

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

catena-Poly[[bis(3-acetylpyridine- κ N)-cadmium]-di- μ -selenocyanato- κ^2 N:Se; κ^2 Se:N]

Julia Werner,* Jan Boeckmann, Inke Jess and Christian Näther

Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Max-Eyth-Strasse 2, 24118 Kiel, Germany

Correspondence e-mail: jwerner@ac.uni-kiel.de

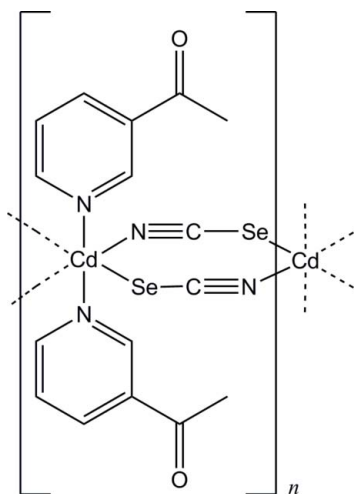
Received 23 April 2012; accepted 24 April 2012

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.031; wR factor = 0.061; data-to-parameter ratio = 21.0.

In the crystal structure of the title compound, $[\text{Cd}(\text{NCSe})_2(\text{C}_7\text{H}_7\text{NO})_2]_n$, the Cd^{2+} cation is coordinated by two 3-acetylpyridine ligands and four μ -1,3-bridging selenocyanate anions within a slightly distorted CdN_4Se_2 octahedron. The asymmetric units consists of one Cd^{2+} cation, which is situated on a center of inversion, as well as one selenocyanate anion and one 3-acetylpyridine ligand in general positions. The metal cations are μ -1,3-bridged *via* the selenocyanate anions into chains along the a axis.

Related literature

For general background information including details on thermal decomposition reactions and magnetic properties of the precursor and μ -1,3 bridging compounds, see: Näther & Greve (2003); Boeckmann & Näther (2010, 2011); Wöhlert *et al.* (2011). For a description of the Cambridge Structural Database, see: Allen (2002).



Experimental

Crystal data

$[\text{Cd}(\text{NCSe})_2(\text{C}_7\text{H}_7\text{NO})_2]$
 $M_r = 564.63$
 Monoclinic, $P2_1/c$
 $a = 5.9447$ (3) Å
 $b = 18.7233$ (10) Å
 $c = 8.7548$ (5) Å
 $\beta = 94.020$ (4)°

$V = 972.05$ (9) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 4.88$ mm⁻¹
 $T = 293$ K
 $0.16 \times 0.07 \times 0.02$ mm

Data collection

Stoe IPDS-2 diffractometer
 Absorption correction: numerical
 (X -SHAPE and X -RED32;
 Stoe & Cie, 2008)
 $T_{\min} = 0.667$, $T_{\max} = 0.902$

17180 measured reflections
 2458 independent reflections
 2256 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.061$
 $S = 1.13$
 2458 reflections

117 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.47$ e Å⁻³
 $\Delta\rho_{\min} = -0.47$ e Å⁻³

Data collection: X -AREA (Stoe & Cie, 2008); cell refinement: X -AREA; data reduction: X -AREA; program(s) used to solve structure: $SHELXS97$ (Sheldrick, 2008); program(s) used to refine structure: $SHELXL97$ (Sheldrick, 2008); molecular graphics: XP (Sheldrick, 2008) and $DIAMOND$ (Brandenburg, 2011); software used to prepare material for publication: $PUBLICIF$ (Westrip, 2010).

We gratefully acknowledge financial support by the DFG (project No. NA 720/3-1) and the State of Schleswig-Holstein. We thank Professor Dr Wolfgang Bensch for access to his experimental facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5897).

References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
 Boeckmann, J. & Näther, C. (2010). *Dalton Trans.* **39**, 11019–11026.
 Boeckmann, J. & Näther, C. (2011). *Chem. Commun.* **47**, 7104–7106.
 Brandenburg, K. (2011). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
 Näther, C. & Greve, J. (2003). *J. Solid State Chem.*, **176**, 259–265.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Stoe & Cie (2008). X -AREA, X -RED32 and X -SHAPE. Stoe & Cie, Darmstadt, Germany.
 Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
 Wöhlert, S., Boeckmann, J., Wriedt, M. & Näther, C. (2011). *Angew. Chem. Int. Ed.* **50**, 6920–6923.

