

C19—Sn—C25	123.1 (2)	O1—Sn—Cl2	82.30 (11)
C19—Sn—O1	86.6 (3)	C19—Sn—Cl1	94.9 (2)
C25—Sn—O1	88.6 (2)	C25—Sn—Cl1	96.9 (2)
C19—Sn—Cl2	117.6 (2)	O1—Sn—Cl1	172.27 (10)
C25—Sn—Cl2	117.8 (2)	Cl2—Sn—Cl1	90.31 (6)

The title structure was solved by direct methods and refined by full-matrix least-squares techniques. All non-water H atoms were located from a difference Fourier map, idealized and allowed to ride on their carrier atoms. The maximum and minimum ΔF extremes were found at 0.97 and 1.04 Å, respectively, from the Sn atom. The poor agreement between expected and reported transmission-factor ranges is partly attributed to the absence of suitably strong reflections near $\chi = 90^\circ$ which could be utilized for ψ -scan measurements.

Data collection: XSCANS (Siemens, 1994). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXTL/PC (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC. Software used to prepare material for publication: SHELXL93 and PARST (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1258). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 22–24

Bis(3,5-dimethylpyrazole-*N*²)bis(thiocyanato-*N*)cobalt(II) monohydrate

XIAO-FENG CHEN,^a SHU-HUA LIU,^a XIAO-ZENG YOU,^a HOONG-KUN FUN,^b KANDASAMY CHINNAKALI^{b†} AND IBRAHIM ABDUL RAZAK^b

^aCoordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China, and ^bX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: hkfun@usm.my

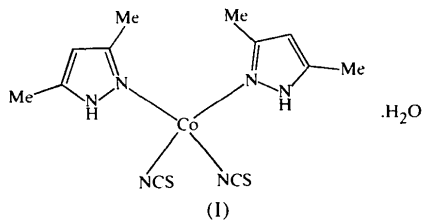
(Received 18 May 1998; accepted 24 July 1998)

Abstract

In the title compound, [Co(NCS)₂(C₅H₈N₂)₂]·H₂O, the metal atom exhibits an approximately tetrahedral configuration. The Co—N bond distances and N—Co—N bond angles are in the ranges 1.936(3)–2.010(3) Å and 101.6(1)–113.7(2)°, respectively. The water molecule is involved in O—H···S and N—H···O hydrogen bonds, which result in the formation of an infinite chain parallel to the *c* axis.

Comment

Extensive investigations have been carried out on imidazole derivatives of cobalt(II) complexes containing thiocyanate (Maslejovala *et al.*, 1997). In order to investigate the influence of steric properties on the stoichiometry as well as on the stereochemistry of the resulting species, some complexes of the isomeric pyrazoles have been studied. Although the title complex, (I), has been prepared previously (Anagnostopoulos, 1976) and some of its physical properties described (Bagley *et al.*, 1970), its crystal structure has not, to our knowledge, been reported before now.



The Co atom is tetrahedrally coordinated by four N atoms, two from the monodentate thiocyanate groups and the others from the two 3,5-dimethylpyrazole ligands, building up the CoN₄ chromophore. The

† On leave from: Department of Physics, Anna University, Chennai 600 025, India.

Co—N_{pyrazole} bonds [Co—N1 2.000 (3) and Co—N3 2.010 (3) Å] are longer than the Co—N_{thiocyanate} bonds [Co—N5 1.936 (3) and Co—N6 1.942 (3) Å]. The dimethylpyrazole moiety is planar and the dihedral angle between the adjacent pyrazole planes is 80.6 (1)°. The NCS groups are almost linear, but significant bending is observed in the Co—N—C(—S) linkages [Co—N5—C11 173.6 (3) and Co—N6—C12 171.8 (3)°]. In the solid state, molecules along the *c* direction are linked by water molecules, through O—H...S and N—H...O hydrogen bonds involving the thiocyanate and pyrazole groups, respectively, to form an infinite chain. Due to the N—H...O hydrogen bonds, the O atom acquires full tetracoordination.

