Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

1,1,1,1,4,4,4,4-Octacarbonyl-2,2,3,3,5,5,6,6-octamethyl-*cyclo*-2,3,5,6-tetraantimony-1,4-dichromium

Hans Joachim Breunig,* Enno Lork, Ovidiu Moldovan and Ciprian I. Raţ

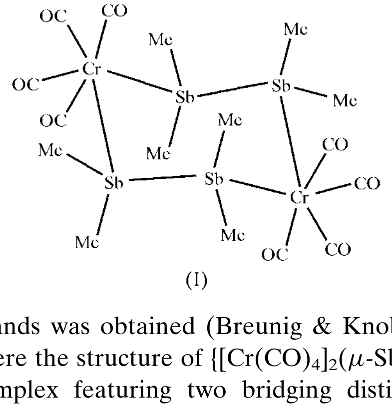
Universität Bremen, Leobener Strasse NW2, 28334 Bremen, Germany
Correspondence e-mail: breunig@chemie.uni-bremen.de

Received 27 August 2007
Accepted 11 October 2007
Online 14 November 2007

The structure of the title compound, alternatively called bis(μ-tetramethyldistibinediyl)bis(tetracarbonylchromium), [$Cr_2Sb_4(CH_3)_8(CO)_8$], consists of two Me_4Sb_2 bridging units between $Cr(CO)_4$ complex fragments. The centre of the molecule is located on a special position of $2/m$ symmetry. This is the first characterized Sb_4Cr_2 heterocycle.

Comment

Distibines act as donor ligands through the lone pairs of the antimony atoms to one or two transition metal complex fragments (Breunig & Fichtner, 1979, 1981; Breunig & Knobloch, 1980; von Seyerl & Huttner, 1980; Breunig *et al.*, 1981, 2003; Bernal *et al.*, 1984; Weber, Huttner *et al.*, 1985; Weber, Zsolnai *et al.*, 1985; Dickson *et al.*, 1993; Breunig & Pawlik, 1995; Sharma *et al.*, 1995; Balázs *et al.*, 2003; Lorenz *et al.*, 2005). From the reaction of [Cr(nbd)(CO)$_4$] (nbd = norbornadiene) and tetramethyldistibine, a polymeric complex [Cr(μ-Sb_2Me_4)(CO)$_4$]$_n$ with a *trans* arrangement of the distibine ligands was obtained (Breunig & Knobloch, 1980). We report here the structure of {[Cr(CO)$_4$]$_2$(μ-Sb_2Me_4)$_2$}, (I), the first complex featuring two bridging distibine ligands between transition metal complex fragments. (I) is a cyclic isomer of the polymer [Cr(μ-Sb_2Me_4)(CO)$_4$]$_n$ (Breunig & Knobloch, 1980).



(I)

A displacement ellipsoid representation of the crystal structure of (I) is shown in Fig. 1. The centre of the molecule is located on a special position of $2/m$ symmetry. The Sb—Sb bond lengths in (I) are in the range of values found in the analogous complexes {[Cr(CO)$_5$]$_2$(μ-Sb_2Me_4)} [2.8097 (9) Å; Breunig *et al.*, 2003] and {[Cr(CO)$_5$]$_2$(μ-Sb_2Ph_4)} [2.865 (4) Å; von Seyerl & Huttner, 1980]. In addition, the Sb—Cr bond lengths are of the same magnitude as other reported values: 2.621 (8) and 2.629 (9) Å in {[Cr(CO)$_5$]$_2$(μ-Sb_2Me_4)} (Breunig *et al.*, 2003) and 2.626 (1) Å in {[Cr(CO)$_5$]$_2$(μ-Sb_2Ph_4)} (von Seyerl & Huttner, 1980).

The geometries of the Cr(CO)$_4$ fragments are normal, the bond angles being close to the ideal value of 90°. The C—Sb—Sb and C—Sb—C bond angles are larger than in the uncoordinated distibine Me_4Sb_2 [mean C—Sb—Sb = 93.6 (6)° and mean C—Sb—C = 93.5 (15)°; Ashe *et al.*, 1984], and are in the range of values found for the corresponding angles in {[Cr(CO)$_5$]$_2$(μ-Sb_2Me_4)} [C—Sb—Sb = 96.41 (11)–102.44 (11)° and C—Sb—C = 99.20 (18)–100.34 (16)°; Breunig *et al.*, 2003]. The geometrical parameters of antimony are consistent with the known transition from being close to a p^3 configuration to an sp^3 hybridization of the coordinating atoms in pnicogen ligands by complexation.

