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تهیه و ساختار بلوری یک کمپلکس شش کوردینهٔ جدید از گالیم (III)

حسین آقا بزرگ[۱]، علی اکبری ساعی[۱]، ابوالقاسم مقیمی[۲]، فرشید رمضانی پور[۱]

۱- دانشکده شیمی، دانشگاه تربیت معلم تهران، ایران.
۲- دانشگاه امام حسین(ع) ، تهران، ایران.
پست الکترونیکی: haghabozorg@yahoo.com

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چکیده: از واکنش گالیم(III) نیترات هشت آبه با سیستم انتقال پروتون $(pydaH_2)^{2+}(pydc)^{2-}$ (pyda = پیریدین دی آمین، $pydcH_2$ = پیریدین دی کربوکسیلیک اسید) کمپلکس $[C_5H_8N_3][Ga(C_7H_3NO_4)_2] \cdot 4H_2O \cdot CH_3OH$ به دست می‌آید که دارای ساختار بلوری سه میل، با گروه فضایی $P\bar{1}$ و دو مولکول در یاختهٔ یکه است. ابعاد و زاویه‌های یاخته یکه عبارتند از:

$a=10/2379(17)$ Å $b=10/4283(17)$ Å $c=13/838(2)$ Å
$\alpha=70/375(3)^\circ$ $\beta=77/938(3)^\circ$ $\gamma=64/120(3)^\circ$

پس از تعیین ساختار مولکولی، مقدار R تا میزان ۰/۰۵۷۹ برای ۴۷۰۳ بازتاب کاهش یافت.

واژه‌های کلیدی: *سیستم خود مجموعه‌ساز، ساختار بلوری، پیوندهای هیدروژنی، کمپلکس گالیم(III).*

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Synthesis and Crystal Structure of a Novel Six-Coordinate Gallium (III) Complex

Hossein Aghabozorg[1], Ali Akbari Saei[1], Abolghasem Moghimi[2], Farshid Ramezanipour[1]

1- Department of Chemistry,Teacher Training University,49 Mofateh Avenue, Tehran-IRAN.
2- Department of Chemistry, University of Imam Hossein, Tehran-IRAN.
E-mail: haghabozorg@yahoo.com

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Abstract: The reaction of gallium (III) nitrate octahydrate with the proton transfer compound $(pydaH_2)^{2+}$ $(pydc)^{2-}$ (where pyda is 2,6-pyridinediamine and $pydcH_2$ is 2,6-pyridinedicarboxylic acid) leads to the formation of $(C_5H_8N_3)$ $[Ga(C_7H_3NO_4)_2]$ · $4H_2O$ · CH_3OH. The crystal system of the anionic complex is triclinic with space group $P\bar{1}$ and two molecules per unit cell. The unit cell parameters are a=10.238(2) Å, b=10.428(2) Å, c=13.838(2) Å, α=70.375(3)°, β=77.938(3)°, γ=64.120(3)°. The structure has been refined to a final value for the crystallographic R factor of 0.0579 based on 4703 reflections with $I > 2\sigma(I)$.

Keywords: *Self assembling systems, Crystal structure, Hydrogen bonds, Gallium(III) complex.*

Introduction

Preparation and characterization of self assembling compounds has been of interest in the recent years [1, 2]. These self assembling systems are formed through noncovalent interactions. The intermolecular forces in such systems may involve hydrogen bonding, ion pairing, π-π stacking, hydrophobic or hydrophilic, donor-acceptor and host-guest interactions. Often one or a combination of these interactions can result in the formation of specific self assembling systems.

