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catena-Poly[[diiodidocadmium]-μ-[4,4′-(2,3,5,6-tetramethyl-1,4-phenylene)bis(methylene)]bis(3,5-dimethyl-1*H*-pyrazole)-$\kappa^2N^2:N^{2'}$]

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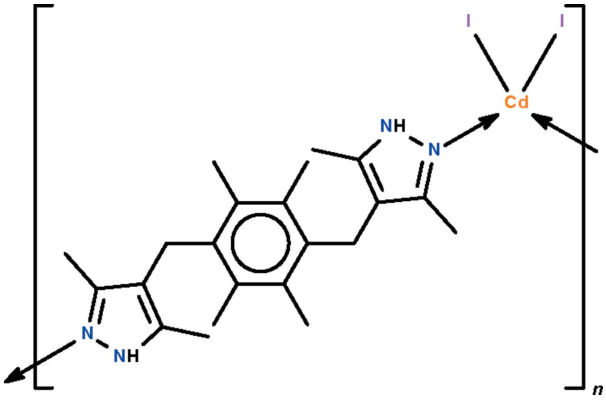
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; R factor = 0.054; wR factor = 0.134; data-to-parameter ratio = 21.5.

The heterocylic ligand of the polymeric title compound, $[CdI_2(C_{22}H_{30}N_4)]$, links two adjacent CdI_2 units, forming a chain running parallel to $[\bar{1}01]$. The Cd^{II} atom is located on a twofold rotation axis and shows a distorted tetrahedral CdI_2N_2 coordination. The mid-point of the benzene ring of the ligand lies on a center of inversion. There are no classical hydrogen-bonding interactions present.

Related literature

For the synthesis of the ligand, see: Trofimenko (1970).



Experimental

Crystal data

$[CdI_2(C_{22}H_{30}N_4)]$
M_r = 716.70
Monoclinic, $C2/c$
a = 22.118 (8) Å
b = 6.840 (2) Å
c = 17.057 (6) Å
β = 93.407 (5)°
V = 2575.8 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.26 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku Saturn 724 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2006)
T_{min} = 0.763, T_{max} = 1.000
14901 measured reflections
2951 independent reflections
2589 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.134
S = 1.18
2951 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.74 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2006); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *publCIF* (Westrip, 2010).

We thank Zhengzhou University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2507).

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supporting information

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S1. Comment

The heterocyclic ligand is a new member of the class of geminal bis-(1-pyrazol-1-yl)alkanes developed fourty years ago by Trofimenko, who also investigated its coordination abilities (Trofimenko, 1970). However, no crystal structures of adducts of the parent 4,4′-(1,4-phenylene)bis(methylene)bis(3,5-dimethyl-1*H*-pyrazole) ligand have been reported to date.

The heterocylic ligand of the polymeric title compound $CdI_2(C_{22}H_{30}N_4)$ (Fig. 1) links two adjacent CdI_2 units to form a chain running parallel to [$\bar{1}$01]. The Cd^{II} atom shows a distorted tetrahedral CdI_2N_2 coordination. The 1*H*-pyrazole H atom is not involved in hydrogen bonding interactions, presumably due to the presence of the bulky methyl group and the CdI_2 unit in its vicinity.

S2. Experimental

A solution of cadmium diiodide (7.3 mg, 0.02 mmol) in methanol (2 ml) was added to a solution of 4,4′-(2,3,5,6-tetramethyl-1,4-phenylene)bis(methylene)bis(3,5-dimethyl-1*H*-pyrazole) (3.5 mg, 0.01 mmol) in ethanol (2 ml). The solution was allowed to evaporate for several days to afford colorless crystals in 80% yield. Calc. for $C_{22}H_{30}N_4CdI_2$: C 36.87, H 4.22, N 7.82%. Found: C 36.71, H 4.25, N 7.69%.

S3. Refinement

H atoms were placed in calculated positions [C—H 0.93–0.98 Å, N–H 0.88 Å; U(H) = 1.2–1.5U_{eq}(C,*N*)]. The highest peak in the final difference Fourier map is in the vicinity (1.35 Å) of H10B.

