Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

(Tetrahydrofuran-κ*O*)bis(tri-*tert*-butoxysilanethiolato-κ^2*O*,*S*)chromium(II)

Anna Ciborska, Katarzyna Baranowska* and Wiesław Wojnowski

Department of Chemistry, Technical University of Gdańsk, 11/12 G. Narutowicz St. 80952, PL Gdańsk, Poland

Correspondence e-mail: kasiab29@wp.pl

Received 21 March 2007
Accepted 23 March 2007

The title compound, [Cr($C_{12}H_{27}O_3SSi$)$_2$(C_4H_8O)], is the first silanethiolate complex of chromium(II). The Cr atom is coordinated by two *O*,*S*-chelating tri-*tert*-butoxysilanethiolate ligands and contains, as an additional ligand, a molecule of tetrahydrofuran. The coordination geometry is square-pyramidal in which the tetrahydrofuran molecule occupies the apical position. The molecule lies on a crystallographic twofold rotation axis.

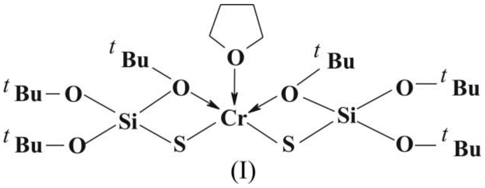
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
R factor = 0.051
wR factor = 0.116
Data-to-parameter ratio = 16.9

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

Comment

Tri-*tert*-butoxysilanethiol has been widely utilized in our studies on the chemistry of derivatives of monothioorthosilicic acid (Piękoś & Wojnowski, 1962) as a source of the unique (^{t}BuO)$_3$SiS group as a structure-forming factor. Metal tri-*tert*-butoxysilanethiolates form a variety of interesting solid-state structures, monomers and dimers as well as more complex systems (Wojnowski, Peters *et al.*, 1985; Becker *et al.*, 1992; Jesionka *et al.*, 2005). The complexes are also used in model studies on structural and catalytic metal centers in proteins (Becker *et al.*, 2002; Dołęga *et al.*, 2004, 2005).



(I)

We present here the structure of the title compound, (I), the first silanethiolate complex of chromium(II). The reaction of anhydrous chromium(II) chloride with sodium tri-*tert*-butoxysilanethiolate leads to the formation of the five-coordinate complex. The Cr atom is coordinated by two S, two O atoms of the two butoxy groups and the O atom of tetrahydrofuran (Fig. 1, Fig. 2). The coordination geometry is square-pyramidal in which the tetrahydrofuran molecule occupies the apical position. The molecule lies on a crystallographic twofold rotation axis. The tri-*tert*-butoxysilanethiolate ligand was observed in homoleptic mercury and cadmium silanethiolates (Wojnowski, Wojnowska *et al.*, 1985, Wojnowski, Becker *et al.*, 1992) and in heteroleptic zinc, cadmium and cobalt silanethiolates (Becker *et al.*, 1995, 1996, 2001; Dołęga *et al.*, 2005, 2006). In (I), the Cr—S bond lengths are typical of bond lengths found in other Cr-thiolate complexes (Okura *et al.*, 1985; Ito 2002). The *trans* angles of the square base are described by S—Cr—S and O—Cr—O angles. Further research on synthetic routes to tri-*tert*-butoxysilanethiolate complexes of chromium with different ligands will be reported later.

Experimental

Manipulations were conducted under an atmosphere of nitrogen using standard Schlenk techniques. All solvents were purified and dried by standard methods (Perrin & Armarego, 1988). The substrate $(^tBuO)_3SiSNa$ was prepared by literature methods (Piękoś & Wojnowski, 1962; Wojnowska & Wojnowski, 1974). The title compound was obtained by the reaction of $CrCl_2$ with $(^tBuO)_3SiSNa$. A tetrahydrofuran solution (10 ml) of $CrCl_2$ (0.174 g, 1.4 mmol) was added to a toluene solution (10 ml) of $(^tBuO)_3SiSNa$ (0.9 g, 3 mmol) at room temperature. The resulting pale-green solution was refluxed for 24 h. Concentration and cooling (250 K) of this solution gave colorless crystals of (I), suitable for X-ray diffraction analysis.

