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Crystal structure of diaquabis(2,6-dimethylpyrazine-κN^4)bis(thiocyanato-κN)cobalt(II) 2,5-dimethylpyrazine monosolvate

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In the crystal structure of the title compound, $[Co(NCS)_2(C_6H_8N_2)_2(H_2O)_2]\cdot C_6H_8N_2$, the Co^{II} cation is coordinated by the N atoms of two terminal thiocyanate anions, the O atoms of two water molecules and two N atoms of two 2,6-dimethylpyrazine ligands. The coordination sphere of the resulting discrete complex is that of a slightly distorted octahedron. The asymmetric unit comprises a Co^{II} cation and half of a 2,5-dimethylpyrazine ligand, both of which are located on centres of inversion, and a water ligand, a 2,6-dimethylpyrazine ligand and one thiocyanate anion in general positions. In the crystal, the discrete complexes are arranged in such a way that cavities are formed in which the 2,5-dimethylpyrazine solvent molecules are located. The coordination of the 2,5-dimethylpyrazine molecules to the metal is apparently hindered due to the bulky methyl groups in vicinal positions to the N atoms, leading to a preferential coordination of the 2,6-dimethylpyrazine ligands. The discrete complexes are linked by O—H···N hydrogen bonds between one water H atom and the non-coordinating N atom of the 2,6-dimethylpyrazine ligands. The remaining water H atom is hydrogen bonded to one N atom of the 2,5-dimethylpyrazine solvent molecule. This arrangement leads to the formation of a two-dimensional network extending parallel to (010).

Keywords: crystal structure; coordination polymer; octahedral coordination; cobalt(II).

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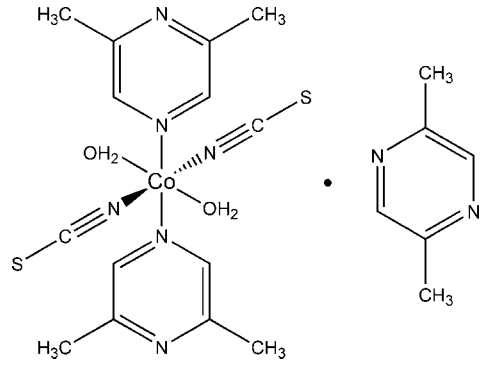
1. Related literature

For structures with metal thiocyanates and 2,5-dimethylpyrazine or 2,6-dimethylpyrazine, see: Otieno *et al.* (2003); Mahmoudi & Morsali (2009).



2. Experimental

2.1. Crystal data

$[Co(NCS)_2(C_6H_8N_2)_2(H_2O)_2]\cdot C_6H_8N_2$
M_r = 535.55
Triclinic, $P\overline{1}$
a = 8.3009 (4) Å
b = 9.0466 (5) Å
c = 10.4200 (6) Å
α = 96.640 (4)°
β = 105.820 (4)°
γ = 116.070 (4)°
V = 650.68 (7) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.85 mm^{-1}
T = 293 K
0.15 × 0.08 × 0.04 mm

2.2. Data collection

Stoe IPDS-2 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
T_{min} = 0.868, T_{max} = 0.959
10848 measured reflections
3447 independent reflections
3175 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.097
S = 1.05
3447 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H2*O*1···N20	0.82	2.01	2.8193 (17)	172
O1—H1*O*1···N10^{i}	0.82	2.01	2.8257 (15)	173

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

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: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Acknowledgements

We thank Professor Dr Wolfgang Bensch for access to his experimental facilities. This project was supported by the Deutsche Forschungsgemeinschaft (Project No. NA 720/5-1) and the State of Schleswig-Holstein.

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5240).

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supporting information

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Crystal structure of diaquabis(2,6-dimethylpyrazine-κN^4)bis(thiocyanato-κN)cobalt(II) 2,5-dimethylpyrazine monosolvate

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S1. Synthesis and crystallization

$Co(SCN)_2$ and 2,5-dimethylpyrazine (97%) were purchased from Alfa Aesar. The title compound was prepared by the reaction of 57.9 mg (0.33 mmol) $Co(SCN)_2$ and 140.0 μl 2,5-dimethylpyrazine (1.28 mmol) in 1.0 ml water at 393 K. After few days block-like crystals of the title compound were obtained that contained 2,6-dimethylpyrazine in addition. Later it was found that the commercially available 2,5-dimethylpyrazine contains about 3% of 2,6-dimethylpyrazine as a contamination.