The distibine fragments in (I) adopt a synclinal conformation, with a Cr—Sb—Sb—Cr torsion angle of 66.88 (4)° [*cf.* ideal antiperiplanar conformation with Cr—Sb—Sb—Cr = 180° in {[Cr(CO)$_5$]$_2$(μ-Sb_2Me_4)} (Breunig *et al.*, 2003) and {[Cr(CO)$_5$]$_2$(μ-$Sb_2{}^tBu_2Cl_2$)} (Weber, Zsolnai *et al.*, 1985); anticlinal conformation with Cr—Sb—Sb—Cr = 131.0 (4)° in {[Cr(CO)$_5$]$_3$($Sb_2{}^tBu_2$)} (Weber, Huttner *et al.*, 1985)]. The Sb and Cr atoms form a six-membered heterocycle in a chair conformation. There are transannular contacts [3.7133 (9) Å] between the Sb atoms of the parallel distibine units which are shorter than the sum of the van der Waals radii [$\Sigma_{\rm rvdW}$(Sb,Sb) = 4.0 Å; Bondi, 1964].

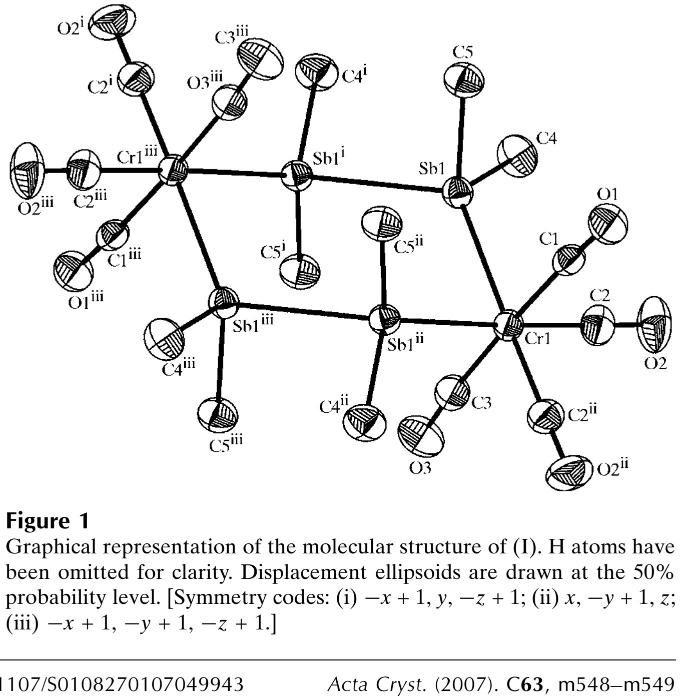


Figure 1
Graphical representation of the molecular structure of (I). H atoms have been omitted for clarity. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x + 1, y, -z + 1$; (ii) $x, -y + 1, z$; (iii) $-x + 1, -y + 1, -z + 1$.]

DOI: 10.1107/S0108270107049943

The crystals consist of stacks of molecules of (I), which are directed along the *a* axis, each stack being surrounded by six neighbouring stacks. There are contacts of 2.73 and 2.74 Å between the carbonyl O atoms and the H atoms of the methyl groups of neighbouring molecules, both inside and between stacks.

Experimental

Crystals of (I) were obtained from a solution of 2,6-Mes$_2$-C$_6$H$_3$Sb(SbMe$_2$)$_2$ (Mes = 2,4,6-Me$_3$C$_6$H$_2$) and [Cr(nbd)(CO)$_4$] in a 1:1 molar ratio in benzene, in a sealed NMR tube at ambient temperature. The synthesis has not been reproduced on a preparative scale.

Crystal data

[Cr$_2$Sb$_4$(CH$_3$)$_8$(CO)$_8$]
M_r = 935.35
Monoclinic, $C2/m$
a = 13.125 (3) Å
b = 11.332 (2) Å
c = 10.185 (2) Å
β = 117.91 (3)°
V = 1338.6 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.80 mm^{-1}
T = 173 (2) K
0.2 × 0.2 × 0.1 mm