Crystal data

$[Cr(C_{12}H_{27}O_3SSi)_2(C_4H_8O)]$
$M_r = 683.08$
Orthorhombic, $Pbcn$
$a = 8.7343$ (3) Å
$b = 17.8256$ (7) Å
$c = 25.0266$ (9) Å
$V = 3896.5$ (2) $Å^3$
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.50$ mm^{-1}
$T = 120$ (2) K
0.24 × 0.16 × 0.04 mm

Data collection

Oxford Diffraction KM-4 CCD diffractometer
Absorption correction: numerical *CrysAlis RED*, Oxford Diffraction (2005)
$T_{min} = 0.846$, $T_{max} = 0.947$
14433 measured reflections
3400 independent reflections
3135 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.116$
$S = 1.08$
3400 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.44$ e $Å^{-3}$
$\Delta\rho_{min} = -0.31$ e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

Cr1—O1	2.1364 (19)	O2—Si1	1.621 (2)
Cr1—O4	2.285 (3)	O3—Si1	1.6253 (18)
Cr1—S1	2.4404 (7)	O4—C13	1.442 (3)
O1—Si1	1.6844 (19)	S1—Si1	2.0633 (10)
O3—C9—C10	105.6 (2)	Si1—S1—Cr1	81.35 (3)
O3—C9—C12	111.0 (2)	O2—Si1—O3	106.07 (11)
O3—C9—C11	108.5 (2)	O2—Si1—O1	104.99 (10)
O1—Cr1—O1^i	174.50 (10)	O3—Si1—O1	110.03 (10)
O1—Cr1—O4	92.75 (5)	O2—Si1—S1	116.91 (8)
O4—Cr1—S1	100.38 (2)	O3—Si1—S1	117.89 (8)
S1—Cr1—S1^i	159.25 (4)	O1—Si1—S1	99.99 (7)
Si1—O1—Cr1	100.38 (9)	O4—C13—C14	104.2 (6)

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

All H atoms were refined as riding on C atoms with methyl C—H = 0.98 Å, methylene C—H = 0.99 Å, and $U_{iso}(H) = 1.3U_{eq}(C)$ for CH_2 groups and $1.5U_{eq}(C)$ for methyl groups. Atom C14 in the THF molecule is disordered [0.60 (4)/0.40 (4)].

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).

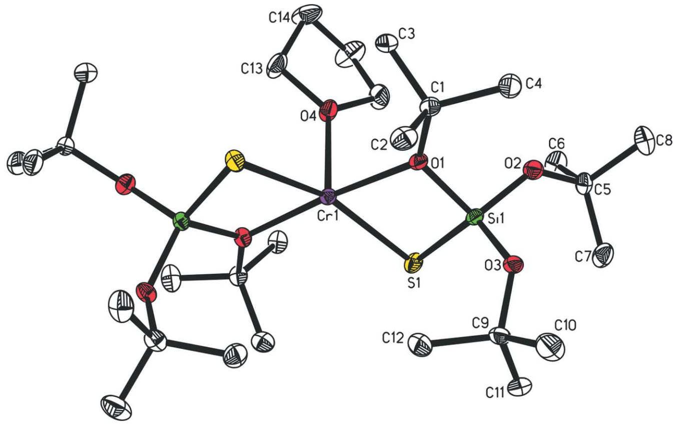


Figure 1
The molecular structure of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted. The molecule lies on a twofold axis that passes through Cr1 and O4, and the unlabeled atoms are related to the labeled ones by $(1 - x, y, \frac{1}{2} - z)$.