supporting information

Acta Cryst. (2012). E68, m704 [doi:10.1107/S1600536812018375]

catena-Poly[[bis(3-acetylpyridine- κ N)cadmium]-di- μ -selenocyanato- κ^2 N:Se; κ^2 Se:N]

Julia Werner, Jan Boeckmann, Inke Jess and Christian Näther

S1. Comment

The title compound was prepared within a project on the synthesis and the magnetic properties of paramagnetic transition metal thio- and selenocyanato coordination polymers in which the metal cations are μ -1,3 bridged by the anionic ligands (Näther & Greve, 2003, Boeckmann & Näther, 2010, 2011 and Wöhlert *et al.*, 2011). In this context, also the corresponding compounds based on diamagnetic cadmium are of interest, because they are structural analogs of the paramagnetic compounds. In the course of systematic investigations crystals of the title compound were prepared and characterized by single crystal X-ray diffraction.

In the crystal structure of the title compound, the cadmium(II) cations each are coordinated by two nitrogen atoms of two terminal *N*-bonded 3-acetylpyridine and two nitrogen and two selenium atoms of μ -1,3 bridging selenocyanato anions (Fig. 1). The coordination polyhedron of the Cd cations can be described as a slightly distorted octahedra with the Cd cation located on a centre of inversion.

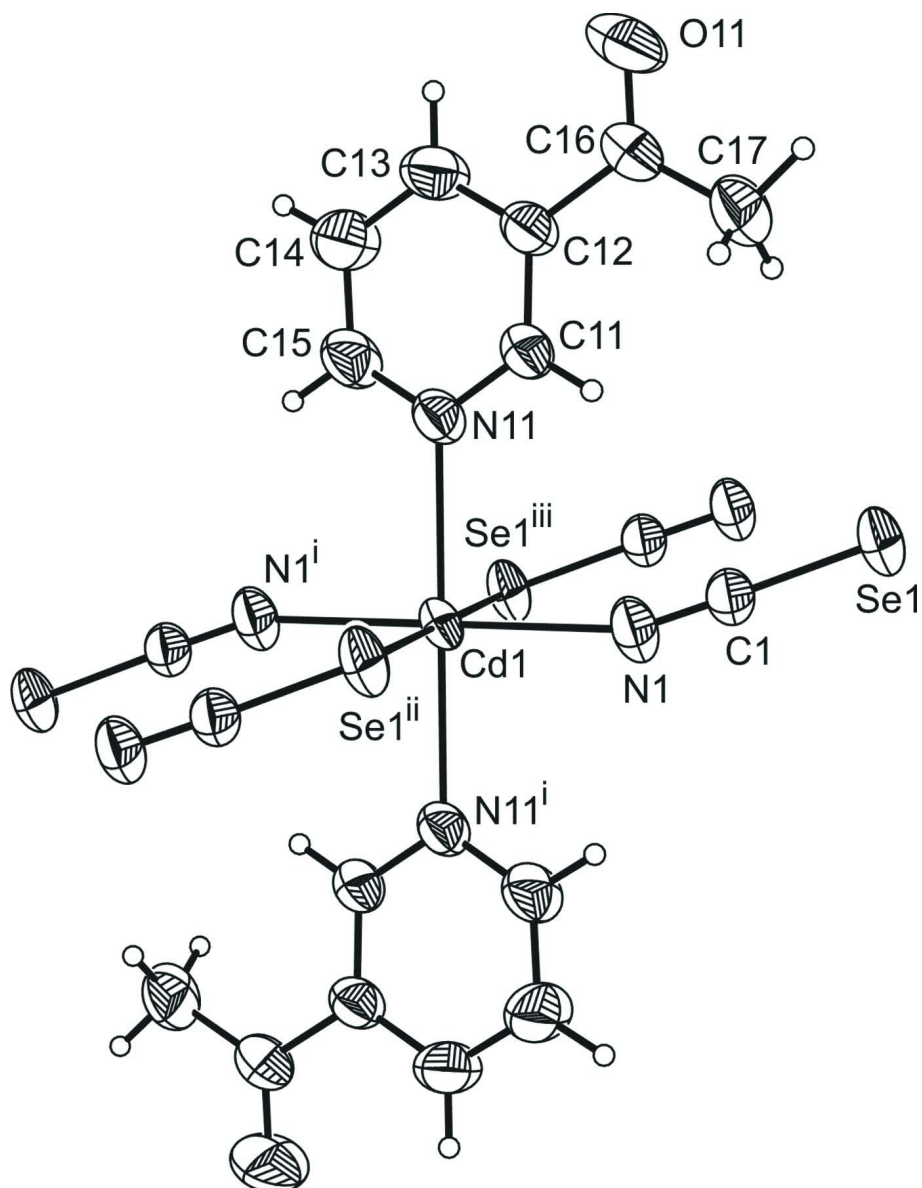
The Cd²⁺ cations are μ -1,3 bridged by selenocyanato anions into chains, which elongate in the direction of the crystallographic *a* axis (Fig. 2). The Cd \cdots Cd intrachain distance amounts to 5.9447 (3) Å and the shortest interchain Cd \cdots Cd distance amounts to 8.7548 (5) Å. It must be noted that according to a search in the CCDC database (ConQuest Ver.1.14.2012) (Allen, 2002) coordination compounds based on metal selenocyanates and 3-acetylpyridine are unknown.