In this regard the proton transfer compound LH_2 (LH_2 is $(pydaH_2)^{2+}$ $(pydc)^{2-}$ where pyda =2,6-pyridinediamine and $pydcH_2$ = 2,6-pyridine-dicarboxylic acid) was prepared by our research group previously[3]. Several complexes were synthesized using this proton transfer system and their X-ray crystal structures were reported. Some of these complexes possess $(pydc)^{2-}$ ligand and also $(pydaH)^{+}$ fragment, where pyda in its protonated form acts as a counter ion of the anionic complex[4-10]. There are also some other complexes of LH_2 in which no counter ion is observed in the structure [7, 11-14]. Here, we report the synthesis and X-ray crystal structure of Ga(III) complex with the above proton transfer system.

Experimental

An aqueous solution of gallium(III) nitrate octahydrate (0.890 g, 0.225 mmol in 20 mL of water) was added to a solution of LH_2 (0.125 g, 0.450 mmol) in water (10 mL). The consequent solution was left at room temperature for 24 hours. The resulting yellow crystals were solved in methanol and suitable crystals for X-ray analysis were obtained after 12 hours.

Results and discussion

The reaction of LH_2 ligand with gallium (III) nitrate octahydrate in a 2:1 molar ratio leads to the formation of the title complex. There is a list of crystal data and structure refinement in the table 1. The molecular structure of the compound exhibits a six coordinate anionic complex accompanied by a counter ion. There is also one uncoordinate methanol molecule in the crystal structure as well as four water molecules. Figure 1 illustrates the molecular structure of the title complex. The central ion is coordinated by two $(pydc)^{2-}$ ligands *via* two O atoms and one N atom from each ligand. The geometry of the complex is distorted octahedral. The two $(pydc)^{2-}$ groups are approximately perpendicular to each other as specified by some of the torsion angles and bond angles. The torsion angles O(8)—Ga(1)—N(1)—C(2) and O(8)—Ga(1)—N(1)—C(6) are -90.7(2)° and 91.4(2)° respectively. Also the bond angles O(4)—Ga(1)—O(6) and O(8)-Ga(1)-O(4) are 91.25(9)˚ and 91.92(9)° respectively (table 2).

Atomic coordinates and equivalent isotropic displacement parameters for the complex are listed in table 3 and anisotropic displacement parameters are collected in table 4. The Ga(III) does not lie on a perfect linear axial

angle, and the N1-N2 axis shows a variation of 12.80° from linearity. An important feature of the structure is the presence of mono protonated form of 2,6-pyridinediamine in the compound. This cationic fragment acts as a counter ion and balances the charge. It is interesting to note that we started the reaction with diprotonated form of pyda, while the resulting compound contains pyda in its monoprotonated form. The protonated site of pyda in the product compound is the nitrogen atom of the ring, while in the starting material LH_2, pyda was diprotonated on the nitrogen atoms of the amine groups.

Figure 2 illustrates the crystal packing of the title compound. The self assembling system of the compound is formed using hydrogen bonding and ion pairing interactions. These two factors cause the compound to form a three-dimensional network. A wide range of hydrogen bonds from weak to strong interactions are observed in the structure. The presence of four uncoordinated water molecules causes the structure to have a complicated lattice of different hydrogen bonds. Hydrogen coordinates and isotropic displacement parameters for the title complex are listed in Table 5 and a list of hydrogen bonds is collected in Table 6.

The atoms O(4W) , C(1S) and O(1S) are disorder atoms. O(4W) and also C(1S) atom exhibit three positions, where two positions are illustrated by O(1S) atom. The distances between different positions of O(4W) atom are O(4W)—O(4W′)=1.520(12)Å and O(4W)—O(4W″)= 0.718(17)Å, and that of the C(1S) atom are C(1S′)—C(1S) = 0.774(16) Å and C(1S″)—C(1S′) = 0.53(3) Å. These distances show much more disordering about O(4W) atom.

Conclusion

The present work supports the strong ability of the LH_2 compound in forming the self assembling systems with metal ions. The extensive hydrogen bonding lattice of the metal-organic compound $(pydaH)[Ga(pydc)_2] \cdot 4H_2O \cdot CH_3OH$ is interesting to note in this regard. The presence of the cationic counter ion $(pydaH)^+$ which causes ion pairing interactions in the structure provides a view of expected uncovalent intermolecular forces for the future works.

