(18)-C(19)-C(20)	-178.4(5)
C(3)#1-C(1)-C(3)-O(2)	166.5(7)	C(16)-C(15)-C(20)-C(19)	-5.7(9)
C(30)-C(4)-C(5)-C(6)	-60.5(6)	C(14)-C(15)-C(20)-C(19)	177.7(5)
C(30)-C(4)-C(5)-C(10)	125.8(5)	C(18)-C(19)-C(20)-C(15)	6.0(9)
C(10)-C(5)-C(6)-C(7)	-1.3(8)	C(23)-N(8)-C(21)-N(6)	1.3(7)
C(4)-C(5)-C(6)-C(7)	-175.5(5)	C(23)-N(8)-C(21)-C(18)	177.6(4)
C(5)-C(6)-C(7)-C(8)	1.5(9)	C(22)-N(6)-C(21)-N(8)	-3.6(7)
C(6)-C(7)-C(8)-C(9)	-0.5(8)	C(22)-N(6)-C(21)-C(18)	179.9(4)
C(6)-C(7)-C(8)-C(11)	178.4(5)	C(17)-C(18)-C(21)-N(8)	-157.5(5)
C(7)-C(8)-C(9)-C(10)	-0.5(8)	C(19)-C(18)-C(21)-N(8)	17.5(7)
C(11)-C(8)-C(9)-C(10)	-179.5(5)	C(17)-C(18)-C(21)-N(6)	19.4(6)
C(8)-C(9)-C(10)-C(5)	0.6(8)	C(19)-C(18)-C(21)-N(6)	-165.6(4)
C(6)-C(5)-C(10)-C(9)	0.3(7)	C(23)-N(7)-C(22)-N(9)	-179.2(4)
C(4)-C(5)-C(10)-C(9)	174.3(5)	C(23)-N(7)-C(22)-N(6)	0.0(7)
C(12)-N(1)-C(11)-N(3)	0.1(9)	C(21)-N(6)-C(22)-N(7)	2.9(6)
C(12)-N(1)-C(11)-C(8)	-180.0(5)	C(21)-N(6)-C(22)-N(9)	-177.9(4)
C(13)-N(3)-C(11)-N(1)	2.2(8)	C(22)-N(7)-C(23)-N(10)	-178.3(5)
C(13)-N(3)-C(11)-C(8)	-177.7(4)	C(22)-N(7)-C(23)-N(8)	-2.7(7)
C(9)-C(8)-C(11)-N(1)	-162.4(5)	C(21)-N(8)-C(23)-N(10)	177.8(5)
C(7)-C(8)-C(11)-N(1)	18.7(8)	C(21)-N(8)-C(23)-N(7)	2.1(7)
C(9)-C(8)-C(11)-N(3)	17.5(7)	C(2)-O(1)-C(30)-O(2)	-52.0(5)
C(7)-C(8)-C(11)-N(3)	-161.4(5)	C(2)-O(1)-C(30)-C(4)	70.5(4)
C(13)-N(2)-C(12)-N(5)	-177.3(6)	C(2)-O(1)-C(30)-C(14)	-163.4(3)
C(13)-N(2)-C(12)-N(1)	0.9(10)	C(3)-O(2)-C(30)-O(1)	44.9(6)
C(11)-N(1)-C(12)-N(5)	176.4(6)	C(3)-O(2)-C(30)-C(4)	-79.6(6)
C(11)-N(1)-C(12)-N(2)	-1.8(10)	C(3)-O(2)-C(30)-C(14)	157.0(5)
C(12)-N(2)-C(13)-N(4)	-178.6(6)	C(5)-C(4)-C(30)-O(1)	70.6(5)
C(12)-N(2)-C(13)-N(3)	1.7(9)	C(5)-C(4)-C(30)-O(2)	-167.8(3)
C(11)-N(3)-C(13)-N(2)	-3.1(8)	C(5)-C(4)-C(30)-C(14)	-50.5(5)
C(11)-N(3)-C(13)-N(4)	177.2(5)	C(15)-C(14)-C(30)-O(1)	-175.6(4)
C(30)-C(14)-C(15)-C(20)	-65.1(6)	C(15)-C(14)-C(30)-O(2)	70.2(5)
C(30)-C(14)-C(15)-C(16)	118.4(5)	C(15)-C(14)-C(30)-C(4)	-50.3(5)