S2. Refinement

The carbon-bound H atoms were positioned with idealized geometry (methyl H atoms were allowed to rotate but not to tip) and were refined with $U_{iso}(H) = 1.2U_{eq}(C)$ (1.5 for methyl H atoms) using a riding model with C—H = 0.93 Å for aromatic and C—H = 0.96 Å for methyl H atoms. The oxygen-bound H atoms were located in a difference map. The O—H bond length was constrained to 0.82 Å, with $U_{iso}(H) = 1.5U_{eq}(O)$ using a riding model.

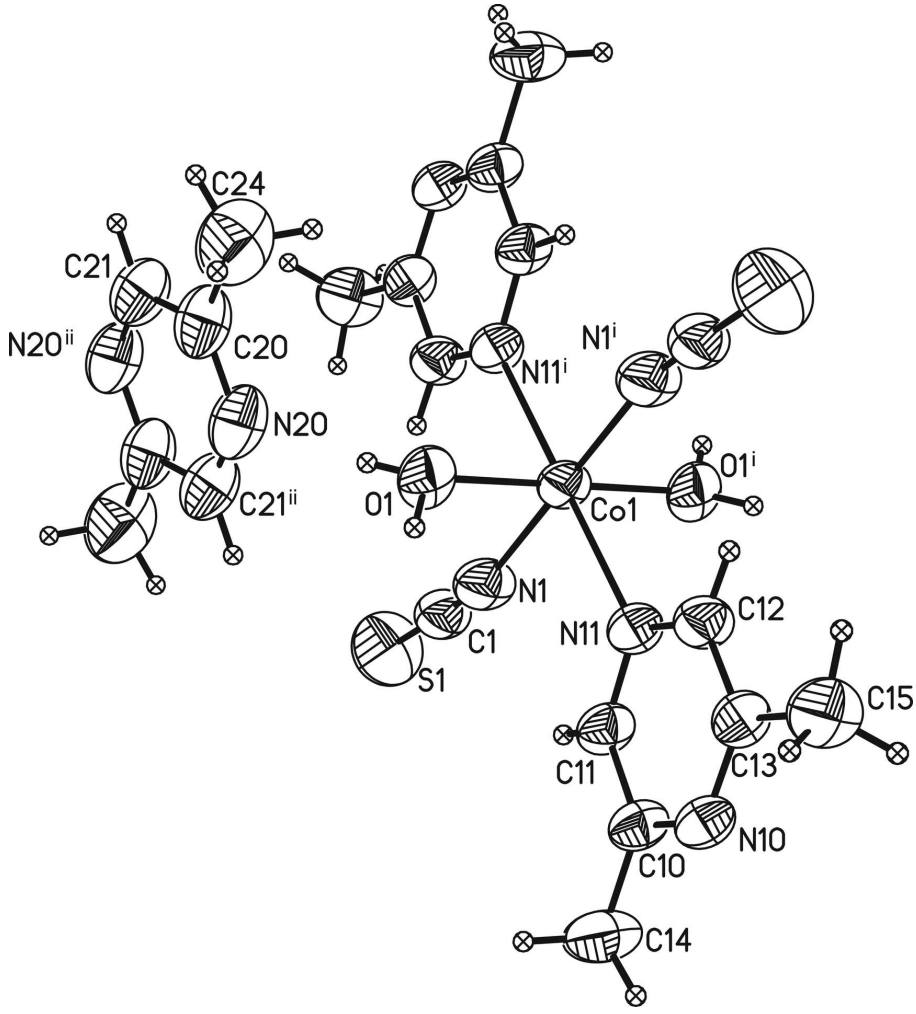

Figure 1

The molecular components in the crystal structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) -*x* + 1, *y* + 1, -*z* + 1; (ii) -*x* + 1, -*y* + 1, -*z*.]

