Crystal structure of 3,7,11,18,22,26-hexaazatricyclo[26.2.2.2^{13,16}] tetratriaconta-1(31),13(34),14,16(33),28(32),29-hexaenedicobalt(II) tetrachloride — chloroform — water (1:2:1), $[Co_2(C_{28}H_{46}N_6)Cl_4] \cdot 2CHCl_3 \cdot H_2O$

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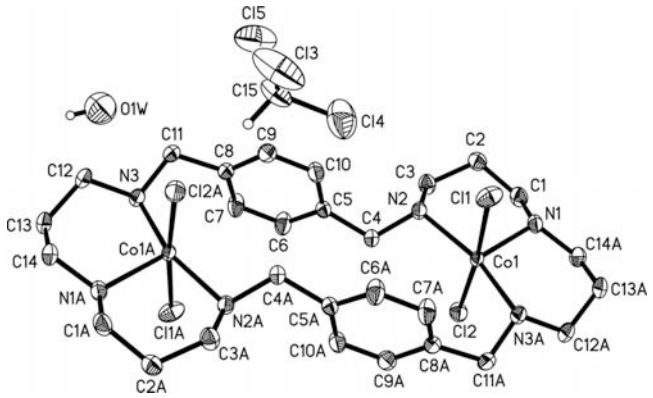

Abstract

$C_{30}H_{50}Cl_{10}Co_2N_6O$, triclinic, $P\bar{1}$ (no. 2), a = 9.310(2) Å, b = 11.085(2) Å, c = 12.000(2) Å, α = 70.23(3)°, β = 83.07(3)°, γ = 65.71(3)°, V = 1062.0 Å^3, Z = 1, $R_{gt}(F)$ = 0.050, $wR_{ref}(F^2)$ = 0.128, T = 293 K.

Source of material

An chloroform solution of 3,7,11,18,22,26-hexaazatricyclo-[26.2.2.2^{13,16}]tetratriaconta-1(31),13(34),14,16(33),28(32),29-hexaene (L, 0.1 mmol) was added to a solution of cobalt chloride (0.2 mmol) in methanol (5 ml). Then a purple solution was obtained. The crystals were obtained by evaporating the solution at room temperature for several days.

Experimental details

All hydrogen atoms on carbon atoms were generated geometrically and refined as riding with d(C—H) = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C). The imido group H atom and water H atoms were located from difference Fourier map and and were refined freely.

Discussion

In the past decade, much attention has been focused on the design and synthesis of macrocyclic polyamines, which can particippate in molecular recognition phenomena with different kinds of substrates, such as organic and inorganic cations, anions and neutral molecules [1,2]. Moreover, hexaaza macrocycles can form dinuclear metal complexes which in turn are capable of coordinating anions [3].

The crystal structure of the title compound contains discrete molecules. The cobalt cation is five-coordinated by two chlorine atoms and three N atoms from ligand in a tetragonal pyramidal coordination. Co1, N2, N3, Cl1 and Cl2 atoms are almost coplanar and the bond angle sum around cobalt is 358.5°. The Co1—Cl1 and Co1—Cl2 distances are 2.489(1) Å and 2.351(1) Å, respectively. The mean Co—N distance is 2.053 Å. The chloroform and water molecule do not coordinate the cobalt center.

Table 1. Data collection and handling.

