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Crystal structure of TrichloroIsoquinoline Cobalt(III) Isoquinoline

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Abstract: The Crystal structure of Isoquinoline Cobalt(III) Isoquinoline Crystallizes in Triclinic P-1 space group. Crystal data were collected using BRUKER SMART APEX II CCD X-ray diffractometer. The structure was solved by direct methods and refined on F2 by full-matrix least-squares procedures to the final R1 of 0.0761 using SHELXL programs.
Key Words: Isoquinoline, crystal structure.

Introduction

Isoquinoline derivatives are of interest in synthesizing new fungicides, insecticides, textile assistants, corrosion inhibitors, dye stabilizers, and pharmaceuticals[4] Against this background and to ascertain the molecular structure and conformation of the title compound, the crystal structure determination has been carried out.

Experimental

X-ray Structure Determination

Single crystal of the compound suitable for x-ray diffraction was obtained by slow evaporation method. Three dimensional intensity data were collected on a Bruker1 SMART APEX CCD Diffractometer using graphite monochromatized Mo-Kα radiation (λ= 0.71073 Å) at the CAS in Crystallography and Biophysics, Pondicherry University, Pondicherry. The structure was solved by direct methods and refined on F2 by full-matrix least-squares procedures using the SHELXL rograms[5]. All the non-hydrogen atoms were refined using isotropic and later anisotropic thermal parameters. The hydrogen atoms were included in the structure factor calculation at idealized positions by using a riding model, but not refined. Images were created with the ORTEP-3[3]. The crystallographic data for the compound are listed in Table 1.

Table 1. Crystal data and structure refinement for the titled compound

Compound	**I**
Empirical formula	C18 H14 Cl3 Co N2
Formula weight	423.59
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic

space group	P-1
Unit cell dimensions	
A	8.2533(6) Å
B	9.7539(8) Å
C	12.0249(9) Å
A	97.915(6)°
B	98.228(6)°
ϒ	104.956(6)°
Volume	910.04(12) $Å^3$
Z, Calculated density	3, 2.319 Mg/m^3
Absorption coefficient	2.076 mm-1
F(000)	642
Crystal size	0.45 x 0.35 x 0.35 mm
Theta range for data collection	2.60 to 25.00°
Limiting indices	
	-9<=h<=9
	-11<=k<=10
	-14<=l<=14
Reflections collected / unique	5800 / 3193 [R(int) = 0.0174]
Completeness to theta = 25.00	99.80%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3193 / 0 / 150
Goodness-of-fit on F^2	1.084
Final R indices [I>2sigma(I)]	R1 = 0.0700, wR2 = 0.1676
R indices (all data)	R1 = 0.0787, wR2 = 0.1743
Largest diff. peak and hole	1.530 and -1.312 $e.A^{-3}$

Synthesis of the compound

To a reaction mixture of 0.5g of *trans*-$[Co^{III}(en)_2Cl_2]Cl$ complex in 10ml of aqueous solution. 0.5ml of isoquinoline (IQ) added, reflection the solution 50C in 12 hours. Pink colour solid from green solution. Solid was filter and dry, recrystallization using hot water. After 10 days growth using single crystal refinement analysis. The resulting solution was subjected to crystallization by slow evaporation of the solvent resulting in single crystals suitable for x-ray diffraction studies.

Results and Discussion

The *ORTEP* plot of the molecule is shown in Fig. 1. The isoquinoline ion is planar with the Co^{III} atom on the special position. The bond lengths and angles in the title compound are within normal ranges and are comparable with those in related structures[2]. The dihedral angle between isoquinoline ring 1 and 2 is0.8(2)°.The crystal packing is stabilized by intermolecular N—H…Cl and C—H…Cl interactions, which are linking the molecules to a (Table 2)

Table 2: Hydrogen-bond geometry [Å] for compound

D—H…A	**D—H**	**H…A**	**D…A**	**D—H…A**
N15 -H15…Cl3	0.95(3)	2.81(2)	3.769(3)	176(3)