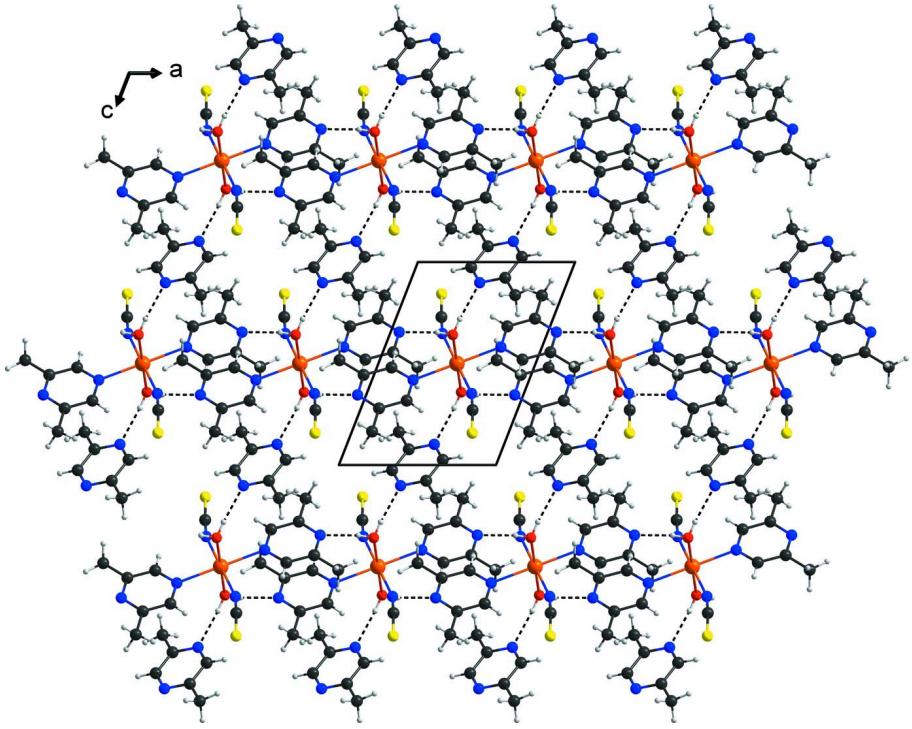

Figure 2

Crystal structure of the title compound in a view along [010]. O—H···N hydrogen bonding is shown as dashed lines.

Diaquabis(2,6-dimethylpyrazine-κN^4)bis(thiocyanato-κN)cobalt(II) 2,5-dimethylpyrazine monosolvate

Crystal data

$[Co(NCS)_2(C_6H_8N_2)_2(H_2O)_2]\cdot C_6H_8N_2$
M_r = 535.55
Triclinic, $P\overline{1}$
a = 8.3009 (4) Å
b = 9.0466 (5) Å
c = 10.4200 (6) Å
α = 96.640 (4)°
β = 105.820 (4)°
γ = 116.070 (4)°
V = 650.68 (7) Å^3
Z = 1
$F(000)$ = 279
D_x = 1.367 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 10848 reflections
θ = 2.1–29.2°
μ = 0.85 mm^{-1}
T = 293 K
Block, purple
0.15 × 0.08 × 0.04 mm

Data collection

Stoe IPDS-2 diffractometer
Radiation source: fine-focus sealed tube
ω scans
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe & Cie, 2008)
T_{min} = 0.868, T_{max} = 0.959
10848 measured reflections
3447 independent reflections
3175 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 29.2°, θ_{min} = 2.9°
h = −11→11
k = −12→12
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.097

S = 1.05
3447 reflections
154 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 0.1118P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

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.