Crystal:	purple block, size 0.25 × 0.3 × 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	14.43 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, ω
$2\theta_{max}$:	54.88°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10516, 4808
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3843
$N(param)_{refined}$:	239
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	2*i*		0.6959	–0.0375	0.9578	0.056
H(1B)	2*i*		0.7195	–0.1786	0.9411	0.056
H(2A)	2*i*		0.5493	–0.0381	0.7665	0.057
H(2B)	2*i*		0.4668	–0.0466	0.8888	0.057
H(3A)	2*i*		0.3566	0.1813	0.7795	0.053
H(3B)	2*i*		0.4716	0.1753	0.8685	0.053
H(4A)	2*i*		0.5730	0.3909	0.6264	0.054
H(4B)	2*i*		0.4868	0.3810	0.7470	0.054
H(6)	2*i*		0.2614	0.5842	0.7033	0.065
H(7)	2*i*		0.0480	0.7508	0.5846	0.062
H(9)	2*i*		0.1453	0.5196	0.3720	0.053
H(10)	2*i*		0.3564	0.3519	0.4925	0.051
H(11A)	2*i*		–0.0562	0.7437	0.3095	0.051
H(11B)	2*i*		–0.1516	0.7907	0.4151	0.051
H(12A)	2*i*		–0.2470	1.0253	0.2709	0.051
H(12B)	2*i*		–0.1340	0.9733	0.1737	0.051
H(13A)	2*i*		–0.2171	1.2179	0.1455	0.056
H(13B)	2*i*		–0.1160	1.1756	0.2580	0.056
H(14A)	2*i*		0.0217	1.1053	0.0553	0.054
H(14B)	2*i*		0.0045	1.2440	0.0735	0.054

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Table 2. Continued.

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(15)	2*i*		0.2762	0.5529	0.1168	0.100
H(1N)	2*i*		0.827(4)	–0.116(4)	0.767(4)	0.05(1)
H(2N)	2*i*		0.529(4)	0.191(4)	0.638(3)	0.04(1)
H(3N)	2*i*		–0.037(5)	0.937(4)	0.393(4)	0.05(1)
HW(1A)	2*i*	0.50	–0.535(3)	0.95(2)	0.48(2)	0.18(9)
HW(1B)	2*i*	0.50	–0.41(2)	0.97(2)	0.51(2)	0.17(9)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Co(1)	2*i*	0.79423(4)	0.12366(4)	0.71121(3)	0.0251(2)	0.0241(2)	0.0269(2)	–0.0063(2)	–0.0040(1)	–0.0063(2)
C(1)	2*i*	0.6989(4)	–0.0811(4)	0.8994(3)	0.051(2)	0.034(2)	0.045(2)	–0.018(2)	0.002(2)	0.000(2)
C(2)	2*i*	0.5388(4)	–0.0113(4)	0.8370(4)	0.046(2)	0.039(2)	0.054(2)	–0.021(2)	0.002(2)	–0.007(2)
C(3)	2*i*	0.4666(4)	0.1470(4)	0.8015(3)	0.042(2)	0.037(2)	0.046(2)	–0.013(2)	0.006(2)	–0.008(2)
C(4)	2*i*	0.4934(4)	0.3618(3)	0.6730(4)	0.038(2)	0.028(2)	0.061(2)	–0.009(1)	–0.014(2)	–0.004(2)
C(5)	2*i*	0.3374(4)	0.4530(3)	0.6079(3)	0.035(2)	0.028(2)	0.040(2)	–0.012(1)	–0.003(1)	–0.004(1)
C(6)	2*i*	0.2403(5)	0.5715(4)	0.6351(4)	0.059(2)	0.044(2)	0.047(2)	–0.001(2)	–0.017(2)	–0.018(2)
C(7)	2*i*	0.1123(5)	0.6722(4)	0.5635(3)	0.052(2)	0.042(2)	0.047(2)	0.003(2)	–0.009(2)	–0.020(2)
C(8)	2*i*	0.0785(4)	0.6579(3)	0.4616(3)	0.031(2)	0.032(2)	0.043(2)	–0.013(1)	–0.004(1)	–0.006(1)
C(9)	2*i*	0.1699(4)	0.5346(4)	0.4376(3)	0.048(2)	0.040(2)	0.043(2)	–0.017(2)	–0.009(2)	–0.010(2)
C(10)	2*i*	0.2970(4)	0.4340(4)	0.5100(3)	0.046(2)	0.030(2)	0.045(2)	–0.007(2)	–0.005(2)	–0.011(2)
C(11)	2*i*	–0.0512(4)	0.7732(4)	0.3758(3)	0.032(2)	0.039(2)	0.053(2)	–0.015(1)	–0.009(2)	–0.006(2)
C(12)	2*i*	–0.1425(4)	1.0094(4)	0.2385(3)	0.032(2)	0.040(2)	0.047(2)	–0.006(1)	–0.013(1)	–0.008(2)
C(13)	2*i*	–0.1229(4)	1.1477(4)	0.1911(3)	0.037(2)	0.032(2)	0.055(2)	–0.002(1)	–0.013(2)	–0.007(2)
C(14)	2*i*	0.0179(4)	1.1481(4)	0.1147(3)	0.046(2)	0.033(2)	0.044(2)	–0.011(2)	–0.014(2)	0.002(2)
C(15)	2*i*	0.2857(8)	0.4600(6)	0.1211(5)	0.118(5)	0.059(3)	0.094(4)	–0.046(3)	0.014(3)	–0.040(3)
N(1)	2*i*	0.8299(3)	–0.0731(3)	0.8183(3)	0.039(2)	0.028(1)	0.038(1)	–0.008(1)	–0.004(1)	–0.008(1)
N(2)	2*i*	0.5498(3)	0.2097(3)	0.7008(2)	0.031(1)	0.030(1)	0.036(1)	–0.009(1)	–0.001(1)	–0.008(1)
N(3)	2*i*	–0.0233(3)	0.9040(3)	0.3315(3)	0.031(1)	0.033(1)	0.037(1)	–0.008(1)	–0.007(1)	–0.008(1)
Cl(1)	2*i*	0.7763(1)	0.0508(1)	0.54047(8)	0.0472(5)	0.0888(8)	0.0456(5)	–0.0303(5)	0.0060(4)	–0.0364(5)
Cl(2)	2*i*	0.7937(1)	0.21363(9)	0.86286(8)	0.0534(5)	0.0415(5)	0.0480(5)	–0.0103(4)	–0.0116(4)	–0.0221(4)
Cl(3)	2*i*	0.3416(4)	0.4380(2)	–0.0160(2)	0.291(3)	0.138(2)	0.095(1)	–0.148(2)	0.078(2)	–0.063(1)
Cl(4)	2*i*	0.4257(3)	0.3355(2)	0.2310(2)	0.129(2)	0.087(1)	0.121(1)	–0.010(1)	0.002(1)	–0.047(1)
Cl(5)	2*i*	0.1035(3)	0.4503(3)	0.1571(2)	0.131(2)	0.154(2)	0.151(2)	–0.086(2)	0.024(1)	–0.088(2)
O(1W)	2*i*	–0.435(1)	0.923(2)	0.470(2)	0.069(6)	0.17(1)	0.28(2)	–0.050(7)	0.019(8)	–0.15(1)

