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Dichlorido[*N,N*-dimethyl-*N'*-[1-(pyridin-2-yl)ethylidene]ethane-1,2-diamine- κ^3 *N,N',N''*]cadmium

Nura Suleiman Gwaram, Hamid Khaledi* and Hapipah Mohd Ali

Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: khaledi@siswa.um.edu.my

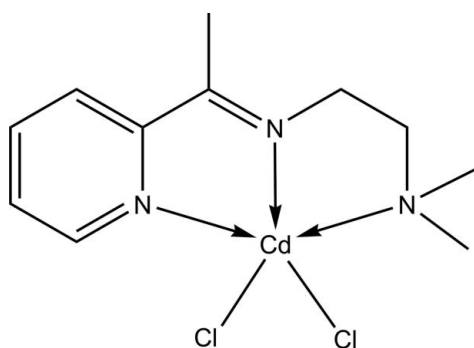
Received 9 February 2011; accepted 15 February 2011

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.016; wR factor = 0.038; data-to-parameter ratio = 16.9.

In the title compound, $[\text{CdCl}_2(\text{C}_{11}\text{H}_{17}\text{N}_3)]$, the Schiff base acts as an *N,N',N''*-tridentate ligand towards the Cd^{II} ion. Two Cl atoms complete a distorted square-pyramidal geometry around the metal atom. In the crystal, a $\text{C}-\text{H}\cdots\text{Cl}$ interaction connects pairs of molecules into centrosymmetric dimers.

Related literature

For the structure of a CuCl_2 complex of the same Schiff base, see: Saleh Salga *et al.* (2010). For the structure of a similar Cd^{II} complex, see: Bian *et al.* (2003). For a description of the geometry of complexes with five-coordinate metal atoms, see: Addison *et al.* (1984).



Experimental

Crystal data

$[\text{CdCl}_2(\text{C}_{11}\text{H}_{17}\text{N}_3)]$

$M_r = 374.58$

Triclinic, $P\bar{1}$
 $a = 8.0276$ (2) Å
 $b = 9.6048$ (2) Å
 $c = 10.0851$ (2) Å
 $\alpha = 102.534$ (1)°
 $\beta = 103.365$ (1)°
 $\gamma = 97.850$ (1)°

$V = 724.09$ (3) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 1.86$ mm⁻¹
 $T = 100$ K
 $0.23 \times 0.11 \times 0.04$ mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.674$, $T_{\max} = 0.929$

5023 measured reflections
2652 independent reflections
2547 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.014$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.016$
 $wR(F^2) = 0.038$
 $S = 1.08$
2652 reflections

157 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.39$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C}7-\text{H}7\text{B}\cdots\text{Cl}2^i$ | 0.98 | 2.77 | 3.679 (2) | 155 |

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: SHELXL97 and publCIF (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2387).

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supporting information

Acta Cryst. (2011). E67, m348 [doi:10.1107/S1600536811005538]

Dichlorido{*N,N*-dimethyl-*N'*-[1-(pyridin-2-yl)ethylidene]ethane-1,2-diamine- κ^3 *N,N',N''*}cadmium

Nura Suleiman Gwaram, Hamid Khaledi and Hapipah Mohd Ali

S1. Comment

The title compound was obtained *via* the complexation of CdCl₂ by *N,N*-dimethyl-*N'*-[methyl(2-pyridyl)methylene]ethane-1,2-diamine. Similar to the structure of the analogous copper(II) complex (Saleh Salga *et al.*, 2010), the cadmium(II) ion is penta-coordinated by the *N,N,N''*-tridentate Schiff base ligand and two Cl atoms in a distorted square-pyramidal geometry, the τ value (Addison *et al.*, 1984) being 0.17. This arrangement is similar to what was observed in the structure of [CdCl₂(C₁₀H₁₅N₃)], the closest analogous cadmium complex (Bian *et al.*, 2003). In the crystal, pairs of the molecules, related by symmetry $-x, -y, -z + 1$, are bonded into centrosymmetric dimers *via* C7—H7B \cdots Cl2 interaction.