Acknowledgments

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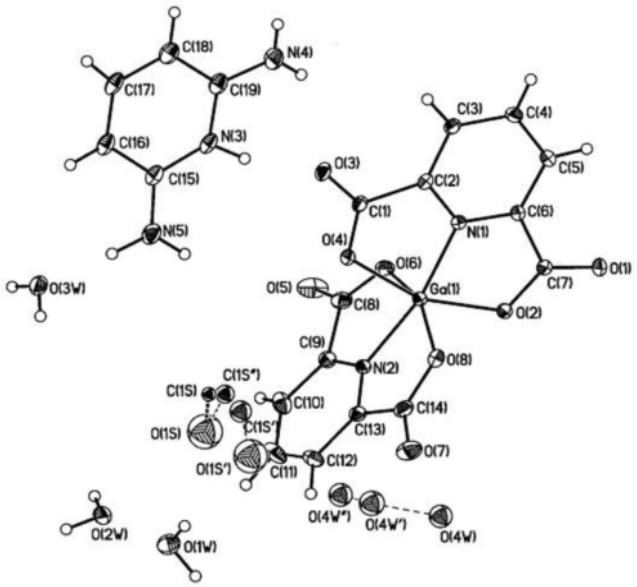

Figure 1 The molecular structure of the title compound.

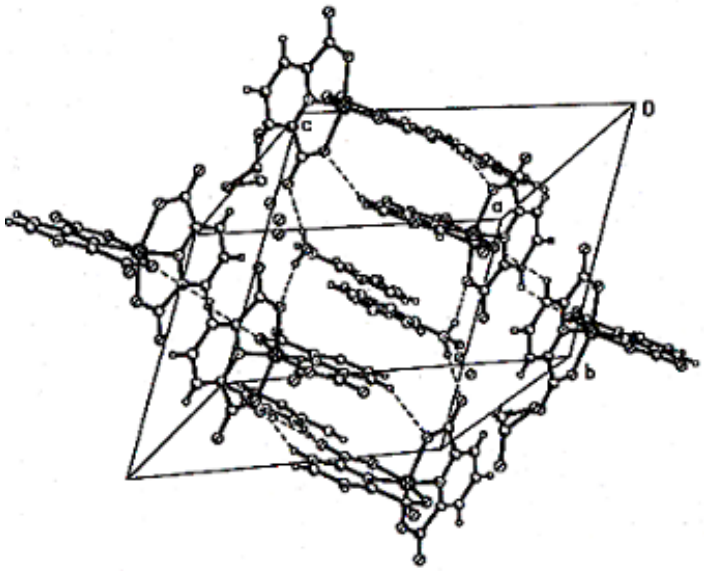

Figure 2 The crystal packing of the title compound.

Table 1 Crystal data and structure refinement of the title complex.

Empirical formula	C20 H26 Ga N5 O13
Formula weight	614.18
Temperature	120(2) K
Wavelength(λMoK_α)	0.71073 Å
Crystal system, space group	Triclinic, $P\bar{1}$
Unit cell dimensions	a = 10.238(2) Å b = 10.428(2) Å c = 13.838(2) Å α = 70.375(3)° β = 77.938(3)° γ = 64.120(3)°
Volume	1248.6(4) $Å^3$
Z, Calculated density	2, 1.634 g/cm^3
Absorption coefficient	1.180 mm^{-1}
F(000)	632
Crystal size	0.8 x 0.5 x 0.4 mm
Theta range for data collection	2.22 to 28.04 deg.
Limiting indices	$-13<h<13$, $-13<k<13$, $-18<l<18$
Reflections collected / unique	12892 / 6010 [R(int) = 0.0259]
Completeness to theta = 28.04	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.802 and 0.271
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6010 / 3 / 393
Goodness-of-fit on F^2	1.082
Final R indices [for 4703 refs with I>2sigma(I)]	$R1$ = 0.0579,$wR2$ = 0.1543
R indices (all data)	$R1$ = 0.0725, $wR2$ = 0.1660
Largest diff. peak and hole	1.919 and -0.595 $eÅ^{-3}$

Table 2 Selected geometric parameters of the title complex (Å, °).

