

Solvated  $\text{CrBr}_2(\text{thf})_2$ , a linear chain in the solid stateBrendan Twamley,<sup>a</sup> Ralph  
Zehnder<sup>b</sup> and Pamela J.  
Shapiro<sup>b\*</sup><sup>a</sup>University Research Office, 109 Morrill Hall,  
University of Idaho, Moscow, ID 83844-3010,  
USA, and <sup>b</sup>Department of Chemistry, University  
of Idaho, Moscow, ID 83844-2343, USA

Correspondence e-mail: btwamley@uidaho.edu

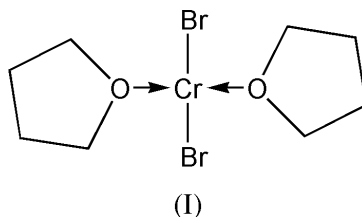
## Key indicators

Single-crystal X-ray study  
 $T = 203 \text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$   
 $R$  factor = 0.027  
 $wR$  factor = 0.071  
Data-to-parameter ratio = 15.8For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

In the title compound, dibromobis(tetrahydrofuran-*O*)-chromium(II),  $[\text{CrBr}_2(\text{C}_4\text{H}_8\text{O})_2]$ ,  $\text{CrBr}_2$  is solvated by two tetrahydrofuran (thf) molecules, with a  $\text{Cr}-\text{O}$  distance of  $2.072(3) \text{ \AA}$  and a  $\text{Cr}-\text{Br}$  distance of  $2.5825(5) \text{ \AA}$ . The Cr atom lies on the center of inversion. The Cr atom is octahedrally coordinated, with the thf  $\text{Cr}-\text{O}$  bond orthogonal to the  $\text{Cr}-\text{Br}$  bond, and the vacant sites are occupied with a  $\text{Cr}\cdots\text{Br}$  intermolecular interaction of  $2.9874(5) \text{ \AA}$ . The latter interaction is almost orthogonal to the  $\text{Cr}-\text{O}$  vector. This extends the system into a linear chain along the  $[100]$  direction.

## Comment

$\text{CrBr}_2(\text{thf})_2$ , (I), bears a close resemblance to the parent compound  $\text{CrBr}_2$  (Tracy *et al.*, 1962). Both show slightly distorted octahedral coordination and both have similar in-plane  $\text{Cr}-\text{Br}$  bond distances,  $2.5825(5) \text{ \AA}$  in (I) and  $2.545(1) \text{ \AA}$  in  $\text{CrBr}_2$ . The solvent molecule, thf in this case, occupies an in-plane coordination site with a  $\text{Cr}-\text{O}$  distance of  $2.072(3) \text{ \AA}$ . The solvent molecule is coordinated orthogonally to the  $\text{Cr}-\text{Br}$  vector with an angle of  $90.009(8)^\circ$ . The thf molecule is in a half-chair conformation with a total puckering amplitude of  $Q_T = 0.363 \text{ \AA}$  (Cremer & Pople, 1975). The orientation of the thf molecule is influenced by the intramolecular interactions  $\text{C}4-\text{H}4\text{A}\cdots\text{Br}1$  and  $\text{C}1-\text{H}1\text{A}\cdots\text{Br}1^i$  (see Table 1) which tilt the ring towards each Br atom. The out-of-plane coordination site is occupied by an intermolecular  $\text{Cr}1\cdots\text{Br}1^{ii}$  interaction of  $2.9874(5) \text{ \AA}$  [symmetry code: (ii)  $1 + x, y, z$ ]. The corresponding interaction in  $\text{CrBr}_2$  has a  $\text{Cr}\cdots\text{Br}$  distance of  $2.998(1) \text{ \AA}$ . The distortion