S2. Experimental

A mixture of 2-acetylpyridine (0.61 g, 5 mmol) and *N,N*-dimethylethyldiamine (0.44 g, 5 mmol) in ethanol (50 ml) was refluxed for 2 hr followed by addition of a solution of cadmium(II) chloride (0.92 g, 5 mmol) in a minimum amount of water. The resulting solution was refluxed for 30 min, then set aside at room temperature. The colorless crystals of the title compound were obtained after a few days.

S3. Refinement

Hydrogen atoms were placed at calculated positions at distances C—H = 0.95, 0.98 and 0.99 ° for aryl, methyl and methylene type H-atoms, respectively, and were treated as riding on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5$ times $U_{\text{eq}}(\text{C})$.

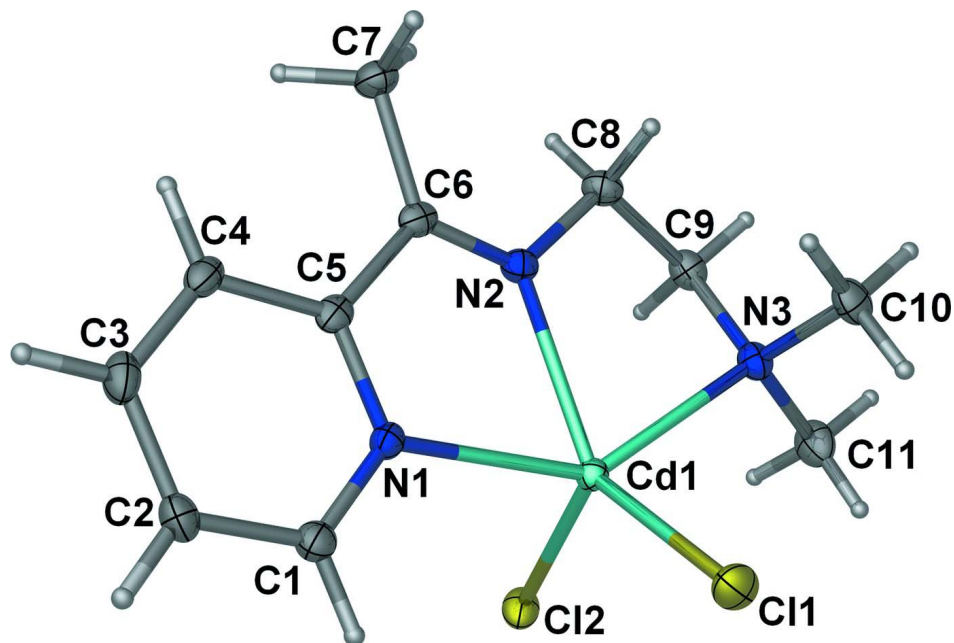


Figure 1

Thermal ellipsoid plot of the title compound at the 50% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

Dichlorido{*N,N*-dimethyl-*N'*- [1-(pyridin-2-yl)ethylidene]ethane-1,2-diamine- κ^3N,N',N'' }cadmium

Crystal data

[CdCl₂(C₁₁H₁₇N₃)]

$M_r = 374.58$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.0276$ (2) Å

$b = 9.6048$ (2) Å

$c = 10.0851$ (2) Å

$\alpha = 102.534$ (1)°

$\beta = 103.365$ (1)°

$\gamma = 97.850$ (1)°

$V = 724.09$ (3) Å³

$Z = 2$

$F(000) = 372$

$D_x = 1.718$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5105 reflections

$\theta = 2.2$ – 29.7 °

$\mu = 1.86$ mm⁻¹

$T = 100$ K

Plate, colorless

$0.23 \times 0.11 \times 0.04$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.674$, $T_{\max} = 0.929$

5023 measured reflections

2652 independent reflections

2547 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 25.5$ °, $\theta_{\min} = 2.2$ °