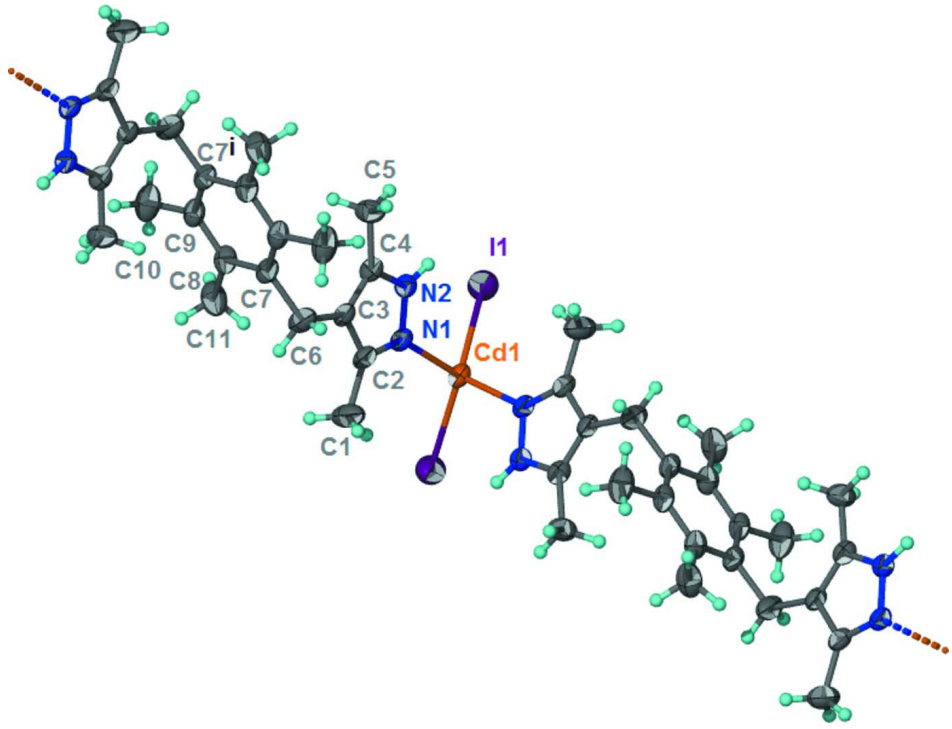

Figure 1

Thermal ellipsoid plot of a portion of the polymeric chain structure of $CdI_2(C_{22}H_{30}N_4)$. Ellipsoids are drawn at the 50% probability level. [Symmetry code: i) -x+1/2, -y+5/2, -z.]

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M_r = 716.70
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Hall symbol: -C 2yc
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b = 6.840 (2) Å
c = 17.057 (6) Å
β = 93.407 (5)°
V = 2575.8 (16) Å^3
Z = 4
$F(000)$ = 1376
D_x = 1.848 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3092 reflections
θ = 2.1–30.6°
μ = 3.26 mm^{-1}
T = 293 K
Cuboid, colorless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku Saturn 724 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2006)
T_{min} = 0.763, T_{max} = 1.000
14901 measured reflections
2951 independent reflections
2589 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −28→28
k = −8→8
l = −22→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.134$
$S = 1.18$
2951 reflections
137 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
$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 7.4182P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.12$ e Å^{-3}
$\Delta\rho_{min} = -0.74$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00092 (15)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.53637 (2)	0.41499 (7)	0.12116 (3)	0.0692 (2)
Cd1	0.5000	0.60117 (8)	0.2500	0.0424 (2)
N1	0.43399 (19)	0.8146 (7)	0.1908 (2)	0.0438 (10)
N2	0.44190 (19)	0.8589 (6)	0.1144 (2)	0.0421 (10)
H2	0.4695	0.8045	0.0863	0.051*
C1	0.3667 (4)	0.9231 (11)	0.2924 (4)	0.071 (2)
H1A	0.3239	0.8970	0.2889	0.106*
H1B	0.3741	1.0464	0.3181	0.106*
H1C	0.3873	0.8213	0.3220	0.106*
C2	0.3894 (2)	0.9304 (8)	0.2115 (3)	0.0433 (12)
C3	0.3678 (2)	1.0496 (8)	0.1477 (3)	0.0409 (11)
C4	0.4023 (2)	0.9968 (8)	0.0868 (3)	0.0421 (11)
C5	0.4040 (3)	1.0599 (9)	0.0030 (3)	0.0554 (15)
H5A	0.3636	1.0599	−0.0210	0.083*
H5B	0.4287	0.9710	−0.0246	0.083*
H5C	0.4206	1.1892	0.0009	0.083*
C6	0.3180 (3)	1.1960 (10)	0.1503 (3)	0.0590 (16)
H6A	0.3353	1.3203	0.1674	0.071*
H6B	0.2905	1.1547	0.1893	0.071*
C7	0.2817 (2)	1.2271 (8)	0.0728 (3)	0.0438 (12)
C8	0.2379 (2)	1.0890 (7)	0.0473 (4)	0.0469 (13)
C9	0.2061 (2)	1.1132 (8)	−0.0256 (3)	0.0460 (12)
C10	0.1571 (3)	0.9684 (10)	−0.0503 (5)	0.0701 (19)
H10A	0.1639	0.9195	−0.1018	0.105*
H10B	0.1183	1.0318	−0.0512	0.105*
H10C	0.1578	0.8617	−0.0137	0.105*
C11	0.2249 (3)	0.9125 (9)	0.0990 (5)	0.0687 (19)

