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Key indicators

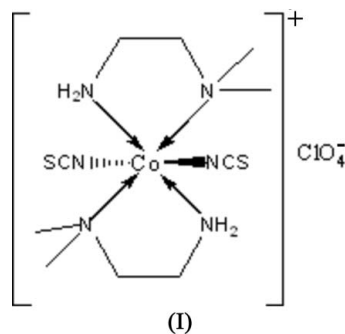
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{N}-\text{C}) = 0.006\text{ \AA}$
Disorder in main residue
 R factor = 0.052
 wR factor = 0.138
Data-to-parameter ratio = 14.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.Bis(*N,N*-dimethylethane-1,2-diamine)-
dithiocyanatocobalt(III) perchlorate:
a redetermination

The crystal structure of the title compound, $[\text{Co}(\text{NCS})_2(\text{C}_4\text{H}_{12}\text{N}_2)_2]\text{ClO}_4$, has been reported previously by Eminger, Fallab, Zehnder & Dobler [*Helv. Chim. Acta* (1992), **75**, 1245–1250], with an R value of 0.074. However, no coordinates are available. We present here a redetermination of this structure using new intensity data. The Co^{III} atom, lying on an inversion centre, is six-coordinate in an octahedral geometry. The perchlorate anion, located on a twofold axis, is disordered over two orientations.

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Comment

The crystal structure of the title compound, (I), has previously been reported by Eminger *et al.* (1992), with an R value of 0.074; however, no coordinates are available in the Cambridge Structural Database (CSD; Allen, 2002). We report here a redetermination of (I) using new intensity data.



Complex (I) is a perchlorate salt of a mononuclear cobalt(III) compound (Fig. 1). The asymmetric unit contains one half of a $[\text{Co}(\text{NCS})_2(\text{C}_4\text{H}_{12}\text{N}_2)_2]^+$ cation and half of a perchlorate anion. The cation is located on a center of inversion and the anion is disordered over a twofold axis. The Co^{III} atom is six-coordinated by four N atoms from two *N,N*-dimethylethane-1,2-diamine ligands and by two N atoms from two isothiocyanate ligands. All the bond lengths and angles in (I) (Table 1) are comparable with those observed in the previous determination (Eminger *et al.*, 1992).

In the crystal structure, the molecules are linked *via* intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{S}$ hydrogen bonds (Table 2), forming a three-dimensional framework (Fig. 2).

Experimental

N,N-Dimethylethane-1,2-diamine (17.6 mg, 0.2 mmol), ammonium thiocyanate (15.2 mg, 0.2 mmol) and $\text{Co}(\text{ClO}_4)_3\cdot 7\text{H}_2\text{O}$ (48.3 mg, 0.1 mmol) were dissolved in methanol (20 ml). The mixture was stirred for 1 h at room temperature. After allowing the filtrate to

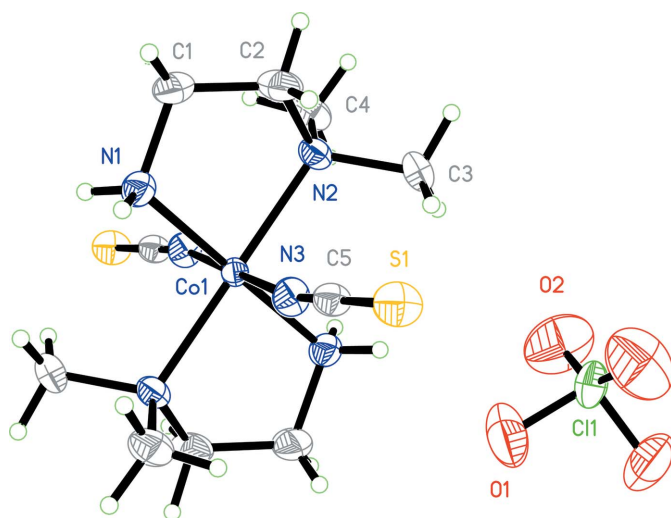


Figure 1

The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Only the major components of the disordered groups are shown. Unlabelled atoms are related to the labelled atoms by the symmetry code ($\frac{1}{2} - x, \frac{1}{2} - y, -z$) in the cation and by $(-x, y, \frac{1}{2} - z)$ in the anion.