$h = -9 \rightarrow 9$

$k = -11 \rightarrow 11$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.016$
 $wR(F^2) = 0.038$
 $S = 1.08$
 2652 reflections
 157 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0151P)^2 + 0.4207P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.29 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.39 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|----------------|---------------|---------------|----------------------------------|
| Cd1 | -0.076971 (16) | 0.190459 (13) | 0.205336 (13) | 0.01438 (5) |
| Cl1 | -0.19397 (6) | 0.14396 (5) | -0.04976 (5) | 0.02368 (11) |
| Cl2 | -0.21885 (7) | 0.34705 (5) | 0.35214 (5) | 0.02337 (11) |
| N1 | -0.1880 (2) | -0.04027 (16) | 0.23109 (16) | 0.0155 (3) |
| N2 | 0.1417 (2) | 0.10346 (17) | 0.33828 (16) | 0.0171 (3) |
| N3 | 0.1925 (2) | 0.34724 (17) | 0.22918 (16) | 0.0187 (3) |
| C1 | -0.3543 (3) | -0.1088 (2) | 0.1769 (2) | 0.0193 (4) |
| H1 | -0.4351 | -0.0611 | 0.1283 | 0.023* |
| C2 | -0.4143 (3) | -0.2472 (2) | 0.1886 (2) | 0.0214 (4) |
| H2 | -0.5330 | -0.2941 | 0.1471 | 0.026* |
| C3 | -0.2973 (3) | -0.3147 (2) | 0.2619 (2) | 0.0233 (4) |
| H3 | -0.3346 | -0.4091 | 0.2719 | 0.028* |
| C4 | -0.1247 (3) | -0.2432 (2) | 0.3208 (2) | 0.0202 (4) |
| H4 | -0.0429 | -0.2874 | 0.3732 | 0.024* |
| C5 | -0.0729 (2) | -0.1063 (2) | 0.30211 (19) | 0.0166 (4) |
| C6 | 0.1124 (2) | -0.0244 (2) | 0.35613 (19) | 0.0173 (4) |
| C7 | 0.2517 (3) | -0.0968 (2) | 0.4217 (2) | 0.0244 (4) |
| H7A | 0.2968 | -0.1510 | 0.3482 | 0.037* |
| H7B | 0.2025 | -0.1639 | 0.4697 | 0.037* |
| H7C | 0.3470 | -0.0229 | 0.4902 | 0.037* |
| C8 | 0.3182 (2) | 0.1896 (2) | 0.3762 (2) | 0.0212 (4) |
| H8A | 0.3840 | 0.1458 | 0.3117 | 0.025* |
| H8B | 0.3803 | 0.1910 | 0.4737 | 0.025* |
| C9 | 0.3074 (3) | 0.3439 (2) | 0.3656 (2) | 0.0211 (4) |
| H9A | 0.2626 | 0.3932 | 0.4431 | 0.025* |