H11A	0.2565	0.8998	0.1397	0.103*
H11B	0.2232	0.7962	0.0675	0.103*
H11C	0.1868	0.9310	0.1223	0.103*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0695 (3)	0.0714 (4)	0.0668 (3)	0.0130 (2)	0.0040 (2)	−0.0205 (2)
Cd1	0.0394 (3)	0.0439 (3)	0.0428 (3)	0.000	−0.0077 (2)	0.000
N1	0.039 (2)	0.053 (3)	0.038 (2)	0.007 (2)	−0.0068 (18)	−0.001 (2)
N2	0.037 (2)	0.048 (2)	0.041 (2)	0.0054 (19)	−0.0001 (18)	−0.0006 (19)
C1	0.084 (5)	0.087 (5)	0.040 (3)	0.029 (4)	0.004 (3)	−0.001 (3)
C2	0.043 (3)	0.048 (3)	0.037 (3)	0.003 (2)	−0.010 (2)	−0.005 (2)
C3	0.037 (3)	0.046 (3)	0.038 (2)	0.007 (2)	−0.008 (2)	−0.005 (2)
C4	0.044 (3)	0.039 (3)	0.042 (3)	0.003 (2)	−0.006 (2)	−0.001 (2)
C5	0.067 (4)	0.058 (3)	0.042 (3)	0.018 (3)	0.010 (3)	0.001 (3)
C6	0.060 (4)	0.072 (4)	0.043 (3)	0.025 (3)	−0.008 (3)	−0.011 (3)
C7	0.039 (3)	0.045 (3)	0.047 (3)	0.017 (2)	−0.007 (2)	−0.005 (2)
C8	0.040 (3)	0.039 (3)	0.062 (3)	0.007 (2)	−0.001 (2)	0.006 (2)
C9	0.036 (3)	0.042 (3)	0.060 (3)	0.006 (2)	−0.004 (2)	−0.009 (2)
C10	0.052 (4)	0.055 (4)	0.101 (5)	−0.002 (3)	−0.011 (4)	−0.016 (4)
C11	0.050 (4)	0.063 (4)	0.093 (5)	0.004 (3)	0.009 (3)	0.024 (4)

Geometric parameters (Å, º)

I1—Cd1	2.7034 (8)	C5—H5B	0.9600
Cd1—N1^{i}	2.260 (4)	C5—H5C	0.9600
Cd1—N1	2.260 (4)	C6—C7	1.520 (7)
Cd1—I1^{i}	2.7034 (8)	C6—H6A	0.9700
N1—C2	1.329 (7)	C6—H6B	0.9700
N1—N2	1.358 (6)	C7—C9^{ii}	1.392 (8)
N2—C4	1.353 (6)	C7—C8	1.403 (8)
N2—H2	0.8800	C8—C9	1.403 (8)
C1—C2	1.497 (8)	C8—C11	1.532 (8)
C1—H1A	0.9600	C9—C7^{ii}	1.392 (8)
C1—H1B	0.9600	C9—C10	1.509 (8)
C1—H1C	0.9600	C10—H10A	0.9600
C2—C3	1.420 (7)	C10—H10B	0.9600
C3—C4	1.375 (7)	C10—H10C	0.9600
C3—C6	1.491 (7)	C11—H11A	0.9600
C4—C5	1.494 (7)	C11—H11B	0.9600
C5—H5A	0.9600	C11—H11C	0.9600
N1^{i}—Cd1—N1	99.6 (2)	C4—C5—H5C	109.5
N1^{i}—Cd1—I1	116.88 (12)	H5A—C5—H5C	109.5
N1—Cd1—I1	98.99 (11)	H5B—C5—H5C	109.5
N1^{i}—Cd1—I1^{i}	98.99 (11)	C3—C6—C7	114.9 (4)
N1—Cd1—I1^{i}	116.88 (12)	C3—C6—H6A	108.5