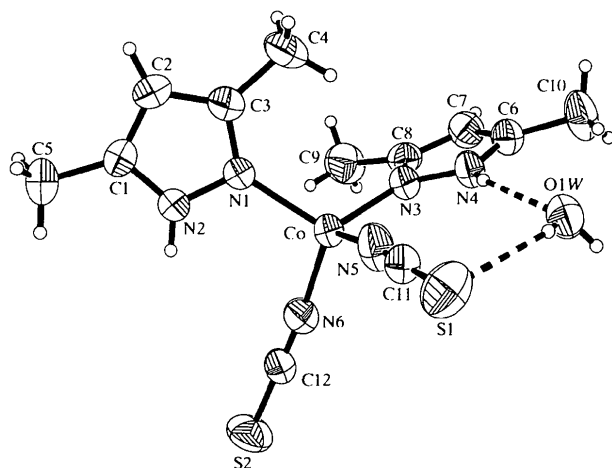


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

The title complex was prepared by adding a methanolic solution of 3,5-dimethylpyrazole to an aqueous solution containing Co(NO₃)₂·6H₂O and KSCN under continuous stirring. The product separated immediately as polycrystalline products, which were filtered off. Violet single crystals were obtained from the mother liquor by slow evaporation at room temperature.

Crystal data

[Co(NCS)₂(C₅H₈N₂)₂]·H₂O

M_r = 385.37

Monoclinic

*P*2₁/*c*

a = 8.0204 (3) Å

b = 27.1881 (11) Å

c = 8.9420 (4) Å

β = 106.332 (1)°

V = 1871.21 (13) Å³

Z = 4

D_x = 1.368 Mg m⁻³

D_m not measured

Mo *K*α radiation

λ = 0.71073 Å

Cell parameters from 5695 reflections

θ = 1.37–33.22°

μ = 1.149 mm⁻¹

T = 293 (2) K

Block

0.66 × 0.58 × 0.48 mm

Violet

Data collection

Siemens SMART CCD area-detector diffractometer

ω scans

Absorption correction:

empirical (SADABS;

Sheldrick, 1996)

T_{min} = 0.442, *T_{max}* = 0.576

12 132 measured reflections

4286 independent reflections

3901 reflections with

I > 2σ(*I*)

R_{int} = 0.033

θ_{max} = 27.5°

h = −10 → 10

k = 0 → 35

l = 0 → 11

Refinement

Refinement on *F*²

R[*F*² > 2σ(*F*²)] = 0.059

wR(*F*²) = 0.135

S = 1.264

4286 reflections

224 parameters

H atoms: see below

w = 1/[σ²(*F_o*²) + (0.0309*P*)²

+ 2.2531*P*]

where *P* = (*F_o*² + 2*F_c*²)/3

(Δ/σ)_{max} < 0.001

Δρ_{max} = 0.394 e Å⁻³

Δρ_{min} = −0.428 e Å⁻³

Extinction correction:

SHELXTL

Extinction coefficient:

0.0021 (13)

Scattering factors from

International Tables for

Crystallography (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Co—N5	1.936 (3)	N1—N2	1.361 (4)
Co—N6	1.942 (3)	N2—C1	1.333 (4)
Co—N1	2.000 (3)	N3—C8	1.334 (5)
Co—N3	2.010 (3)	N3—N4	1.359 (4)
S1—C11	1.612 (4)	N4—C6	1.337 (5)
S2—C12	1.620 (4)	N5—C11	1.153 (5)
N1—C3	1.333 (5)	N6—C12	1.156 (5)
N5—Co—N6	113.7 (2)	N5—Co—N3	101.61 (13)
N5—Co—N1	112.21 (14)	N6—Co—N3	111.41 (13)
N6—Co—N1	104.75 (13)	N1—Co—N3	113.44 (12)

Table 2. Hydrogen-bonding geometry (Å, °)

<i>D</i> — <i>H</i> ... <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> — <i>H</i> ... <i>A</i>
N2—H1N2...O1W ⁱ	0.86 (5)	2.19 (4)	2.979 (5)	152 (4)
N4—H1N4...O1W	0.76 (4)	2.22 (4)	2.944 (6)	159 (4)
O1W—H1O1...S1	0.77 (6)	2.67 (6)	3.417 (5)	163 (6)
O1W—H2O1...S2 ⁱⁱ	0.86 (8)	2.50 (8)	3.363 (5)	176 (7)

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

The data collection covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm (2θ_{max} = 66.2°) and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible. Only reflections with 2θ less than 55° were used for the structure refinement. The structure was solved by direct methods and refined by full-matrix least-squares techniques. The methyl H atoms were geometrically fixed and allowed to ride on the parent C atoms; the remaining six H atoms were located from a difference Fourier map and refined isotropically.