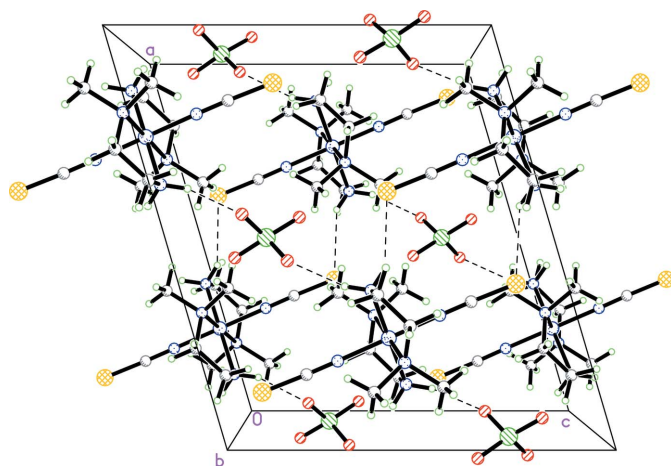


Figure 2

The crystal packing of (I), viewed along the *b* axis. Hydrogen bonds are shown as dashed lines. Only the major disorder components are shown.

stand in air for 15 d, brown block-shaped crystals, suitable for X-ray single-crystal structure determination, formed at the bottom of the vessel on slow evaporation of the solvent. Analysis found: C 26.50, H 5.43, N 18.72%; calculated for $C_{10}H_{24}ClCoN_6O_4S_2$: C 26.64, H 5.37, N 18.64%.

Crystal data

$[Co(NCS)_2(C_4H_{12}N_2)_2]ClO_4$
 $M_r = 450.85$
 Monoclinic, $C2/c$
 $a = 14.075$ (3) Å
 $b = 11.481$ (3) Å
 $c = 12.366$ (3) Å
 $\beta = 106.380$ (3)°
 $V = 1917.2$ (8) Å³
 $Z = 4$

$D_x = 1.562$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 1131 reflections
 $\theta = 2.3$ – 20.4 °
 $\mu = 1.28$ mm⁻¹
 $T = 298$ (2) K
 Block, brown
 $0.11 \times 0.11 \times 0.07$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.872$, $T_{\max} = 0.916$
 8063 measured reflections

2199 independent reflections
 1505 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.046$
 $\theta_{\max} = 27.5$ °
 $h = -18 \rightarrow 18$
 $k = -14 \rightarrow 14$
 $l = -16 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.138$
 $S = 1.03$
 2199 reflections
 150 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 3.9721P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.55$ e Å⁻³
 $\Delta\rho_{\min} = -0.36$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Co1—N1	1.946 (3)	Co1—N3	1.883 (4)
Co1—N2	2.055 (3)		
N1—Co1—N1 ⁱ	180	N3—Co1—N2	89.46 (13)
N2—Co1—N2 ⁱ	180	N3—Co1—N1 ⁱ	91.00 (14)
N3 ⁱ —Co1—N3	180	N3 ⁱ —Co1—N2	90.54 (13)
N1—Co1—N2	86.03 (13)	N1—Co1—N2 ⁱ	93.97 (13)
N3—Co1—N1	89.00 (14)		

Symmetry code: (i) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1B \cdots S1 ⁱⁱ	0.90	2.64	3.439 (3)	148
N1—H1A \cdots O1 ⁱ	0.90	2.34	3.217 (18)	165
N1—H1A \cdots O1 ⁱ	0.90	2.21	2.982 (8)	143

Symmetry codes: (i) $-x + \frac{1}{2}, -y + \frac{1}{2}, -z$; (ii) $-x + 1, y, -z + \frac{1}{2}$.

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.96 or 0.97 Å and N—H distances of 0.90 Å, and with $U_{\text{iso}}(\text{H}) = 1.2$ or 1.5 (methyl) times $U_{\text{eq}}(\text{C}, \text{N})$. The O atoms of the perchlorate anion are disordered over two distinct sites, with occupancies of 0.695 (3) and 0.305 (3). The Cl—O and O \cdots O distances in the disordered perchlorate anion were restrained to be equal. The C1—C2—N2—C3—C4 fragment is also disordered over two distinct sites, with occupancies of 0.637 (3) and 0.363 (3).

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1998); software used to prepare material for publication: SHELXTL.

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