metal-organic papers

Bis(tri-*tert*-butoxysilanethiolato-κS)-bis(pyrrolidine-κN)cobalt(II)

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.039
wR factor = 0.097
Data-to-parameter ratio = 16.6

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

The reaction of $[Co\{SSi(O^tBu)_3\}_2(NH_3)]_2$ with pyrrolidine leads to the formation of the title silanethiolate complex, $[Co(C_{12}H_{27}O_3SSi)_2(C_4H_9N)_2]$, where the Co^{II} ion is coordinated by the two S atoms derived from the silanethiolate residues and the two N atoms of the pyrrolidine residues. The specific spatial arrangement of the pyrrolidine residues allows them to form two intramolecular N—H$\cdots$O hydrogen bonds.

Comment

The chemistry of metal–thiolate complexes has continuously been the focus of wide interest. Transition metal complexes have been applied to protein purification technology, based on the affinity between histidine residues and immobilized metal ions chelated by reactive groups and covalently attached to solid supports. This method is known as immobilized metal ion affinity chromatography (IMAC). Transition metal thiolates have been found in biological systems, especially in the active sites of metalloproteins, where metals are bonded to cysteine and histidine. Because of the similarity between the coordination chemistry of cobalt and zinc and the 'spectroscopic silence' of Zn, cobalt thiolates have been used as models to simulate the active sites of zinc metalloproteins (Corwin *et al.*, 1987; Kimblin *et al.*, 2000). For many years, we have been interested in the chemistry of silanethiols and metal silanethiolates (Wojnowski *et al.*, 1985; Becker *et al.*, 1995; Peters *et al.*, 1998; Chojnacki *et al.*, 2001; Baranowska *et al.*, 2002; Dołęga *et al.*, 2004). We present here the crystal structure of the title cobalt silanethiolate complex, (I).

(I)
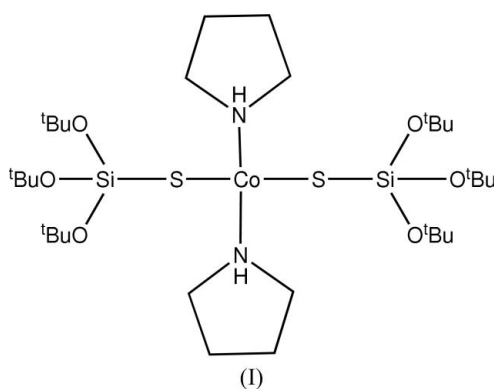
Compound (I) (Fig. 1), with a CoN_2S_2 core and tetrahedrally coordinated cobalt(II), adopts a similar spatial arrangement to heteroleptic cobalt silanethiolates previously obtained and described by us (Becker *et al.*, 2001*a*,*b*). Additional intramolecular hydrogen bonds between siloxy O atoms and pyrrolidine N atoms are formed, and the respective N$\cdots$O
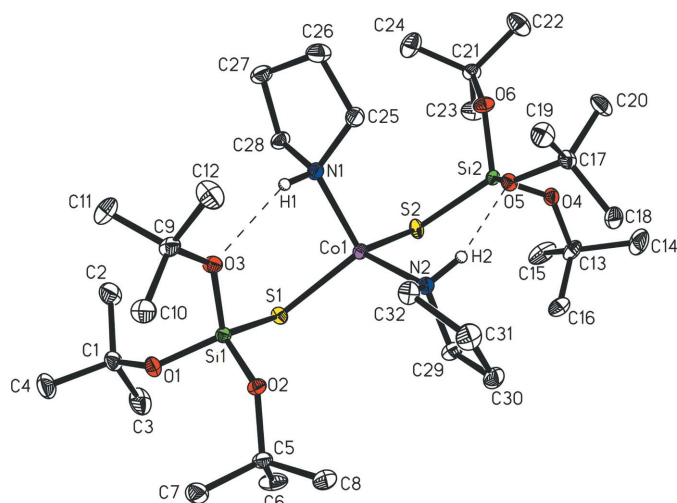
Figure 1
The molecular structure of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. All H atoms bonded to C atoms have been omitted. Dashed lines indicate the intramolecular hydrogen bonds. The minor disorder component has been omitted.

distances are shorter than those observed for [Co{SSi-(O^{t}Bu)$_3$}$_2$(NH$_3$)(2-Pic)] and [Co{SSi(O^{t}Bu)$_3$}$_2$(NH$_3$)$_2$]MeCN (Becker *et al.*, 2002). The angles and Co—S, Co—N, S—Si and Si—O bond lengths differ slightly but are still comparable with those found in other cobalt thiolates and silanethiolates (Corwin *et al.*, 1987; Becker *et al.*, 2001*a,b*).

Experimental

[Co{SSi(O^{t}Bu)$_3$}$_2$(NH$_3$)]$_2$ was obtained as described previously by Becker *et al.* (2001*a*). All other reagents were obtained commercially. Pyrrolidine was dried by standard methods and distilled prior to use. To a solution of [Co{SSi(O^{t}Bu)$_3$}$_2$(NH$_3$)]$_2$ (0.124 g, 0.2 mmol) in *n*-hexane (5 ml), freshly prepared pyrrolidine (33.4 µl, 0.4 mmol) was added. Blue well formed crystals of [Co{SSi(^{t}BuO)$_3$}$_2$]$_2$-(pyrrolidine)$_2$], (I), were obtained from the mixture after one month.

Crystal data

[Co(C$_{12}$H$_{27}$O$_3$SSi)$_2$(C$_4$H$_9$N)$_2$]	$Z = 4$
$M_r = 760.15$	$D_x = 1.183$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 16.3249$ (6) Å	$\mu = 0.59$ mm^{-1}
$b = 15.2868$ (5) Å	$T = 120$ (2) K
$c = 17.3137$ (6) Å	Prism, blue
$\beta = 99.033$ (3)°	$0.25 \times 0.17 \times 0.14$ mm
$V = 4267.1$ (3) Å^3	

Data collection

Oxford Diffraction KM-4 CCD area-detector diffractometer	25897 measured reflections
ω scans	7559 independent reflections
Absorption correction: numerical [*CrysAlis RED* (Oxford Diffraction, 2005); analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1998)]	7025 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.025$
	$\theta_{max} = 25.1°$
$T_{min} = 0.812$, $T_{max} = 0.870$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.052P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$+ 3.2675P]$
$wR(F^2) = 0.097$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
7559 reflections	$\Delta\rho_{max} = 0.74$ e Å^{-3}
455 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

N1—Co1	2.0429 (16)	Si2—S2	2.0826 (7)
N2—Co1	2.0375 (16)	S1—Co1	2.2969 (5)
Si1—S1	2.0797 (7)	S2—Co1	2.3052 (5)
Si1—S1—Co1	105.00 (2)	N1—Co1—S1	103.93 (5)
Si2—S2—Co1	107.69 (2)	N2—Co1—S2	105.83 (5)
N2—Co1—N1	108.69 (6)	N1—Co1—S2	111.55 (5)
N2—Co1—S1	117.68 (5)	S1—Co1—S2	109.257 (19)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O3	0.93	2.22	3.033 (2)	145
N2—H2⋯O5	0.93	2.13	2.964 (2)	150

All H atoms were refined as riding in geometrically idealized positions, with C—H = 0.98–0.99 Å and N—H = 0.93 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(C) for CH$_3$ groups. The disordered *tert*-butyl group C10–C12 has site-occupancy factors of 0.681 (8) and 0.319 (8).

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); 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: *WinGX* (Farrugia, 1999).

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