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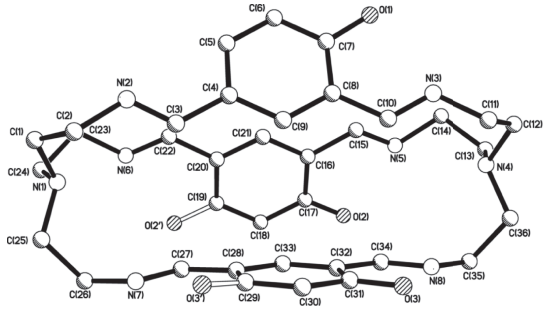
The crystal structure of (4E,11E,31E,38E)-1,4,12,15,18,26,31,39-Octaaza-7,21,24-trihydroxy-pentacyclo[13·13·13·$1^{6,10}$·$1^{20,24}$·$1^{33,37}$]tetratetraconta-4,6(44),7,9,11,18,20(43),21,23,25,31,33(42),34,36,38-pentadecaene, $C_{36}H_{42}N_8O_3$



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Abstract

$C_{36}H_{42}N_8O_3$, trigonal, *R*-3 (no. 148), $a = 30.732(2)$ Å, $c = 18.632(2)$ Å, $V = 15239(2)$ Å^3, $Z = 18$, $R_{gt}(F) = 0.0458$, $wR_{ref}(F^2) = 0.1124$, $T = 293$ K.

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Table 1: Data collection and handling.

Crystal:	Orange, plate, size 0.04×0.1×0.12 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.82 cm^{-1}
Diffractometer, scan mode:	CCD area detector, ϕ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	25929, 5980
$N(param)_{refined}$:	444
Programs:	OLEX2 [7], SHELX [8]

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	18*f*	0.0834	0.1076	0.8201	0.165
H(2)[a]	18*f*	0.1931	0.0392	0.5660	0.093
H(2′)[b]	18*f*	0.2050	−0.0834	0.7672	0.12(2)
H(3)[b]	18*f*	0.2941	0.2253	0.7050	0.096
H(3′)[a]	18*f*	0.3679	0.1782	0.9201	0.102
H(1A)	18*f*	0.2714	−0.0278	1.0834	0.119
H(1B)	18*f*	0.2157	−0.0488	1.0611	0.119
H(2A)	18*f*	0.2572	0.0359	1.1099	0.120
H(2B)	18*f*	0.2860	0.0520	1.0372	0.120
H(3A)	18*f*	0.2552	0.0796	0.9614	0.098
H(5)	18*f*	0.1227	0.0051	0.9867	0.103
H(6)	18*f*	0.0610	0.0146	0.9311	0.105
H(9)	18*f*	0.2282	0.1246	0.8795	0.092
H(10)	18*f*	0.2060	0.1735	0.7994	0.096
H(11A)	18*f*	0.1921	0.2234	0.7318	0.109
H(11B)	18*f*	0.1490	0.2304	0.7627	0.109
H(12A)	18*f*	0.0949	0.1707	0.6758	0.109
H(12B)	18*f*	0.1351	0.2214	0.6426	0.109
H(13A)	18*f*	0.1255	0.1226	0.5357	0.107
H(13B)	18*f*	0.0881	0.1395	0.5609	0.107
H(14A)	18*f*	0.0708	0.0842	0.6617	0.113
H(14B)	18*f*	0.0496	0.0563	0.5893	0.113
H(15)	18*f*	0.0982	0.0438	0.7231	0.095
H(18)	18*f*	0.2079	−0.0287	0.6137	0.095
H(21)	18*f*	0.1262	−0.0012	0.8017	0.087
H(22)	18*f*	0.1497	−0.0476	0.8853	0.099
H(23A)	18*f*	0.1630	−0.1380	0.9308	0.123
H(23B)	18*f*	0.1659	−0.0900	0.9632	0.123
H(24A)	18*f*	0.2330	−0.1038	1.0000	0.121
H(24B)	18*f*	0.2530	−0.0906	0.9219	0.121
H(25A)	18*f*	0.3260	−0.0331	0.9825	0.116
H(25B)	18*f*	0.3324	0.0195	0.9986	0.116
H(26A)	18*f*	0.3648	0.0118	0.8828	0.116
H(26B)	18*f*	0.3086	−0.0181	0.8596	0.116
H(27)	18*f*	0.2852	0.0262	0.8020	0.092
H(30)	18*f*	0.3524	0.2189	0.8490	0.093
H(33)	18*f*	0.2610	0.0722	0.7204	0.081
H(34)	18*f*	0.2356	0.1149	0.6321	0.084
H(35A)	18*f*	0.2100	0.1479	0.5508	0.095
H(35B)	18*f*	0.2524	0.2018	0.5307	0.095
H(36A)	18*f*	0.2108	0.2324	0.6073	0.097
H(36B)	18*f*	0.1820	0.2081	0.5367	0.097