| | | | | |
|------|------------|------------|------------|------------|
| H9B | 0.4260 | 0.3985 | 0.3773 | 0.025* |
| C10 | 0.2668 (3) | 0.2957 (2) | 0.1115 (2) | 0.0246 (4) |
| H10A | 0.3822 | 0.3559 | 0.1282 | 0.037* |
| H10B | 0.1898 | 0.3027 | 0.0233 | 0.037* |
| H10C | 0.2779 | 0.1942 | 0.1045 | 0.037* |
| C11 | 0.1656 (3) | 0.4971 (2) | 0.2359 (2) | 0.0286 (5) |
| H11A | 0.2779 | 0.5613 | 0.2511 | 0.043* |
| H11B | 0.1150 | 0.5301 | 0.3140 | 0.043* |
| H11C | 0.0860 | 0.4995 | 0.1470 | 0.043* |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|--------------|--------------|
| Cd1 | 0.01536 (8) | 0.01317 (8) | 0.01511 (8) | 0.00330 (5) | 0.00430 (5) | 0.00417 (5) |
| Cl1 | 0.0233 (2) | 0.0301 (3) | 0.0159 (2) | 0.0048 (2) | 0.00258 (18) | 0.00570 (19) |
| Cl2 | 0.0298 (3) | 0.0205 (2) | 0.0265 (2) | 0.0112 (2) | 0.0154 (2) | 0.00792 (19) |
| N1 | 0.0182 (8) | 0.0151 (7) | 0.0148 (7) | 0.0045 (6) | 0.0055 (6) | 0.0048 (6) |
| N2 | 0.0165 (8) | 0.0180 (8) | 0.0158 (8) | 0.0040 (6) | 0.0032 (6) | 0.0031 (6) |
| N3 | 0.0202 (8) | 0.0168 (8) | 0.0194 (8) | 0.0030 (6) | 0.0076 (7) | 0.0029 (6) |
| C1 | 0.0193 (10) | 0.0203 (10) | 0.0203 (9) | 0.0059 (8) | 0.0072 (8) | 0.0063 (8) |
| C2 | 0.0200 (10) | 0.0221 (10) | 0.0230 (10) | 0.0011 (8) | 0.0091 (8) | 0.0063 (8) |
| C3 | 0.0294 (11) | 0.0165 (9) | 0.0289 (11) | 0.0041 (8) | 0.0149 (9) | 0.0084 (8) |
| C4 | 0.0264 (10) | 0.0196 (10) | 0.0211 (10) | 0.0107 (8) | 0.0114 (8) | 0.0095 (8) |
| C5 | 0.0220 (10) | 0.0162 (9) | 0.0141 (9) | 0.0075 (8) | 0.0081 (7) | 0.0033 (7) |
| C6 | 0.0205 (10) | 0.0200 (9) | 0.0129 (9) | 0.0080 (8) | 0.0056 (7) | 0.0036 (7) |
| C7 | 0.0222 (10) | 0.0253 (10) | 0.0275 (11) | 0.0083 (8) | 0.0044 (9) | 0.0107 (9) |
| C8 | 0.0169 (10) | 0.0226 (10) | 0.0218 (10) | 0.0035 (8) | 0.0028 (8) | 0.0038 (8) |
| C9 | 0.0206 (10) | 0.0194 (10) | 0.0198 (10) | -0.0004 (8) | 0.0057 (8) | -0.0001 (8) |
| C10 | 0.0239 (11) | 0.0297 (11) | 0.0202 (10) | 0.0027 (9) | 0.0100 (8) | 0.0032 (8) |
| C11 | 0.0326 (12) | 0.0171 (10) | 0.0388 (12) | 0.0030 (9) | 0.0150 (10) | 0.0083 (9) |

Geometric parameters (Å, °)

| | | | |
|---------|-------------|----------|-----------|
| Cd1—N2 | 2.3188 (15) | C4—C5 | 1.390 (3) |
| Cd1—N1 | 2.3644 (15) | C4—H4 | 0.9500 |
| Cd1—N3 | 2.3909 (16) | C5—C6 | 1.499 (3) |
| Cd1—Cl1 | 2.4434 (5) | C6—C7 | 1.497 (3) |
| Cd1—Cl2 | 2.4561 (5) | C7—H7A | 0.9800 |
| N1—C1 | 1.335 (2) | C7—H7B | 0.9800 |
| N1—C5 | 1.350 (2) | C7—H7C | 0.9800 |
| N2—C6 | 1.277 (2) | C8—C9 | 1.522 (3) |
| N2—C8 | 1.459 (2) | C8—H8A | 0.9900 |
| N3—C10 | 1.471 (2) | C8—H8B | 0.9900 |
| N3—C11 | 1.474 (2) | C9—H9A | 0.9900 |
| N3—C9 | 1.478 (2) | C9—H9B | 0.9900 |
| C1—C2 | 1.389 (3) | C10—H10A | 0.9800 |
| C1—H1 | 0.9500 | C10—H10B | 0.9800 |
| C2—C3 | 1.379 (3) | C10—H10C | 0.9800 |