S2. Experimental

Potassium selenocyanate and 3-acetylpyridine were purchased from Alfa Aesar, Cd(NO₃)₂·4H₂O were obtained from Merck. The title compound was prepared by the reaction of 77.1 mg Cd(NO₃)₂·4H₂O (0.25 mmol), 64.8 mg KSeCN (0.45 mmol) and 109 μ L 3-acetylpyridine (1.00 mmol) in 1.5 mL H₂O at RT in a closed 3 ml snap cap vial. After several days colourless needles of the title compound were obtained.

S3. Refinement

H atoms were positioned with idealized geometry (methyl H atoms allowed to rotate but not to tip) and were refined isotropically with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ for aromatic H atoms (1.5 for methyl H atoms) using a riding model with C—H = 0.93 Å (aromatic) and with C—H = 0.96 Å (methyl).

**Figure 1**

Crystal structure of the title compound with labelling and displacement ellipsoids drawn at the 50% probability level.

Symmetry code: i = -x,-y+1,-z, ii = x-1, y, z, iii = -x+1, -y+1, -z

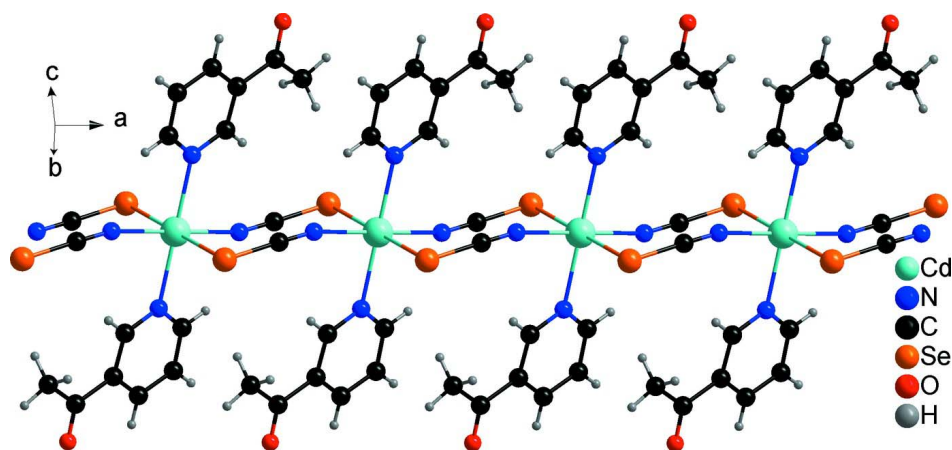


Figure 2

Crystal structure of the title compound with view of the chains that elongate in the direction of the crystallographic *a* axis.

catena-Poly[[bis(3-acetylpyridine- κ N)cadmium]-di- μ -selenocyanato- κ^2 N:Se; κ^2 Se:N]

Crystal data

[Cd(NCSe)₂(C₇H₇NO)₂]

M_r = 564.63

Monoclinic, *P*2₁/*c*

Hall symbol: -P 2ybc

a = 5.9447 (3) Å

b = 18.7233 (10) Å

c = 8.7548 (5) Å

β = 94.020 (4)°

V = 972.05 (9) Å³

Z = 2

F(000) = 540

D_x = 1.929 Mg m⁻³

Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 17180 reflections

θ = 2.2–28.6°

μ = 4.88 mm⁻¹

T = 293 K

Needle, colourless

0.16 × 0.07 × 0.02 mm

Data collection

Stoe IPDS-2

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: numerical

(*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)

T_{min} = 0.667, *T_{max}* = 0.902

17180 measured reflections

2458 independent reflections

2256 reflections with *I* > 2 σ (*I*)