[a]Occupancy factor: 0.33333; [b]Occupancy factor: 0.66667.

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	18*f*	0.26186(9)	−0.03164(9)	0.9775(1)	0.087(2)	0.091(2)	0.072(1)	0.044(1)	0.012(1)	0.013(1)
N(2)	18*f*	0.21299(9)	0.02853(9)	1.0262(1)	0.088(2)	0.098(2)	0.086(2)	0.036(2)	0.019(1)	0.007(1)
N(3)	18*f*	0.14003(9)	0.16327(9)	0.7849(1)	0.072(2)	0.086(2)	0.089(2)	0.035(1)	0.013(1)	−0.007(1)
N(4)	18*f*	0.14914(7)	0.16594(8)	0.6230(1)	0.056(1)	0.077(1)	0.086(1)	0.032(1)	0.003(1)	0.001(1)
N(5)	18*f*	0.10959(8)	0.05302(8)	0.6228(1)	0.071(2)	0.080(2)	0.104(2)	0.033(1)	−0.002(1)	−0.001(1)
N(6)	18*f*	0.18394(9)	−0.08283(9)	0.8590(1)	0.097(2)	0.086(2)	0.086(2)	0.040(2)	−0.000(1)	0.001(1)
N(7)	18*f*	0.33275(8)	0.05433(8)	0.8773(1)	0.078(2)	0.100(2)	0.080(2)	0.044(1)	0.008(1)	0.018(1)
N(8)	18*f*	0.25542(7)	0.18421(8)	0.6334(1)	0.057(1)	0.084(2)	0.082(1)	0.034(1)	0.005(1)	0.009(1)
O(1)	18*f*	0.07110(7)	0.08300(7)	0.8466(1)	0.070(1)	0.113(2)	0.134(2)	0.036(1)	0.019(1)	−0.001(1)
O(2)[a]	18*f*	0.1645(2)	0.0197(2)	0.5799(2)	0.080(4)	0.101(4)	0.044(3)	0.040(3)	0.005(2)	−0.001(3)
O(2′)[b]	18*f*	0.2120(1)	−0.0704(1)	0.7274(2)	0.103(2)	0.124(2)	0.093(2)	0.080(2)	0.007(2)	−0.002(2)
O(3)[b]	18*f*	0.31183(9)	0.23069(9)	0.7404(1)	0.091(2)	0.055(2)	0.097(2)	0.038(1)	−0.019(1)	−0.006(1)
O(3′)[a]	18*f*	0.3539(2)	0.1491(2)	0.9058(3)	0.068(3)	0.100(4)	0.080(3)	0.036(3)	−0.013(3)	−0.006(3)
C(1)	18*f*	0.2497(1)	−0.0238(1)	1.0504(1)	0.099(2)	0.107(3)	0.081(2)	0.045(2)	0.008(2)	0.017(2)
C(2)	18*f*	0.2555(1)	0.0275(1)	1.0599(1)	0.097(2)	0.115(3)	0.077(2)	0.045(2)	0.006(2)	0.001(2)
C(3)	18*f*	0.2221(1)	0.0593(1)	0.9760(2)	0.070(2)	0.078(2)	0.080(2)	0.024(2)	0.014(2)	−0.013(2)
C(4)	18*f*	0.1826(1)	0.0645(1)	0.9397(1)	0.067(2)	0.069(2)	0.071(2)	0.019(2)	0.018(1)	−0.013(1)
C(5)	18*f*	0.1317(1)	0.0313(1)	0.9545(1)	0.078(2)	0.077(2)	0.086(2)	0.027(2)	0.025(2)	−0.013(1)
C(6)	18*f*	0.0947(1)	0.0372(1)	0.9218(2)	0.065(2)	0.072(2)	0.099(2)	0.014(2)	0.031(2)	−0.009(2)
C(7)	18*f*	0.1077(1)	0.0770(1)	0.8750(2)	0.059(2)	0.089(2)	0.087(2)	0.028(2)	0.014(2)	−0.019(2)
C(8)	18*f*	0.1585(1)	0.1107(1)	0.8586(1)	0.057(2)	0.066(2)	0.078(2)	0.018(1)	0.019(1)	−0.012(1)
C(9)	18*f*	0.1946(1)	0.1031(1)	0.8909(1)	0.059(2)	0.074(2)	0.078(2)	0.020(1)	0.015(1)	−0.014(2)
C(10)	18*f*	0.1723(1)	0.1530(1)	0.8112(1)	0.064(2)	0.076(2)	0.087(2)	0.025(2)	0.014(2)	−0.017(2)
C(11)	18*f*	0.1565(1)	0.2070(1)	0.7394(2)	0.080(2)	0.083(2)	0.111(2)	0.041(2)	0.014(2)	−0.007(2)