| | | | |
|-------------|--------------|---------------|-------------|
| C2—H2 | 0.9500 | C11—H11A | 0.9800 |
| C3—C4 | 1.388 (3) | C11—H11B | 0.9800 |
| C3—H3 | 0.9500 | C11—H11C | 0.9800 |
| N2—Cd1—N1 | 69.42 (5) | N1—C5—C6 | 115.96 (16) |
| N2—Cd1—N3 | 73.39 (5) | C4—C5—C6 | 122.77 (17) |
| N1—Cd1—N3 | 141.34 (5) | N2—C6—C7 | 123.96 (18) |
| N2—Cd1—C11 | 131.13 (4) | N2—C6—C5 | 116.71 (16) |
| N1—Cd1—C11 | 98.62 (4) | C7—C6—C5 | 119.29 (16) |
| N3—Cd1—C11 | 97.82 (4) | C6—C7—H7A | 109.5 |
| N2—Cd1—C12 | 112.49 (4) | C6—C7—H7B | 109.5 |
| N1—Cd1—C12 | 100.86 (4) | H7A—C7—H7B | 109.5 |
| N3—Cd1—C12 | 102.71 (4) | C6—C7—H7C | 109.5 |
| C11—Cd1—C12 | 116.315 (17) | H7A—C7—H7C | 109.5 |
| C1—N1—C5 | 119.09 (16) | H7B—C7—H7C | 109.5 |
| C1—N1—Cd1 | 124.01 (12) | N2—C8—C9 | 109.01 (16) |
| C5—N1—Cd1 | 116.87 (12) | N2—C8—H8A | 109.9 |
| C6—N2—C8 | 121.86 (16) | C9—C8—H8A | 109.9 |
| C6—N2—Cd1 | 120.70 (13) | N2—C8—H8B | 109.9 |
| C8—N2—Cd1 | 116.44 (11) | C9—C8—H8B | 109.9 |
| C10—N3—C11 | 110.06 (16) | H8A—C8—H8B | 108.3 |
| C10—N3—C9 | 111.56 (15) | N3—C9—C8 | 112.34 (15) |
| C11—N3—C9 | 108.99 (15) | N3—C9—H9A | 109.1 |
| C10—N3—Cd1 | 110.95 (11) | C8—C9—H9A | 109.1 |
| C11—N3—Cd1 | 110.21 (12) | N3—C9—H9B | 109.1 |
| C9—N3—Cd1 | 104.94 (11) | C8—C9—H9B | 109.1 |
| N1—C1—C2 | 122.72 (18) | H9A—C9—H9B | 107.9 |
| N1—C1—H1 | 118.6 | N3—C10—H10A | 109.5 |
| C2—C1—H1 | 118.6 | N3—C10—H10B | 109.5 |
| C3—C2—C1 | 118.40 (18) | H10A—C10—H10B | 109.5 |
| C3—C2—H2 | 120.8 | N3—C10—H10C | 109.5 |
| C1—C2—H2 | 120.8 | H10A—C10—H10C | 109.5 |
| C2—C3—C4 | 119.34 (18) | H10B—C10—H10C | 109.5 |
| C2—C3—H3 | 120.3 | N3—C11—H11A | 109.5 |
| C4—C3—H3 | 120.3 | N3—C11—H11B | 109.5 |
| C3—C4—C5 | 119.17 (18) | H11A—C11—H11B | 109.5 |
| C3—C4—H4 | 120.4 | N3—C11—H11C | 109.5 |
| C5—C4—H4 | 120.4 | H11A—C11—H11C | 109.5 |
| N1—C5—C4 | 121.26 (17) | H11B—C11—H11C | 109.5 |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------|-------|-------------|-------------|---------------|
| $C7-H7B\cdots Cl2^i$ | 0.98 | 2.77 | 3.679 (2) | 155 |

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