Symmetry code: i) -1+x,y,z

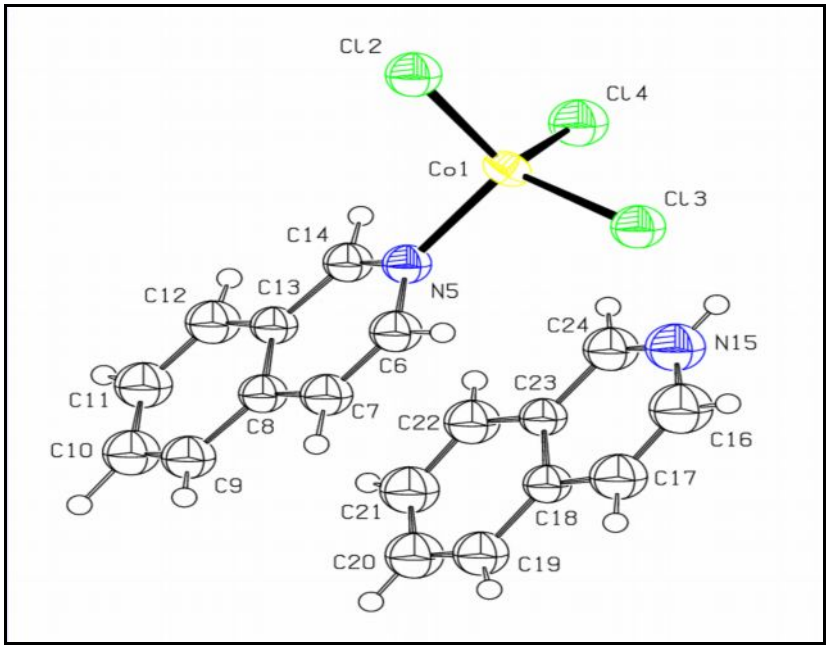

Figure 1: The molecular structure of the title compound, showing the atomic numbering and displacement ellipsoids drawn at 30% probability level.

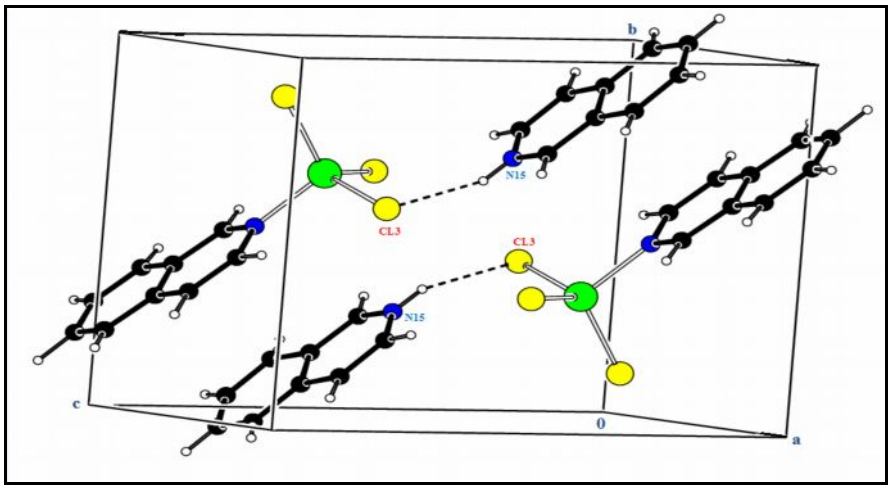

Figure 2: The crystal packing of the titled compound viewed down *a*-axis. The hydrogen bonds are shown as dashed lines (see Table 2 for details; H- atoms involved in H – bonds have been excluded for clarity).