Symmetry transformations used to generate equivalent atoms:

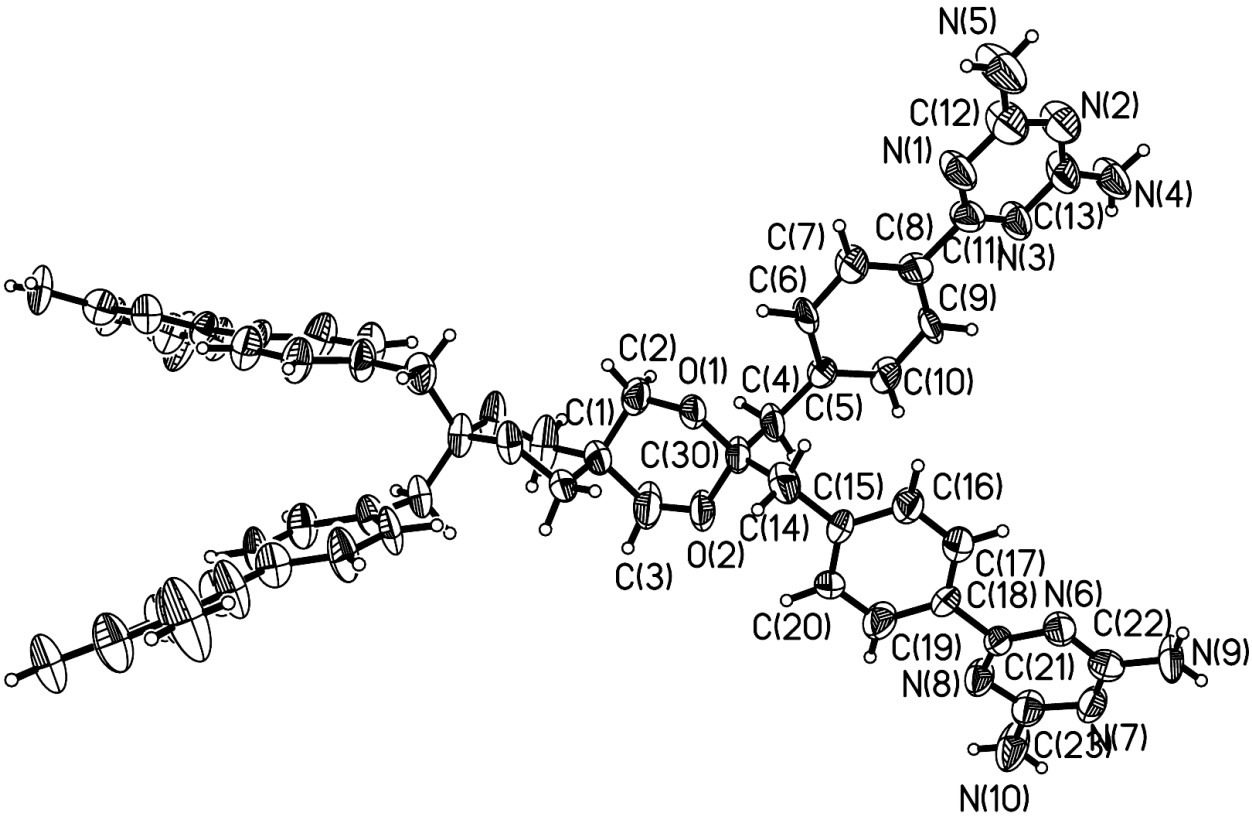
#1 -x+1,y,-z+5/2

Table 7. Hydrogen bond length (Å) and angle [°] for C83 H132 N32 O16

D-H	d(D-H)	d(H..A)	<DHA	d(D..A)	A
N4-H4B	0.86	2.215	172.85	3.070(6)	N6#1
N5-H5A	0.86	2.222	174.89	3.079(6)	N7 []
N9-H9B	0.860	2.147	170.75	2.999(6)	N3#1
N10-H10A	0.860	2.137	169.58	2.987(6)	N2#3

Symmetry transformations used to generate equivalent atoms:

#1 : -x+1, y, -z+3/2 #2 : x+1/2, y-1/2, -z+3/2
#3 : x-1/2, y+1/2, -z+3/2



ORTEP view of the C83 H132 N32 O16 compound with the numbering scheme adopted. Ellipsoids drawn at 30% probability level. Hydrogens represented by sphere of arbitrary size.

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