Ga(1)-N(1)	1.958(2)	N(2)-Ga(1)-O(6)	79.15(10)
Ga(1)-N(2)	1.959(2)	N(2)-Ga(1)-O(4)	90.21(9)
Ga(1)-O(2)	1.968(2)	O(2)-Ga(1)-O(6)	94.22(10)
Ga(1)-O(8)	2.000(2)	O(2)-Ga(1)-O(4)	157.68(9)
Ga(1)-O(6)	2.005(2)	O(2)-Ga(1)-O(8)	91.10(10)
Ga(1)-O(4)	2.075(2)	O(4)-Ga(1)-O(6)	91.25(9)
N(1)-Ga(1)-N(2)	167.20(10)	O(6)-Ga(1)-O(8)	157.87(10)
N(1)-Ga(1)-O(2)	80.57(10)	O(8)-Ga(1)-O(4)	91.92 (9)
N(1)-Ga(1)-O(4)	77.22 (9)	O(6)-Ga(1)-N(1)-C(6)	-90.3(2)
N(1)-Ga(1)-O(6)	98.43(10)	O(6)-Ga(1)-N(1)-C(2)	87.5(2)
N(1)-Ga(1)-O(8)	103.63(10)	O(8)-Ga(1)-N(1)-C(2)	-90.7(2)
N(2)-Ga(1)-O(2)	112.07(10)	O(8)-Ga(1)-N(1)-C(6)	91.4(2)
N(2)-Ga(1)-O(8)	78.94(10)		

Table 3 Atomic coordinates($x10^4$) and equivalent isotropic displacemen parameters ($Å^2x10^3$). *U(eq)* is defined as one third of the trace of the orthogonalized *Uij* tensor.

	x	*y*	*z*	*U(eq)*
Ga(l)	-5296(1)	2909(1)	2491(1)	31(1)
N(1)	-5901(3)	2740(3)	3951(2)	28(1)
O(1)	-9355(3)	5398(3)	3310(2)	42(1)
C(2)	-4960(3)	1809(3)	4664(2)	27 (1)
O (4)	-3401(2)	1390(2)	3200(2)	31(1)
C(4)	-6852(4)	2469(3)	5959(2)	32(1)
N(2)	-4300(3)	2692(3)	1147(2)	29 (1)
O(2)	-7327(2)	4364(3)	2376(2)	39 (1)
C(3)	-5400(3)	1644(3)	5693(2)	30(1)
O(3)	-2485(2)	129(2)	4740(2)	35 (1)
C(5)	-7808 (4)	3433(3)	5205(2)	33(1)
O(6)	-5730(2)	1273(3)	2372(2)	35(1)
C(6)	-7279(3)	3549(3)	4183(2)	30(1)
C(7)	-8082(3)	4532(3)	3228(2)	35 (1)
O(5)	-5177(4)	–258(4)	1406(2)	66(1)
C(l)	-3483(3)	1030(3)	4187(2)	29(1)
O(7)	-3301(3)	5617(3)	805(2)	57(1)
O(8)	-4575(3)	4535(2)	2052(2)	38(1)
C(9)	-4238(3)	1594(4)	848(2)	35 (1)
C(10)	-3400(4)	1292(5)	-33(3)	45(1)
C(l1)	-2623(4)	2159(5)	-563(3)	52(1)
C(12)	-2690(4)	3290(5)	-220(2)	43 (1)
C(13)	-3559(3)	3534(4)	656(2)	33(1)
C(14)	-3801(4)	4664(4)	1188(3)	38 (1)
C (8)	-5123(4)	776 (4)	1584(3)	40(1)
C(19)	200(4)	-2470(4)	6167(3)	41(1)
C(18)	1440(4)	-3568(4)	6663(3)	47(1)
C(17)	2716(4)	-4059(4)	6085(3)	43(1)
C(16)	2838(4)	-3487(4)	5028(3)	41(1)
C(15)	1602(4)	-2391(3)	4537(3)	38(1)
N(3)	332 (3)	-1939(3)	5127(2)	39(1)
N(4)	-1113 (3)	-1928(4)	6645(3)	49(1)
N(5)	1592(3)	-1755 (3)	3527(3)	48(1)
O(lW)	824(3)	2165(3)	-1758(2)	56(1)
O(2W)	739(3)	-176(3)	-2136(2)	48(1)
O(3W)	4431(3)	-2784(3)	2620(2)	44(1)
O(4W)	-2557(9)	7763(8)	465(6)	59(2)
O(4W')	-1091(11)	6454(11)	532(7)	70(2)
O(4W")	-620(20)	5710(20)	513(13)	61(4)
O (lS)	606(13)	1694 (13)	454(7)	133 (4)
O(lS')	1182(12)	3633(12)	882(9)	128(4)
C(1S")	740 (30)	1890(30)	1378(13)	37(6)
C(lS')	604 (11)	2473(11)	1222(7)	50(2)
C(lS)	1123(14)	1642 (13)	1363(7)	20(3)