from ideal octahedral geometry is also reflected in the  $\text{Br}1-\text{Cr}1-\text{Br}1^{ii}$  angle of  $94.65(2)^\circ$ . With the thf molecules occupying the other coordination sites, (I) can only form a staggered chain instead of the planar sheets found in  $\text{CrBr}_2$ . In this case, the direction of propagation is along  $[100]$  (Fig. 1). Other solvent coordinated  $\text{Cr}^{II}\text{Br}_2$  species that are octahedrally coordinated show a wide range of  $\text{Cr}-\text{Br}$  distances. The complex  $\text{CrBr}_2(\text{CH}_3\text{CN})_2$  (Halepoto *et al.*, 1990), which also forms linear chains, has an in-plane  $\text{Cr}-\text{Br}$  distance of  $2.545 \text{ \AA}$  and an out-of-plane distance of  $2.976(1) \text{ \AA}$ . However,

Received 2 January 2001  
Accepted 23 January 2001  
Online 30 January 2001

the molecular species *trans*-CrBr<sub>2</sub>(pyridine)<sub>4</sub> (Holah *et al.*, 1998) has a Cr–Br bond distance of 2.998 (1) Å which indicates a very weakly associated Br atom.

## Experimental

CrBr<sub>2</sub>(thf)<sub>2</sub> was synthesized as a side product during the attempted synthesis of a chromocene bromide. Tetramethylethylenediyl( $\eta^5$ -bicyclopentadienyl)chromocene<sup>II</sup> carbonyl (331 mg, 1.1 mmol) was reacted with 1-bromo-2-methylpropane (155 mg, 1.1 mmol) in tetrahydrofuran for 20 h at 378 K, with no color change. Dibromomethane (excess) was then added to increase the bromide concentration. Excess solvent and reagents were removed under reduced pressure and the green residue redissolved in tetrahydrofuran (20 ml) and filtered. The resulting solution was concentrated to promote crystallization and cooled in a 278 K refrigerator overnight. The supernatant was decanted yielding a green crystalline product. The supernatant was concentrated under reduced pressure to yield a fine pale green powder. Combined yield 68 mg, 19.1%, m.p. > 573 K.

### Crystal data

[CrBr <sub>2</sub> (C <sub>4</sub> H <sub>8</sub> O) <sub>2</sub> ]	<i>Z</i> = 1
<i>M<sub>r</sub></i> = 356.03	<i>D<sub>x</sub></i> = 2.126 Mg m <sup>-3</sup>
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> $\alpha$ radiation
<i>a</i> = 4.1043 (7) Å	Cell parameters from 839 reflections
<i>b</i> = 7.4530 (13) Å	$\theta$ = 2.2–27.4°
<i>c</i> = 9.2875 (16) Å	$\mu$ = 8.18 mm <sup>-1</sup>
$\alpha$ = 87.777 (3)°	<i>T</i> = 203 (2) K
$\beta$ = 78.619 (3)°	Plate, pale yellow
$\gamma$ = 87.281 (3)°	0.25 × 0.20 × 0.15 mm
<i>V</i> = 278.07 (8) Å <sup>3</sup>	

### Data collection

Siemens SMART 1000 diffractometer	$\theta_{\max}$ = 25.0°
$\omega$ scans	<i>h</i> = -4 → 4
Absorption correction: empirical ( <i>SADABS</i> ; Sheldrick, 1999)	<i>k</i> = -8 → 8
<i>T<sub>min</sub></i> = 0.184, <i>T<sub>max</sub></i> = 0.293	<i>l</i> = -11 → 11
2917 measured reflections	50 frames remeasured standard reflections
962 independent reflections	every beginning and end reflections
897 reflections with <i>I</i> > 2 $\sigma$ ( <i>I</i> )	intensity decay: none
<i>R<sub>int</sub></i> = 0.029	

### Refinement

Refinement on <i>F</i> <sup>2</sup>	$w = 1/[\sigma^2(F_o^2) + (0.0436P)^2 + 0.1523P]$
$R[F^2 > 2\sigma(F^2)] = 0.027$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.071$	( $\Delta/\sigma$ ) <sub>max</sub> = 0.001
<i>S</i> = 1.08	$\Delta\rho_{\max} = 0.58 \text{ e \AA}^{-3}$
962 reflections	$\Delta\rho_{\min} = -0.70 \text{ e \AA}^{-3}$
61 parameters	
H-atom parameters constrained	