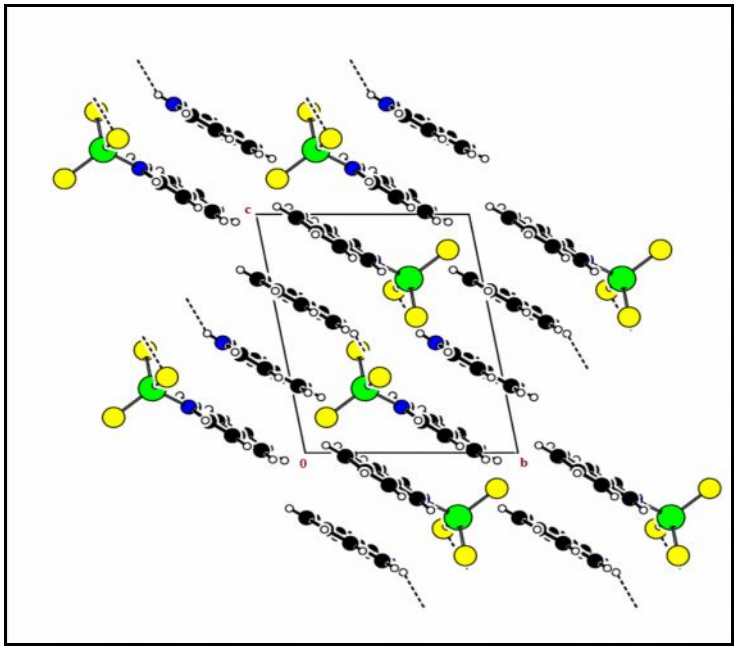

Figure 3: The crystal packing of the titled compound viewed down *a*-axis.

Table 3: Selected Bond lengths (Å)

Selected bonds	Bond lengths (Å)	Selected bonds	Bond lengths (Å)
Co(1)-N(5)	2.049(5)	C 18 -C 19	1.409(9)
Co 1 -Cl 2	2.230(4)	C 8 -C 7	1.401(8)
Co 1 -Cl 4	2.246(2)	C 8 -C 13	1.413(8)
Co 1 -Cl 3	2.271(3)	C 8 -C 9	1.418(8)
N 5 -C 14	1.317(7)	C 19 -C 20	1.355(9)
N 5 -C 6	1.376(7)	C 9 -C 10	1.350(9)
N 15 -C 24	1.304(8)	C 14 -C 13	1.410(8)
N 15 -C 16	1.353(9)	C 13 -C 12	1.412(8)
C 23 -C 24	1.393(8)	C 12 -C 11	1.361(9)
C 23 -C 18	1.414(8)	C 22 -C 21	1.350(10)
C 23 -C 22	1.420(9)	C 21 -C 20	1.385(10)
C 6 -C 7	1.357(8)	C 17 -C 16	1.350(10)
C 18 -C 17	1.406(9)	C 11 -C 10	1.402(10)

Table 4: Selected Bond angles (deg)

Selected angles	Bond angles (deg.)	Selected angles	Bond angles (deg.)
N 5 -Co 1 -Cl 2	108.89(1)	C 7 -C 8 -C 9	123.6(3)
N 5 -Co 1 -Cl 4	104.97(2)	C 13 -C 8 -C 9	118.4(2)
Cl 2 -Co 1 -Cl 4	115.49(2)	C 20 -C 19 -C 18	119.9(2)
N 5 -Co 1 -Cl 3	106.27(1)	C 10 -C 9 -C 8	120.3(1)
Cl 2 -Co 1 -Cl 3	111.23(2)	N 5 -C 14 -C 13	123.6(1)
Cl 4 -Co 1 -Cl 3	109.42(2)	C 14 -C 13 -C 12	122.3(1)
C 14 -N 5 -C 6	118.0(3)	C 14 -C 13 -C 8	117.7(2)
C 14 -N 5 -Co 1	122.9(2)	C 12 -C 13 -C 8	120.0(1)

C 6 -N 5 -Co 1	119.0(2)	C 11 -C 12 -C 13	119.5(2)
C 24 -N 15 -C 16	122.5(2)	C 6 -C 7 -C 8	120.0(2)
C 24 -C 23 -C 18	118.8(2)	C 21 -C 22 -C 23	119.8(1)
C 24 -C 23 -C 22	121.8(1)	C 22 -C 21 -C 20	120.6(2)
C 18 -C 23 -C 22	119.3(2)	C 16 -C 17 -C 18	119.8(2)
C 7 -C 6 -N 5	122.7(2)	C 12 -C 11 -C 10	120.7(2)
C 17 -C 18 -C 19	123.6(1)	N 15 -C 24 -C 23	120.5(2)
C 17 -C 18 -C 23	117.8(2)	C 19 -C 20 -C 21	121.7(1)
C 19 -C 18 -C 23	118.6(1)	C 9 -C 10 -C 11	121.0(2)
C 7 -C 8 -C 13	118.0(1)	C 17 -C 16 -N 15	120.6(2)

Conclusion

The crystal structure analysis of a novel isoquinoline compound was studied using x-ray diffraction method. In the compound the packing of the crystal structure is stabilized by intermolecular C—H…Cl hydrogen bonds. It also features a N—H…Cl hydrogen bond.

Acknowledgments

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