Table 4 Anisotropic displacement parameters ($Å^2$ x 10^3) for the title complex. The anisotropic displacement factor exponent takes the form:

$$-2\pi^2 [h^2 a^{*2} U11 + ... + 2 h k a^* b^* U12]$$

	*U*11	*U*22	*U*33	*U*23	*U*13	*U*12
Ga(l)	26(1)	29(1)	23 (1)	-2(1)	1(1)	-3 (1)
N(l)	29(1)	25(1)	23(1)	-4(1)	-1(1)	-7(1)
O (1)	29 (1)	38(1)	37(1)	-6(1)	2(1)	2 (1)
C(2)	28(1)	22(1)	29(1)	-5(1)	-6(1)	-7(1)
O (4)	26(1)	29(1)	27 (1)	-5(1)	-2(1)	-3(1)
C(4)	39(2)	32 (2)	24(1)	-9(1)	2(1)	-14(1)
N (2)	25(1)	31(1)	20(1)	-2(1)	-3 (1)	-5(1)
O (2)	28(1)	38(1)	26(1)	0(1)	0(1)	1(1)
C(3)	35(2)	25 (1)	26(1)	-4(1)	-5(1)	-10(1)
O (3)	32 (1)	29(1)	35 (1)	-7(1)	-12(1)	-2(1)
C(5)	31(2)	34(2)	29(2)	-10(1)	4(1)	-10(1)
O (6)	33(1)	40(1)	26(1)	-1(1)	-3(1)	-15(1)
C(6)	26(1)	28 (1)	28(1)	-6(1)	1(1)	-6(1)
C(7)	28(2)	31(2)	30(2)	-1(1)	1(1)	-4(1)
O (5)	112(3)	72 (2)	43(2)	-13(1)	-7(2)	-64(2)
C(l)	29(2)	23 (1)	30(2)	-7(1)	-5(1)	-6(1)
O (7)	63(2)	50(2)	57(2)	4(1)	-9(1)	-32(1)
O (8)	38(1)	27(1)	39(1)	-7(1)	-2(1)	-7(1)
C(9)	30(2)	40(2)	27(2)	-5(1)	-7(1)	-8(1)
C(10)	38(2)	62(2)	35(2)	-24(2)	-5(2)	-12(2)
C (11)	34(2)	93(3)	29(2)	-23(2)	3(1)	-23(2)
C(12)	30(2)	66(2)	26(2)	-4(2)	0(1)	-19(2)
C (13)	25(1)	38(2)	25(1)	2(1)	-7(1)	-6(1)
C(14)	37(2)	33(2)	36(2)	2(1)	-10(1)	-11(1)
C(8)	44(2)	46(2)	29(2)	-3(1)	-12(1)	-18(2)
C(19)	31(2)	36(2)	53(2)	-9(2)	-12(2)	-9(1)
C (18)	39(2)	44(2)	53(2)	-7(2)	-19(2)	-8(2)
C(17)	31(2)	30(2)	63(2)	-6(2)	-22(2)	-5(1)
C(16)	30(2)	29(2)	61(2)	-8(2)	-14(2)	-9(1)
C(15)	31(2)	28(2)	53(2)	-5(1)	-11 (1)	-11(1)
N(3)	31(1)	27 (1)	52(2)	-4(1)	-16(1)	-5(1)
N(4)	32(2)	53(2)	52(2)	-12(2)	-13(1)	-6(1)
N(5)	34(2)	42(2)	55(2)	-3(2)	-13(1)	-9(1)
O(lW)	58(2)	60(2)	40(1)	-6(1)	-15(1)	-15(1)
O(2W)	30(1)	52(2)	41 (1)	-4(1)	3(1)	-8(1)
O(3W)	49(2)	35(1)	42(1)	-13 (1)	-7(1)	-7(1)