C(12)	18*f*	0.1303(1)	0.1921(1)	0.6677(2)	0.073(2)	0.091(2)	0.117(2)	0.047(2)	0.010(2)	0.004(2)
C(13)	18*f*	0.1101(1)	0.1277(1)	0.5770(1)	0.073(2)	0.089(2)	0.103(2)	0.039(2)	−0.014(2)	0.004(2)
C(14)	18*f*	0.07991(9)	0.0779(1)	0.6151(2)	0.057(2)	0.086(2)	0.130(2)	0.029(2)	−0.013(2)	−0.006(2)
C(15)	18*f*	0.11357(9)	0.0379(1)	0.6843(2)	0.059(2)	0.069(2)	0.097(2)	0.023(1)	0.000(2)	−0.016(2)
C(16)	18*f*	0.14151(9)	0.01158(9)	0.6964(1)	0.056(2)	0.062(2)	0.076(2)	0.022(1)	−0.001(1)	−0.012(1)
C(17)	18*f*	0.16637(9)	0.0030(1)	0.6394(2)	0.063(2)	0.074(2)	0.071(2)	0.025(2)	0.000(1)	−0.015(1)
C(18)	18*f*	0.19097(9)	−0.0237(1)	0.6513(1)	0.076(2)	0.090(2)	0.071(2)	0.040(2)	0.006(1)	−0.016(2)
C(19)	18*f*	0.1906(1)	−0.0432(1)	0.7186(2)	0.069(2)	0.079(2)	0.080(2)	0.036(2)	−0.004(1)	−0.017(2)
C(20)	18*f*	0.16601(9)	−0.03483(9)	0.7763(1)	0.066(2)	0.067(2)	0.069(2)	0.026(1)	0.001(1)	−0.011(1)
C(21)	18*f*	0.14214(9)	−0.00721(9)	0.7638(1)	0.066(2)	0.067(2)	0.076(2)	0.026(1)	0.004(1)	−0.017(1)
C(22)	18*f*	0.1648(1)	−0.0550(1)	0.8476(1)	0.078(2)	0.073(2)	0.082(2)	0.026(2)	0.006(2)	−0.006(2)
C(23)	18*f*	0.1822(1)	−0.1020(1)	0.9314(2)	0.106(3)	0.087(2)	0.096(2)	0.035(2)	0.005(2)	0.015(2)
C(24)	18*f*	0.2348(1)	−0.0845(1)	0.9586(2)	0.123(3)	0.096(2)	0.093(2)	0.062(2)	0.002(2)	0.018(2)
C(25)	18*f*	0.3158(1)	−0.0095(1)	0.9681(1)	0.092(2)	0.117(2)	0.094(2)	0.061(2)	0.012(2)	0.032(2)
C(26)	18*f*	0.3321(1)	0.0075(1)	0.8914(1)	0.095(2)	0.116(2)	0.102(2)	0.070(2)	0.022(2)	0.033(2)
C(27)	18*f*	0.30571(9)	0.0557(1)	0.8271(1)	0.062(2)	0.082(2)	0.082(2)	0.034(2)	0.013(1)	0.008(2)
C(28)	18*f*	0.30531(8)	0.10129(9)	0.8068(1)	0.050(1)	0.069(2)	0.068(2)	0.025(1)	0.005(1)	0.003(1)
C(29)	18*f*	0.33257(9)	0.1462(1)	0.8458(1)	0.055(2)	0.083(2)	0.069(2)	0.028(2)	0.003(1)	−0.006(2)
C(30)	18*f*	0.33410(9)	0.1895(1)	0.8228(1)	0.067(2)	0.072(2)	0.084(2)	0.026(2)	−0.005(1)	−0.012(2)
C(31)	18*f*	0.30877(9)	0.1902(1)	0.7611(1)	0.056(2)	0.072(2)	0.080(2)	0.027(1)	0.002(1)	−0.002(2)
C(32)	18*f*	0.28067(8)	0.14505(9)	0.7222(1)	0.047(1)	0.071(2)	0.066(2)	0.024(1)	0.003(1)	0.001(1)
C(33)	18*f*	0.27961(8)	0.10182(9)	0.7461(1)	0.052(2)	0.068(2)	0.073(2)	0.023(1)	0.004(1)	−0.004(1)
C(34)	18*f*	0.25414(8)	0.1449(1)	0.6571(1)	0.050(2)	0.078(2)	0.074(2)	0.026(1)	0.007(1)	−0.001(1)
C(35)	18*f*	0.22873(9)	0.1821(1)	0.5673(1)	0.062(2)	0.101(2)	0.073(2)	0.039(2)	0.005(1)	0.013(1)
C(36)	18*f*	0.19308(9)	0.2014(1)	0.5813(1)	0.065(2)	0.084(2)	0.090(2)	0.035(2)	0.002(1)	0.011(2)