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References

1. Anda, C.; Llobet, A.; Reibenspies, S. J.; Motekaitis, R. J.; Martell, A. E.: A Systematic Evaluation of Molecular Recognition Phenomena. 1. Interaction between Phosphates and Nucleotides with Hexaazamacrocyclic Ligands Containing *m*-Xylylic Spacers. Inorg. Chem. **39** (2000) 2986-2999.
2. Anda, C.; Llobet, A.; Martell, S. E; Motekaitis, R. J.: A Systematic Evaluation of Molecular Recognition Phenomena. 2. Interaction between Phosphates and Nucleotides with Hexaazamacrocyclic Ligands Containing Diethylic Ether Spacers. Inorg. Chem. **39** (2000) 3000-3008.
3. Costas, M.; Anda, C.; Llobet, A.; Parella, T.; Evans, H. S.; Pinilla, E.: Synthesis, structure and hydrolytic properties of a family of new Zn complexes containing hexaazamacrocyclic ligands. Eur. J. Inorg. Chem. (2004) 857-865.
4. Sheldrick, G. M.: SHELXS-97. Program for the Solution of Crystal Structures. University of Göttingen, Germany 1997.
5. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.
6. Sheldrick, G. M.: SHELXTL. Structure Determination Software Suite. Version 6.14. Bruker AXS, Madison, Wisconsin, USA 2000.