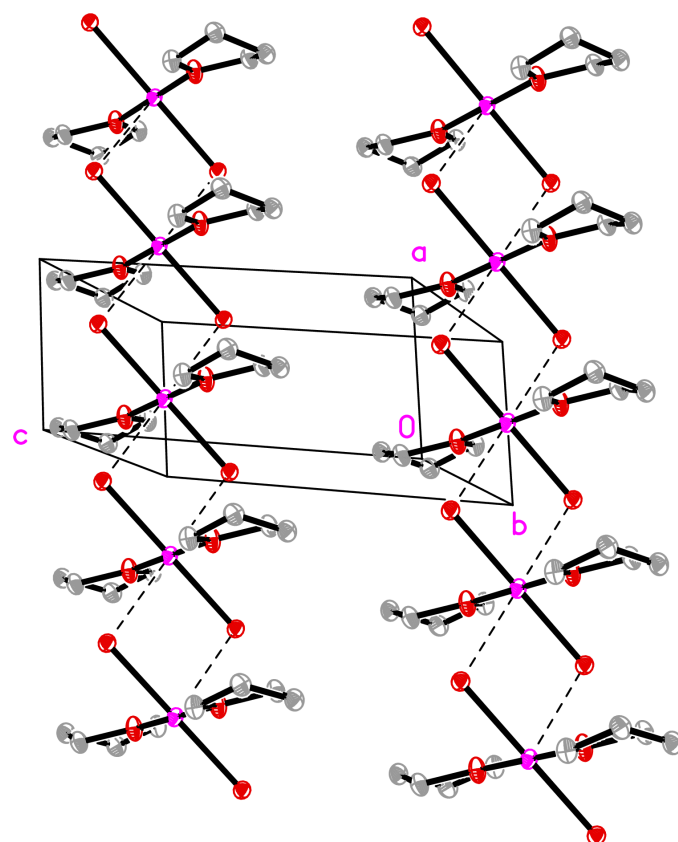
**Table 1**

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
C4–H4A...Br1	0.98	2.99	3.599 (4)	122
C1–H1A...Br1 <sup>i</sup>	0.98	2.99	3.607 (4)	122

Symmetry code: (i) 1 - *x*, 1 - *y*, 1 - *z*.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINTE-Plus* (Bruker, 1999); program(s) used to solve



**Figure 1**

Packing diagram of (I) showing the staggered propagation along the [100] direction. H atoms have been omitted for clarity.

structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This work was supported by a grant from the National Science Foundation (CHE-9816730), the DOE EPSCoR Program (Notice 98-02) and the Petroleum Research Foundation (ACS-PRF No. 33831-AC). The Bruker (Siemens) SMART CCD diffraction facility was established at the University of Idaho with the assistance of the NSF-EPSCoR program under NSF OSR-9350539 and the M. J. Murdock Charitable Trust, Vancouver, Wa, USA.

## References

- Bruker (1997). *SMART*. Version 5.059. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (1999). *SAINTE-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Halepoto, D. M., Larkworthy, L. F., Povey, D. C., Tucker, B. J. & Smith, G. W. (1990). *J. Chem. Soc. Dalton Trans.* pp. 699–701.
- Holah, D. G., Hughes, A. N., Lundeen, C. & Magnuson, V. R. (1998). *Polyhedron*, **17**, 2101–2104.
- Sheldrick, G. M. (1998). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick G. M. (1999). *SADABS*. Version 2.01. Bruker AXS Inc., Madison, Wisconsin, USA.
- Tracy, J. W., Gregory, N. W. & Lingafelter, E. C. (1962). *Acta Cryst.* **15**, 672–674.