R_{int} = 0.045

θ_{\max} = 28.6°, θ_{\min} = 2.2°

h = -7→7

k = -25→25

l = -11→11

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2 σ (*F*²)] = 0.031

wR(*F*²) = 0.061

S = 1.13

2458 reflections

117 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/[$\sigma^2(F_o^2) + (0.0164P)^2 + 1.1235P$]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} = 0.001

$\Delta\rho_{\max}$ = 0.47 e Å⁻³

$\Delta\rho_{\min}$ = -0.47 e Å⁻³

Extinction correction: *SHELXL97* (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFe^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0074 (5)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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_{iso}^*/U_{eq} |
|------|-------------|---------------|-------------|--------------------|
| Cd1 | 0.0000 | 0.5000 | 0.0000 | 0.03766 (9) |
| N1 | 0.3333 (4) | 0.44388 (15) | 0.0928 (3) | 0.0478 (6) |
| C1 | 0.5139 (4) | 0.43756 (15) | 0.1480 (3) | 0.0377 (6) |
| Se1 | 0.79466 (5) | 0.429324 (18) | 0.23721 (4) | 0.04545 (11) |
| N11 | 0.0930 (4) | 0.60142 (13) | 0.1566 (3) | 0.0422 (5) |
| O11 | 0.5790 (6) | 0.72219 (18) | 0.5227 (4) | 0.1005 (13) |
| C11 | 0.2729 (5) | 0.60423 (17) | 0.2585 (3) | 0.0430 (6) |
| H11 | 0.3604 | 0.5633 | 0.2736 | 0.052* |
| C12 | 0.3348 (5) | 0.66449 (16) | 0.3420 (4) | 0.0442 (7) |
| C13 | 0.2032 (6) | 0.72501 (18) | 0.3206 (5) | 0.0592 (9) |
| H13 | 0.2402 | 0.7666 | 0.3746 | 0.071* |
| C14 | 0.0168 (7) | 0.72283 (19) | 0.2183 (5) | 0.0675 (11) |
| H14 | -0.0757 | 0.7626 | 0.2034 | 0.081* |
| C15 | -0.0302 (6) | 0.66083 (17) | 0.1387 (4) | 0.0533 (8) |
| H15 | -0.1548 | 0.6602 | 0.0683 | 0.064* |
| C16 | 0.5372 (6) | 0.6672 (2) | 0.4547 (4) | 0.0570 (9) |
| C17 | 0.6815 (6) | 0.6032 (2) | 0.4794 (4) | 0.0632 (10) |
| H17A | 0.8070 | 0.6144 | 0.5503 | 0.095* |
| H17B | 0.7360 | 0.5884 | 0.3837 | 0.095* |
| H17C | 0.5951 | 0.5653 | 0.5202 | 0.095* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|---------------|---------------|---------------|
| Cd1 | 0.02405 (13) | 0.04299 (16) | 0.04447 (17) | -0.00160 (10) | -0.00794 (10) | -0.00268 (12) |
| N1 | 0.0285 (11) | 0.0554 (15) | 0.0583 (17) | 0.0002 (10) | -0.0059 (11) | 0.0068 (12) |
| C1 | 0.0305 (13) | 0.0398 (14) | 0.0425 (15) | 0.0013 (10) | 0.0002 (11) | 0.0028 (11) |
| Se1 | 0.02761 (14) | 0.0576 (2) | 0.04962 (19) | 0.00067 (12) | -0.00791 (11) | 0.01084 (14) |
| N11 | 0.0365 (12) | 0.0447 (13) | 0.0439 (14) | -0.0023 (10) | -0.0074 (10) | 0.0003 (10) |
| O11 | 0.088 (2) | 0.082 (2) | 0.123 (3) | 0.0018 (18) | -0.053 (2) | -0.043 (2) |
| C11 | 0.0372 (14) | 0.0447 (15) | 0.0457 (16) | -0.0003 (12) | -0.0075 (12) | -0.0022 (12) |
| C12 | 0.0418 (15) | 0.0442 (15) | 0.0453 (16) | -0.0047 (12) | -0.0064 (12) | -0.0030 (12) |
| C13 | 0.067 (2) | 0.0412 (17) | 0.066 (2) | -0.0009 (15) | -0.0177 (18) | -0.0074 (15) |

| | | | | | | |
|-----|-------------|-------------|-----------|--------------|--------------|--------------|
| C14 | 0.074 (2) | 0.0444 (18) | 0.079 (3) | 0.0125 (17) | -0.029 (2) | -0.0031 (17) |
| C15 | 0.0510 (18) | 0.0498 (18) | 0.056 (2) | -0.0003 (14) | -0.0182 (15) | 0.0033 (14) |
| C16 | 0.0466 (18) | 0.061 (2) | 0.062 (2) | -0.0043 (15) | -0.0136 (15) | -0.0132 (16) |
| C17 | 0.0481 (19) | 0.080 (3) | 0.058 (2) | 0.0041 (18) | -0.0163 (16) | -0.0063 (19) |