[a]Occupancy factor: 0.33333; [b]Occupancy factor: 0.66667.

The crystal structure is shown in the figure, Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A solution of tris(2-aminoethyl)amine (0.29 g, 2.0 mmol) in 50 mL acetonitrile was slowly added to the solution of 4-hydroxybenzene-1,3-dicarboxaldehyde (0.45 g, 3.0 mmol)

in 100 mL acetonitrile with stirring. The resultant mixture was stirred at room temperature for another 10 h. During the reaction, an orange solid precipitated. The precipitate was collected by filtration and washed with acetonitrile. Recrystallization from dichloromethane and acetonitrile afforded 0.43 g product (yield: 68%). Crystals suitable for X-ray diffraction were obtained by slowly evaporation of the title compound in dichloromethane and acetonitrile at room temperature.

Experimental details

Hydrogen atoms were positioned geometrically with d(C—H) ranging from 0.93 Å to 0.96 Å and d(O—H) = 0.82 Å.

Discussion

Cryptands are a class of macrocyclic compounds with unique three dimensional structures [1, 2]. These compounds can act as receptors for anions [3, 4], cations [5] or neutral molecules [6]. A Schiff-base cryptand was synthesized and its crystal structure was determined. The lone pair of both pivotal nitrogen atoms (N1 and N4) is directed inward the cryptand in an *endo-endo* arrangement. The distance between the two nitrogen atoms is 10.65 Å. The crystal of this compound presents a disorder at least at two hydroxy groups (O2/O2′ and O3/O3′; see the figure). The sites occupancy factors are 0.33333 and 0.66667.

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