Table 5 Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for the title complex.

	x	*y*	*z*	*U(eq)*
H(4C)	-7180	2370	6649	38
H(3C)	-4751	1003	6195	36
H(5C)	-8775	3986	5377	39
H(10C)	-3358	532	-263	53
H (11C)	-2049	1977	-1159	62
H(12C)	-2163	3866	-570	52
H (18C)	1395	-3958	7375	57
H(17C)	3533	-4804	6413	51
H(16C)	3725	-3827	4653	49
H(3N)	-532	-1206	4909	58
H(2N4)	-1208	-2303	7238	74
H(lN4)	-1886	-1369	6352	74
H(2N5)	2561	-2110	3172	72
H(1N5)	728	-1033	3197	72
H(2Wl)	902	2443	-1217	84
H(lW1)	236	3002	-2233	84
H(2W2)	1534	-598	-2434	72
H(1W2)	386	-844	-2076	72
H(lW3)	4614	-3435	2358	66
H (2W3)	4723	-2177	2273	66

Table 6 Hydrogen bonds of the title compound (Å,º).

D-H...A	(D-H)	(H...A)	<(DHA)	(D.. .A)	A	
N5-H2N5	0.980	1.822	174.64	2.799(5)	03W	
N5-H1N5	0.956	2.005	161.26	2.927(4)	02W	[*-x, -y, z*]
N4-H2N4	0.787	2.505	173.10	3.288(4)	01S'	[*-x, -y, z*+l]
N4-H1N4	0.838	2.277	136.13	2.939(5)	03	
N3-H3N	0.908	1.881	167.46	2.774(4)	03	
01W-H2W1	0.918	1.811	159.35	2.690(4)	04W'	[*-x, -y*+1, *-z*]
01W-H2W1	0.918	2.068	137.22	2.812(4)	04W	[*-x, -y*+1, *-z*]
01W-H2W1	0.918	2.179	135.89	2.909(6)	01S	
01W-H2W1	0.918	2.328	149.94	3.157(5)	04W'	[*-x, -y*+1, *-z*]
01W-H1W1	0.937	1.843	170.60	2.771(4)	01	[*-x*-1, *-y*+l, *-z*]
03W-H1W3	0.809	2.067	163.58	2.852(4)	08	[*x*+1, *y*-1, *z*]
03W-H2W3	0.788	1.993	162.72	2.755(5)	05	[*x*+1, *y*, *z*]
02W-H2W2	0.835	1.955	174.32	2.787(4)	04	[*-x, -y, -z*]
02W-H1W2	0.889	2.315	115.35	2.813(5)	01S	[*-x, -y, -z*]

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