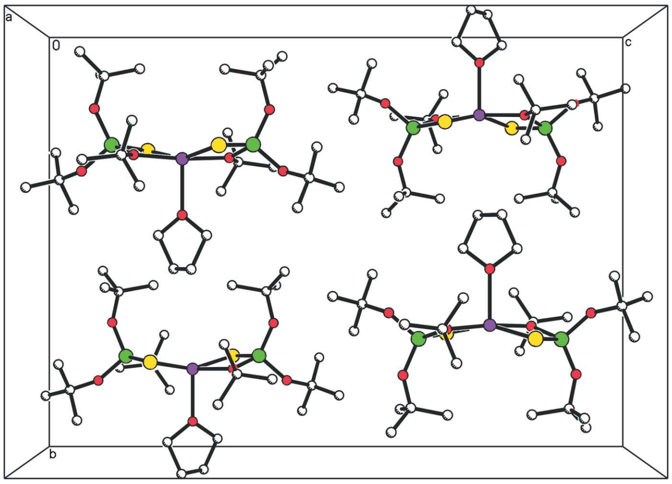


Figure 2
A packing diagram of (I), viewed along the *a* axis. Cr atoms are purple, S atoms are yellow, Si atoms are green, O atoms are red, C atoms are white. H atoms have been omitted.

This work was carried out with financial support from the Polish State Committee grant No. 3 T09A 12028.

References

Becker, B., Pladzyk, A., Konitz, A. & Wojnowski, W. (2002). *Appl. Organomet. Chem.* **16**, 517–524.
Becker, B., Radacki, K., Konitz, A. & Wojnowski, W. (1995). *Z. Anorg. Allg. Chem.* **621**, 904–908.
Becker, B., Radacki, K. & Wojnowski, W. (1996). *J. Organomet. Chem.* **521**, 39–49.
Becker, B., Wojnowski, W., Peters, K., von Peters, E.-M. & von Schnering, H. G. (1992). *Polyhedron*, **6**, 613–616.
Becker, B., Zalewska, A., Konitz, A. & Wojnowski, W. (2001). *Polyhedron*, **20**, 2567–2576.
Dołęga, A., Becker, B., Chojnacki, J., Konitz, A. & Wojnowski, W. (2004). *Inorg. Chim. Acta*, **357**, 461–467.
Dołęga, A., Chojnacki, J., Konitz, A., Komuda, W. & Wojnowski, W. (2006). *Acta Cryst.* E**62**, m636–m639.

m1240 Ciborska *et al.* • $[Cr(C_{12}H_{27}O_3SSi)_2(C_4H_8O)]$ *Acta Cryst.* (2007). E**63**, m1239–m1241

Dołęga, A., Konitz, A., Baum, E. & Wojnowski, W. (2005). *Acta Cryst.* E**61**, m2582–m2584.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Ito, T. (2002). *Acta Cryst.* E**58**, m449–m450.

Jesionka, E., Ciborska, A., Chojnacki, J. & Wojnowski, W. (2005). *Acta Cryst.* C**61**, m321–m323.

Okura, I., Kaji, N., Aono, S., Kita, T. & Yamada, A. (1985). *Inorg. Chem.* **24**, 453–454.

Oxford Diffraction, (2005). *CrysAlis CCD* and *CrysAlis RED*. Version 1.171. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.

Perrin, D. D. & Armarego, W. L. F. (1988). *Purification of Laboratory Chemicals.* Oxford: Pergamon Press.

Piękoś, R. & Wojnowski, W. (1962). *Z. Anorg. Allg. Chem.* **318**, 212–216.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Wojnowska, M. & Wojnowski, W. (1974). *Z. Anorg. Allg. Chem.* **403**, 179–185.

Wojnowski, W., Becker, B., Walz, L., Peters, E.-M. & von Schnering, H. G. (1992). *Polyhedron*, **11**, 607–612.

Wojnowski, W., Peters, K., Peters, E.-M. & von Schnering, H. G. (1985). *Z. Anorg. Allg. Chem.* **531**, 147–152.

Wojnowski, W., Wojnowska, M. & von Schnering, H. G. (1985). *Z. Anorg. Allg. Chem.* **531**, 153–157.

Acta Cryst. (2007). E**63**, m1239–m1241 Ciborska *et al.* • $[Cr(C_{12}H_{27}O_3SSi)_2(C_4H_8O)]$ **m1241**