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

	x	y	z	U_{iso}*/U_{eq}
Co1	0.5000	0.5000	0.5000	0.04299 (10)
N1	0.3328 (2)	0.26365 (19)	0.35238 (16)	0.0590 (3)
C1	0.2857 (2)	0.1399 (2)	0.27093 (18)	0.0558 (4)
S1	0.23050 (11)	−0.02995 (8)	0.15745 (7)	0.0943 (2)
N10	−0.05473 (17)	0.39022 (16)	0.65466 (14)	0.0467 (3)
C10	−0.0663 (2)	0.27462 (19)	0.55572 (16)	0.0476 (3)
C11	0.0930 (2)	0.30432 (19)	0.51972 (16)	0.0470 (3)
H11	0.0815	0.2212	0.4508	0.056*
C12	0.2717 (2)	0.5600 (2)	0.68289 (16)	0.0496 (3)
H12	0.3881	0.6602	0.7301	0.059*
C13	0.1150 (2)	0.5321 (2)	0.72101 (16)	0.0472 (3)
C14	−0.2559 (3)	0.1149 (2)	0.4835 (2)	0.0718 (5)
H14A	−0.3458	0.1151	0.5251	0.108*
H14B	−0.2400	0.0171	0.4918	0.108*
H14C	−0.3038	0.1098	0.3873	0.108*
C15	0.1285 (3)	0.6597 (3)	0.8350 (2)	0.0706 (5)
H15A	0.0349	0.6942	0.7993	0.106*
H15B	0.2551	0.7579	0.8702	0.106*
H15C	0.1043	0.6087	0.9083	0.106*
N11	0.26138 (16)	0.44839 (16)	0.58104 (13)	0.0451 (3)
N20	0.4442 (3)	0.5506 (2)	0.10190 (16)	0.0667 (4)
C20	0.5941 (3)	0.6633 (3)	0.07748 (19)	0.0653 (4)
C21	0.6487 (3)	0.6097 (3)	−0.0256 (2)	0.0676 (5)
H21	0.7542	0.6894	−0.0414	0.081*
C24	0.7003 (5)	0.8448 (3)	0.1628 (3)	0.1000 (9)
H24A	0.6111	0.8861	0.1601	0.150*
H24B	0.7935	0.9135	0.1263	0.150*
H24C	0.7647	0.8517	0.2569	0.150*
O1	0.40223 (16)	0.61584 (16)	0.35860 (12)	0.0560 (3)
H1O1	0.2978	0.6105	0.3470	0.084*
H2O1	0.4074	0.6011	0.2810	0.084*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.03641 (14)	0.05128 (17)	0.04350 (15)	0.02088 (11)	0.02181 (10)	0.00580 (10)
N1	0.0497 (7)	0.0613 (8)	0.0578 (8)	0.0190 (6)	0.0283 (6)	0.0010 (6)
C1	0.0474 (7)	0.0589 (9)	0.0522 (8)	0.0181 (6)	0.0230 (6)	0.0064 (7)
S1	0.1115 (5)	0.0684 (3)	0.0760 (4)	0.0327 (3)	0.0290 (3)	−0.0124 (3)
N10	0.0437 (6)	0.0543 (7)	0.0542 (7)	0.0274 (5)	0.0288 (5)	0.0168 (5)
C10	0.0441 (6)	0.0505 (7)	0.0552 (8)	0.0230 (6)	0.0282 (6)	0.0152 (6)
C11	0.0457 (7)	0.0508 (7)	0.0521 (7)	0.0248 (6)	0.0279 (6)	0.0115 (6)
C12	0.0398 (6)	0.0572 (8)	0.0516 (7)	0.0225 (6)	0.0214 (6)	0.0083 (6)
C13	0.0462 (7)	0.0567 (8)	0.0486 (7)	0.0293 (6)	0.0250 (6)	0.0123 (6)
C14	0.0537 (9)	0.0600 (10)	0.0881 (14)	0.0129 (8)	0.0396 (9)	0.0031 (9)
C15	0.0662 (10)	0.0745 (12)	0.0702 (11)	0.0334 (9)	0.0348 (9)	−0.0027 (9)
N11	0.0396 (5)	0.0555 (7)	0.0479 (6)	0.0256 (5)	0.0235 (5)	0.0131 (5)
N20	0.0921 (11)	0.0828 (10)	0.0545 (8)	0.0558 (9)	0.0451 (8)	0.0220 (7)
C20	0.0919 (13)	0.0714 (11)	0.0511 (9)	0.0488 (10)	0.0364 (9)	0.0185 (8)
C21	0.0838 (12)	0.0782 (12)	0.0593 (10)	0.0441 (10)	0.0434 (9)	0.0228 (9)
C24	0.141 (2)	0.0768 (15)	0.0808 (16)	0.0478 (16)	0.0543 (17)	0.0092 (12)
O1	0.0550 (6)	0.0824 (8)	0.0515 (6)	0.0436 (6)	0.0317 (5)	0.0194 (5)