Geometric parameters (Å, °)

| | | | |
|---|-------------|---------------|-----------|
| Cd1—N1 ⁱ | 2.337 (2) | C11—H11 | 0.9300 |
| Cd1—N1 | 2.337 (2) | C12—C13 | 1.382 (5) |
| Cd1—N11 | 2.384 (2) | C12—C16 | 1.502 (4) |
| Cd1—N11 ⁱ | 2.384 (2) | C13—C14 | 1.376 (5) |
| Cd1—Se1 ⁱⁱ | 2.8124 (3) | C13—H13 | 0.9300 |
| Cd1—Se1 ⁱⁱⁱ | 2.8124 (3) | C14—C15 | 1.373 (5) |
| N1—C1 | 1.152 (4) | C14—H14 | 0.9300 |
| C1—Se1 | 1.799 (3) | C15—H15 | 0.9300 |
| Se1—Cd1 ^{iv} | 2.8124 (3) | C16—C17 | 1.481 (5) |
| N11—C15 | 1.335 (4) | C17—H17A | 0.9600 |
| N11—C11 | 1.345 (3) | C17—H17B | 0.9600 |
| O11—C16 | 1.206 (4) | C17—H17C | 0.9600 |
| C11—C12 | 1.380 (4) | | |
| N1 ⁱ —Cd1—N1 | 180.00 (12) | C12—C11—H11 | 118.2 |
| N1 ⁱ —Cd1—N11 | 89.92 (9) | C11—C12—C13 | 118.1 (3) |
| N1—Cd1—N11 | 90.08 (9) | C11—C12—C16 | 123.2 (3) |
| N1 ⁱ —Cd1—N11 ⁱ | 90.08 (9) | C13—C12—C16 | 118.7 (3) |
| N1—Cd1—N11 ⁱ | 89.92 (9) | C14—C13—C12 | 119.1 (3) |
| N11—Cd1—N11 ⁱ | 180.0 | C14—C13—H13 | 120.5 |
| N1 ⁱ —Cd1—Se1 ⁱⁱ | 86.16 (7) | C12—C13—H13 | 120.5 |
| N1—Cd1—Se1 ⁱⁱ | 93.84 (7) | C15—C14—C13 | 118.8 (3) |
| N11—Cd1—Se1 ⁱⁱ | 87.41 (6) | C15—C14—H14 | 120.6 |
| N11 ⁱ —Cd1—Se1 ⁱⁱ | 92.59 (6) | C13—C14—H14 | 120.6 |
| N1 ⁱ —Cd1—Se1 ⁱⁱⁱ | 93.84 (7) | N11—C15—C14 | 123.7 (3) |
| N1—Cd1—Se1 ⁱⁱⁱ | 86.16 (7) | N11—C15—H15 | 118.2 |
| N11—Cd1—Se1 ⁱⁱⁱ | 92.59 (6) | C14—C15—H15 | 118.2 |
| N11 ⁱ —Cd1—Se1 ⁱⁱⁱ | 87.41 (6) | O11—C16—C17 | 121.4 (3) |
| Se1 ⁱⁱ —Cd1—Se1 ⁱⁱⁱ | 180.0 | O11—C16—C12 | 118.8 (3) |
| C1—N1—Cd1 | 159.1 (3) | C17—C16—C12 | 119.8 (3) |
| N1—C1—Se1 | 178.7 (3) | C16—C17—H17A | 109.5 |
| C1—Se1—Cd1 ^{iv} | 94.39 (9) | C16—C17—H17B | 109.5 |
| C15—N11—C11 | 116.7 (3) | H17A—C17—H17B | 109.5 |
| C15—N11—Cd1 | 119.5 (2) | C16—C17—H17C | 109.5 |
| C11—N11—Cd1 | 123.7 (2) | H17A—C17—H17C | 109.5 |
| N11—C11—C12 | 123.6 (3) | H17B—C17—H17C | 109.5 |
| N11—C11—H11 | 118.2 | | |

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x+1, -y+1, -z$; (iii) $x-1, y, z$; (iv) $x+1, y, z$.