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Tris(pyrrolidine-1-carbodithioato-κ^2S,S')-cobalt(III) chloroform disolvate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.007 Å
Disorder in solvent or counterion
R factor = 0.059
wR factor = 0.140
Data-to-parameter ratio = 23.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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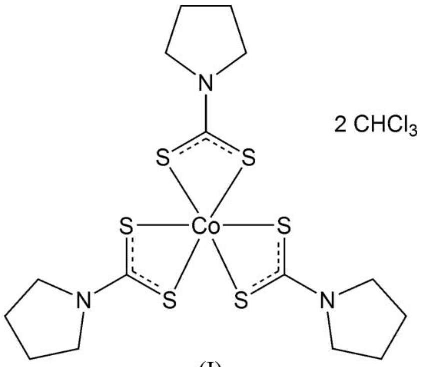
The title complex, $[Co\{S_2CN(CH_2)_4\}_3]\cdot 2CHCl_3$, contains Co^{III} octahedrally coordinated by three bidentate dithiocarbamate ligands. Chloroform molecules form C—H⋯S interactions with the complex.

Comment

N,N-disubstituted dithiocarbamates (dtc) are amongst the most frequently used bidentate sulfur ligands. There are more than 1500 compounds with at least one such dtc group in the Cambridge Structural Database (Version 5.28; Allen, 2002). Amongst these, the number of structurally characterized $M(dtc)_3$ species is surprisingly small. Likewise, amongst approximately 70 structurally characterised cobalt dithiocarbamates, there are very few homoleptic $Co(dtc)_3$ complexes. Here we describe the structure of the title compound, (I), which crystallizes as a chloroform disolvate. The structure of the unsolvated parent compound, $[Co(S_2CN(CH_2)_4)_3]$, has been reported previously (Healy *et al.* 1990).



(I)

The molecular structure of (I) is shown in Fig. 1. Apart from slightly longer Co—S bonds (by *ca.* 0.02 Å), there are no significant differences compared to the unsolvated compound (Healy *et al.* 1990). The most notable feature of (I) is the location of the two chloroform molecules, both of which have their C—H bonds directed between the S atoms of two different pairs of dtc ligands (Fig. 1), forming C—H⋯S interactions (Table 2). A similar arrangement of chloroform molecules is present in the closely comparable compound $[Cr\{S_2CN(CH_2)_5\}_3]\cdot 2CHCl_3$ (Kettmann *et al.* 1981).

Experimental

$CoCl_2\cdot 6H_2O$ (0.595 g, 0.0025 mol) dissolved in 50 ml methanol/water (10/1 *v*/*v*) was added dropwise to the ammonium salt of pyrrolidinecarbodithioic acid ($C_4H_9NCS_2NH_4$, 0.82 g, 0.005 mol) dissolved in methanol/water (50 ml, 10:1 *v*/*v*). The mixture was stirred vigorously under an argon atmosphere for 20 min, then filtered and left to stand at 278 K. Green crystals of (I) were collected after approximately 2 weeks.

Crystal data

$[Co(C_5H_8NS_2)_3]\cdot 2CHCl_3$
$M_r = 736.40$
Monoclinic, $P2_1/c$
$a = 13.879$ (2) Å
$b = 10.341$ (2) Å
$c = 22.406$ (4) Å
$\beta = 101.80$ (1)°
$V = 3147.8$ (10) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 1.47$ mm^{-1}
$T = 295$ (2) K
0.32 × 0.20 × 0.14 mm

Data collection

Kuma KM-4 CCD diffractometer
Absorption correction: numerical (*CrysAlis RED*; Oxford Diffraction, 2004)
$T_{min} = 0.645$, $T_{max} = 0.812$
39457 measured reflections
7579 independent reflections
3924 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.140$
$S = 1.12$
7579 reflections
325 parameters
18 restraints
H-atom parameters constrained
$\Delta\rho_{max} = 0.68$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Co—S31	2.2638 (12)	Co—S32	2.2882 (12)
Co—S12	2.2733 (12)	Co—S11	2.2893 (11)
Co—S21	2.2796 (12)	Co—S22	2.2921 (12)
S31—Co—S12	95.27 (4)	S21—Co—S11	94.64 (4)
S31—Co—S21	168.36 (4)	S32—Co—S11	166.03 (4)
S12—Co—S21	94.05 (4)	S31—Co—S22	95.18 (5)
S31—Co—S32	76.50 (4)	S12—Co—S22	167.34 (4)
S12—Co—S32	93.55 (4)	S21—Co—S22	76.55 (4)
S21—Co—S32	96.00 (4)	S32—Co—S22	95.84 (4)
S31—Co—S11	94.27 (4)	S11—Co—S22	95.41 (4)
S12—Co—S11	76.68 (4)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C36—H36···S12	0.98	2.81	3.696 (4)	152
C36—H36···S31	0.98	3.18	3.970 (5)	139
C37—H37···S22	1.00	3.02	3.748 (3)	132
C37—H37···S32	1.00	2.72	3.538 (3)	140

H atoms were positioned geometrically with C—H = 0.97 Å (CH_2) or 0.98 Å ($CHCl_3$), and treated as riding with $U_{iso}(H) = 1.5U_{eq}(C)$. One chloroform molecule is disordered over two orientations related by rotation about its C—H bond, with site occupancy factors of 0.5 for the Cl atoms.

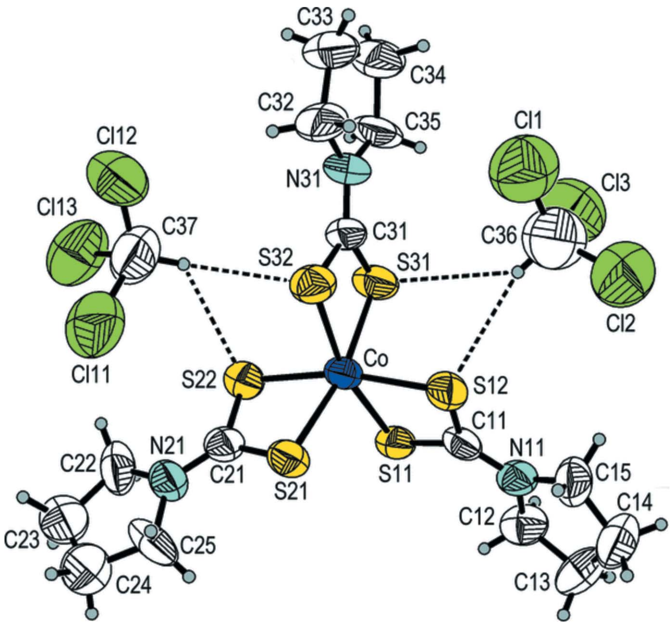


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level for non-H atoms. One chloroform molecule [C37/Cl11/Cl12/Cl13] is disordered; only one disorder component is shown. Dashed lines denote C—H···S interactions.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2004); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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