Geometric parameters (Å, º)

Co1—N1^{i}	2.0812 (15)	C14—H14A	0.9600
Co1—N1	2.0812 (15)	C14—H14B	0.9600
Co1—O1	2.0930 (12)	C14—H14C	0.9600
Co1—O1^{i}	2.0930 (12)	C15—H15A	0.9600
Co1—N11	2.2460 (11)	C15—H15B	0.9600
Co1—N11^{i}	2.2460 (11)	C15—H15C	0.9600
N1—C1	1.158 (2)	N20—C21^{ii}	1.322 (3)
C1—S1	1.6232 (18)	N20—C20	1.330 (3)
N10—C10	1.3321 (19)	C20—C21	1.389 (2)
N10—C13	1.336 (2)	C20—C24	1.493 (3)
C10—C11	1.3935 (18)	C21—N20^{ii}	1.322 (3)
C10—C14	1.495 (2)	C21—H21	0.9300
C11—N11	1.3343 (18)	C24—H24A	0.9600
C11—H11	0.9300	C24—H24B	0.9600
C12—N11	1.3342 (18)	C24—H24C	0.9600
C12—C13	1.3893 (18)	O1—H1O1	0.8201
C12—H12	0.9300	O1—H2O1	0.8200
C13—C15	1.501 (2)		
N1^{i}—Co1—N1	180.00 (9)	C10—C14—H14B	109.5
N1^{i}—Co1—O1	89.38 (6)	H14A—C14—H14B	109.5
N1—Co1—O1	90.62 (6)	C10—C14—H14C	109.5
N1^{i}—Co1—O1^{i}	90.62 (6)	H14A—C14—H14C	109.5
N1—Co1—O1^{i}	89.38 (6)	H14B—C14—H14C	109.5
O1—Co1—O1^{i}	180.0	C13—C15—H15A	109.5

N1^{i}—Co1—N11	88.99 (5)	C13—C15—H15B	109.5
N1—Co1—N11	91.01 (5)	H15A—C15—H15B	109.5
O1—Co1—N11	92.08 (4)	C13—C15—H15C	109.5
O1^{i}—Co1—N11	87.92 (4)	H15A—C15—H15C	109.5
N1^{i}—Co1—N11^{i}	91.01 (5)	H15B—C15—H15C	109.5
N1—Co1—N11^{i}	88.99 (5)	C12—N11—C11	116.38 (12)
O1—Co1—N11^{i}	87.92 (4)	C12—N11—Co1	123.72 (10)
O1^{i}—Co1—N11^{i}	92.08 (4)	C11—N11—Co1	119.73 (9)
N11—Co1—N11^{i}	180.0	C21^{ii}—N20—C20	118.15 (15)
C1—N1—Co1	162.42 (13)	N20—C20—C21	119.60 (19)
N1—C1—S1	177.21 (15)	N20—C20—C24	118.75 (18)
C10—N10—C13	118.09 (12)	C21—C20—C24	121.6 (2)
N10—C10—C11	120.65 (14)	N20^{ii}—C21—C20	122.25 (19)
N10—C10—C14	117.78 (13)	N20^{ii}—C21—H21	118.9
C11—C10—C14	121.56 (15)	C20—C21—H21	118.9
N11—C11—C10	122.01 (13)	C20—C24—H24A	109.5
N11—C11—H11	119.0	C20—C24—H24B	109.5
C10—C11—H11	119.0	H24A—C24—H24B	109.5
N11—C12—C13	122.41 (14)	C20—C24—H24C	109.5
N11—C12—H12	118.8	H24A—C24—H24C	109.5
C13—C12—H12	118.8	H24B—C24—H24C	109.5
N10—C13—C12	120.37 (14)	Co1—O1—H1O1	119.4
N10—C13—C15	117.87 (13)	Co1—O1—H2O1	120.2
C12—C13—C15	121.76 (15)	H1O1—O1—H2O1	105.4
C10—C14—H14A	109.5		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H2*O*1···N20	0.82	2.01	2.8193 (17)	172
O1—H1*O*1···N10^{iii}	0.82	2.01	2.8257 (15)	173

Symmetry code: (iii) −*x*, −*y*+1, −*z*+1.