Data collection: SMART (Siemens, 1996b). Cell refinement: SAINT (Siemens, 1996b). Data reduction: SAINT. Program(s) used to solve structure: SHELXTL (Siemens, 1996a). Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL and PARST (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1228). Services for accessing these data are described at the back of the journal.

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Bis(triethanolamine)strontium(II) bis(2,4-dinitrophenolate)

NARINDER SINGH POONIA,^a NEERA CHHABRA,^a W. S. SHELDRIK,^b GEETA HUNDAL,^c SANGEETA OBRAI^c AND MANINDER SINGH HUNDAL^c

^aDepartment of Chemistry, Devi Ahilya University, Khandwa Road, Indore, India, ^bLehrstuhl für Analytische Chemie, Ruhr-Universität Bochum, D-44780 Bochum, Germany, and ^cDepartment of Chemistry, Guru Nanak Dev University, Amritsar 143 005, India. E-mail: gndu@rocketmail.com

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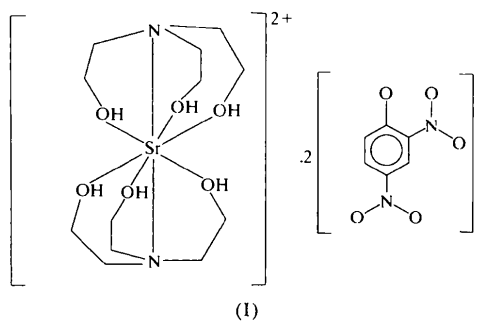
Abstract

In the title compound, bis(2,2',2''-nitrotriethanol-*N,O,O',O'',O'''*) strontium(II) bis(2,4-dinitrophenolate), [Sr(C₆H₁₅NO₃)₂](C₆H₃N₂O₅)₂, the coordination number around the cation is eight, and it is effectively sandwiched by the two tetradentate triethanolamine ligands, which coordinate through all three hydroxyl O atoms and the amine N atom. The Sr—O distances are in the range 2.574 (7)–2.586 (6) Å, and the Sr—N distance is 2.777 (7) Å. The two 2,4-dinitrophenolate anions are charge separated in the lattice. The shortest Sr···O

distance to the 2,4-dinitrophenolate anions is Sr···O1 [4.268 (8) Å].

Comment

The role of 'double-action' ligands, such as triethanolamine, is to stabilize the cation by coordination and the anion by hydrogen bonding (Poonia & Bajaj, 1979; Bajaj & Poonia, 1988), which results in a reduction in the nucleophilic properties of the latter. The title complex, (I), has been studied to investigate the role of hydrogen bonding in complexes of the type *M*(DNP)₂(TEA)_{*n*}, where *M* = Ca²⁺, Sr²⁺ or Ba²⁺, DNP = 2,4-dinitrophenolate and TEA = triethanolamine. The structures of Ca(DNP)₂(TEA) and Ba(DNP)₂(TEA)₂ have already been reported (Hundal *et al.*, 1995; Kanters *et al.*, 1984). It appears that hydrogen bonding is a dominant factor in decreasing cation–anion interactions in the above series. The Sr²⁺ ion is present on a crystallographic centre of symmetry and is eightfold coordinated by two TEA molecules. The coordination around Sr²⁺ is bicapped trigonal antiprismatic. Both of the DNP counter-anions are excluded from the coordination cluster, resulting in the formation of a charge-separated complex. Important bond distances and angles are given in Table 1. The Sr—O distances are in the range 2.574 (7)–2.586 (6) Å, and the Sr—N1 distance is 2.777 (7) Å. The N—C—O torsion angles [−51.3 (13) to −54.6 (13)°] are comparable to the values of 46.8 (4) and 67.4 (4)° reported earlier (Hundal *et al.*, 1995, 1996; Kanters *et al.*, 1984), producing nearly *gauche* conformations of the terminal OH groups with respect to the C—N bonds. There are three nearly planar C—N—C—C fragments with torsion angles ranging from 160.2 (9) to 169.5 (8)°, which are comparable to the values found in the literature.



In the case of (I), the counter-anion does not interact with the cation but it is strongly hydrogen bonded to the TEA ligands. The phenolic O atom forms two hydrogen bonds with the hydroxyl O atoms; it has an intramolecular hydrogen bond [O7···O1 2.70 (1) Å] and is also attached to O8 of the centrosymmetrically related molecule, forming an intermolecular bond [O1···O8 2.65 (1) Å]. Thus, the phenolic O atom acts as a double hydrogen-bond acceptor towards two hydroxyl groups,