I1—Cd1—I1^{i}	123.80 (4)	C7—C6—H6A	108.5
C2—N1—N2	105.2 (4)	C3—C6—H6B	108.5
C2—N1—Cd1	137.3 (4)	C7—C6—H6B	108.5
N2—N1—Cd1	117.2 (3)	H6A—C6—H6B	107.5
C4—N2—N1	111.9 (4)	C9^{ii}—C7—C8	120.3 (5)
C4—N2—H2	124.1	C9^{ii}—C7—C6	120.1 (5)
N1—N2—H2	124.1	C8—C7—C6	119.6 (5)
C2—C1—H1A	109.5	C7—C8—C9	119.7 (5)
C2—C1—H1B	109.5	C7—C8—C11	120.2 (5)
H1A—C1—H1B	109.5	C9—C8—C11	120.1 (5)
C2—C1—H1C	109.5	C7^{ii}—C9—C8	120.0 (5)
H1A—C1—H1C	109.5	C7^{ii}—C9—C10	121.0 (5)
H1B—C1—H1C	109.5	C8—C9—C10	119.0 (5)
N1—C2—C3	111.1 (5)	C9—C10—H10A	109.5
N1—C2—C1	121.4 (5)	C9—C10—H10B	109.5
C3—C2—C1	127.5 (5)	H10A—C10—H10B	109.5
C4—C3—C2	104.6 (4)	C9—C10—H10C	109.5
C4—C3—C6	130.0 (5)	H10A—C10—H10C	109.5
C2—C3—C6	125.4 (5)	H10B—C10—H10C	109.5
N2—C4—C3	107.2 (4)	C8—C11—H11A	109.5
N2—C4—C5	118.8 (5)	C8—C11—H11B	109.5
C3—C4—C5	133.9 (5)	H11A—C11—H11B	109.5
C4—C5—H5A	109.5	C8—C11—H11C	109.5
C4—C5—H5B	109.5	H11A—C11—H11C	109.5
H5A—C5—H5B	109.5	H11B—C11—H11C	109.5

N1^{i}—Cd1—N1—C2	−76.4 (5)	N1—N2—C4—C5	−178.8 (5)
I1—Cd1—N1—C2	164.2 (5)	C2—C3—C4—N2	−1.1 (6)
I1^{i}—Cd1—N1—C2	28.8 (6)	C6—C3—C4—N2	179.4 (6)
N1^{i}—Cd1—N1—N2	96.0 (4)	C2—C3—C4—C5	179.3 (6)
I1—Cd1—N1—N2	−23.4 (4)	C6—C3—C4—C5	−0.2 (11)
I1^{i}—Cd1—N1—N2	−158.7 (3)	C4—C3—C6—C7	29.6 (9)
C2—N1—N2—C4	−1.4 (6)	C2—C3—C6—C7	−149.9 (5)
Cd1—N1—N2—C4	−176.1 (3)	C3—C6—C7—C9^{ii}	−99.5 (7)
N2—N1—C2—C3	0.7 (6)	C3—C6—C7—C8	77.4 (7)
Cd1—N1—C2—C3	173.7 (4)	C9^{ii}—C7—C8—C9	−0.5 (9)
N2—N1—C2—C1	179.2 (5)	C6—C7—C8—C9	−177.5 (5)
Cd1—N1—C2—C1	−7.7 (9)	C9^{ii}—C7—C8—C11	179.5 (5)
N1—C2—C3—C4	0.3 (6)	C6—C7—C8—C11	2.6 (8)
C1—C2—C3—C4	−178.2 (6)	C7—C8—C9—C7^{ii}	0.5 (9)
N1—C2—C3—C6	179.9 (5)	C11—C8—C9—C7^{ii}	−179.5 (5)
C1—C2—C3—C6	1.4 (9)	C7—C8—C9—C10	−177.1 (5)
N1—N2—C4—C3	1.6 (6)	C11—C8—C9—C10	2.9 (8)

Symmetry codes: (i) −*x*+1, *y*, −*z*+1/2; (ii) −*x*+1/2, −*y*+5/2, −*z*.