Data collection

Stoe IPDS-1 diffractometer
Absorption correction: part of the refinement model (ΔF) (Walker & Stuart, 1983)
$T_{\min}$ = 0.447, $T_{\max}$ = 0.646
4676 measured reflections
1367 independent reflections
1173 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.056
S = 0.99
1367 reflections
78 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 1.07 e Å^{-3}
$\Delta\rho_{\min}$ = −0.55 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.134 (8)	C3—Cr1	1.887 (7)
C1—Cr1	1.900 (6)	C4—Sb1	2.148 (4)
C2—O2	1.161 (6)	C5—Sb1	2.148 (4)
C2—Cr1	1.846 (5)	Sb1—Cr1	2.6205 (9)
C3—O3	1.151 (8)	Sb1—Sb1$^{\rm i}$	2.8157 (11)
C4—Sb1—C5	100.00 (19)	Cr1—Sb1—Sb1$^{\rm i}$	121.89 (3)
C4—Sb1—Sb1$^{\rm i}$	97.32 (14)	Sb1$^{\rm ii}$—Cr1—Sb1	90.23 (4)
C5—Sb1—Sb1$^{\rm i}$	97.44 (14)		
Sb1$^{\rm i}$—Sb1—Cr1—Sb1$^{\rm ii}$	−51.34 (4)		

Symmetry codes: (i) $-x+1, y, -z+1$; (ii) $x, -y+1, z$.

During the space-group determination using *XPREP* (Bruker, 1998), the reflections $\overline{2}03$, 003 and $\overline{2}01$ were removed. The H atoms were placed in calculated positions (C—H = 0.98 Å) using a riding model, with $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C). The methyl groups were allowed to rotate. The largest residual electron-density peak is located 1.22 Å from atom H4*C* and the deepest hole is located 0.88 Å from atom Sb1.

Data collection: *IPDS-1* (Stoe & Cie, 1999); cell refinement: *IPDS-1*; data reduction: *IPDS-1*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *publCIF* (Westrip, 2007).

The authors thank Mr P. Brackmann for crystal selection and for the crystallographic measurements. The authors also thank the Deutsche Forschungsgemeinschaft for financial support.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GA3072). Services for accessing these data are described at the back of the journal.

References

Ashe, A. J. III, Ludwig, E. G. Jr, Oleksyszyn, J. & Huffman, J. C. (1984). *Organometallics*, **3**, 337–338.
Balázs, G., Breunig, H. J., Lork, E. & Mason, S. (2003). *Organometallics*, **22**, 576–585.
Bernal, I., Korp, J. D., Calderazzo, F., Poli, R. & Vitali, D. (1984). *J. Chem. Soc. Dalton Trans.* pp. 1945–1950.
Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Brandenburg, K. & Putz, H. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Breunig, H. J., Fichtner, W. & Knobloch, T. P. (1981). *Z. Anorg. Allg. Chem.* **477**, 126–132.
Breunig, H. J. & Fichtner, W. (1979). *Z. Anorg. Allg. Chem.* **454**, 167–174.
Breunig, H. J. & Fichtner, W. (1981). *Z. Anorg. Allg. Chem.* **477**, 119–125.
Breunig, H. J., Ghesner, I., Ghesner, M. E. & Lork, E. (2003). *J. Organomet. Chem.* **677**, 15–20.
Breunig, H. J. & Knobloch, T. P. (1980). *J. Inorg. Nucl. Chem.* **42**, 505–507.
Breunig, H. J. & Pawlik, J. (1995). *Z. Anorg. Allg. Chem.* **621**, 817–822.
Bruker (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Dickson, R. S., Heazle, K. D., Pain, G. N., Deacon, G. B., West, B. O., Fallon, G. D., Rowe, R. S., Leech, P. W. & Faith, M. (1993). *J. Organomet. Chem.* **449**, 131–139.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Lorenz, I.-P., Rudolph, S., Piotrowski, H. & Polborn, K. (2005). *Eur. J. Inorg. Chem.* pp. 82–85.
Seyerl, J. von & Huttner, G. (1980). *Cryst. Struct. Commun.* **9**, 1099–1103.
Sharma, P., Rosas, N., Hernández, S. & Cabrera, A. (1995). *Chem. Commun.* pp. 1325–1326.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (1999). *IPDS-1*. Stoe & Cie, Darmstadt, Germany.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.
Weber, U., Huttner, G., Scheidsteger, O. & Zsolnai, L. (1985). *J. Organomet. Chem.* **289**, 357–366.
Weber, U., Zsolnai, L. & Huttner, G. (1985). *Z. Naturforsch. Teil B*, **40**, 1430–1436.
Westrip, S. P. (2007). *publCIF*. In preparation.