

Brian N. Figgis\* and  
Alexandre N. SobolevChemistry, M313, School of Biomedical and  
Chemical Sciences, University of Western  
Australia, 35 Stirling Highway, Crawley,  
WA 6009, AustraliaCorrespondence e-mail:  
bnf@theochem.uwa.edu.au

## Key indicators

Single-crystal X-ray study  
 $T = 11$  K  
Mean  $\sigma(\text{O}-\text{C}) = 0.001$  Å  
 $R$  factor = 0.020  
 $wR$  factor = 0.054  
Data-to-parameter ratio = 61.9For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

## Hexacarbonylchromium(0) at 11 K

The crystal structure of hexacarbonylchromium(0),  $[\text{Cr}(\text{CO})_6]$ , has been refined with an accurate and extensive X-ray data set collected at 11 K. This data set should be suitable for charge-density analysis studies. The structure is made up of isolated  $\text{Cr}(\text{CO})_6$  molecules of very near octahedral symmetry, containing a crystallographic mirror plane.

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## Comment

$\text{Cr}(\text{CO})_6$ , (I), has long been of interest in inorganic chemistry as it is an archetypal transition metal carbonyl complex. Its room-temperature structure was determined by Whitaker & Jeffery (1967) and an  $X$ -N charge-density study was carried out at 74 K (Rees & Mitschler, 1976).

The cell parameters, bond lengths and angles for the  $\text{Cr}(\text{CO})_6$  molecule at various temperatures are given in Table 1.

The structure consists of isolated  $\text{Cr}(\text{CO})_6$  molecules. Each molecule comprises a near-regular octahedron. There is a plane containing two pairs of CO groups related by crystallographic mirror symmetry and with C–Cr–C angles within  $1^\circ$  of a right angle. The relevant Cr–C bond lengths differ by only 0.005 Å. Above and below this plane are two CO groups within  $0.5^\circ$  of a right angle and which differ in distance only slightly (0.005 Å) and match those in the plane within the same amount, the overall average being 1.916 Å. The Cr–C–O angles are all within  $1^\circ$  of being linear, and the C–O bond lengths all lie within 0.002 Å of the average of 1.142 Å.

The atomic displacement parameters decrease with temperature, largely as expected.

## Experimental

In a sealed glass tube,  $\text{Cr}(\text{CO})_6$  (Aldrich) was sublimed over a temperature gradient of  $ca$   $1 \text{ K mm}^{-1}$  for a period of 1 d. The very low temperature data sets were collected on a locally assembled Huber 512 goniometer equipped with a Displex 202D cryogenic refrigerator (Henriksen *et al.*, 1986; Larsen, 1995). A full sphere of data was collected. The correction for the absorption by the beryllium thermal shields was calculated by the *PROFIT* (Streltsov & Zavodnik, 1989) program.

## Crystal data

$[\text{Cr}(\text{CO})_6]$   
 $M_r = 220.06$   
Orthorhombic,  $Pnma$   
 $a = 11.474$  (1) Å  
 $b = 10.894$  (1) Å  
 $c = 6.1885$  (4) Å  
 $V = 773.55$  (11) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.890 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation  
Cell parameters from 14  
reflections  
 $\theta = 31.4$ – $38.7^\circ$   
 $\mu = 1.47 \text{ mm}^{-1}$   
 $T = 11.0$  (5) K  
Prism, pale yellow  
 $0.46 \times 0.40 \times 0.40 \text{ mm}$

Data collection

Huber 512 goniometer diffractometer  
 $\omega$ -2 $\theta$  scans  
 Absorption correction: Gaussian (*Xtal3.7*; Hall *et al.*, 2000)  
 $T_{\min} = 0.564$ ,  $T_{\max} = 0.639$   
 27 649 measured reflections  
 4212 independent reflections  
 4125 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$   
 $\theta_{\text{max}} = 50.1^\circ$   
 $h = -24 \rightarrow 24$   
 $k = -23 \rightarrow 23$   
 $l = -13 \rightarrow 13$   
 3 standard reflections every 100 reflections  
 intensity decay: 1%

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.020$   
 $wR(F^2) = 0.054$   
 $S = 1.14$   
 4212 reflections  
 68 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0244P)^2 + 0.21P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.84 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -1.00 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.069 (2)

Table 1

Cell parameters ( $\text{\AA}$ ,  $\text{\AA}^3$ ), bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) in the  $\text{Cr}(\text{CO})_6$  molecule at various temperatures (K) by X-ray [X] and neutron [N] diffraction.

Cell parameters	293[X] <sup>a</sup>	74[X] <sup>b</sup>	78[N] <sup>c</sup>	11[X] <sup>d</sup>
<i>a</i>	11.769 (12)	11.505 (4)	11.490 (15)	11.474 (1)
<i>b</i>	11.092 (11)	10.916 (3)	10.905 (13)	10.894 (1)
<i>c</i>	6.332 (6)	6.203 (2)	6.197 (14)	6.1885 (4)
<i>V</i>	826.6 (24)	779.0 (7)	776.5 (37)	773.55 (11)
Cr—C1	1.891 (7)	1.912 (2)	1.913 (2)	1.9121 (5)
Cr—C2	1.928 (7)	1.916 (2)	1.911 (2)	1.9171 (5)
Cr—C3	1.910 (4)	1.915 (1)	1.913 (1)	1.9152 (4)
Cr—C4	1.909 (4)	1.918 (1)	1.919 (1)	1.9201 (4)
C1—O1	1.151 (8)	1.143 (3)	1.140 (1)	1.1432 (7)
C2—O2	1.109 (8)	1.141 (3)	1.141 (1)	1.1421 (7)
C3—O3	1.137 (6)	1.139 (2)	1.141 (1)	1.1412 (5)
C4—O4	1.143 (6)	1.138 (2)	1.138 (1)	1.1399 (5)
Cr—C1—O1	179.6 (6)	179.9 (2)	180.0 (1)	179.94 (5)
Cr—C2—O2	178.8 (6)	179.1 (2)	179.5 (1)	179.45 (5)
Cr—C3—O3	178.3 (4)	179.4 (2)	179.37 (8)	179.38 (4)
Cr—C4—O4	178.5 (4)	179.2 (1)	179.10 (7)	179.19 (3)
C1—Cr—C2	178.9 (3)	179.6 (1)	179.4 (1)	179.45 (2)
C1—Cr—C3	90.0 (2)	90.37 (6)	90.34 (6)	90.36 (2)
C1—Cr—C4	89.8 (3)	90.14 (7)	89.96 (6)	90.09 (2)
C2—Cr—C3	90.7 (2)	89.94 (6)	90.06 (6)	90.03 (2)
C2—Cr—C4	89.4 (2)	89.55 (6)	89.65 (6)	89.53 (2)
C3—Cr—C3 <sup>i</sup>	89.5 (3)	89.40 (8)	89.53 (8)	89.54 (2)
C3—Cr—C4	89.5 (2)	89.85 (5)	89.79 (3)	89.79 (2)
C3—Cr—C4 <sup>i</sup>	179.3 (3)	179.10 (6)	179.26 (8)	179.20 (2)
C4—Cr—C4 <sup>i</sup>	90.8 (3)	90.89 (8)	90.89 (8)	90.88 (2)

References: (a) Whitaker & Jeffery (1967); (b) Rees & Mitschler (1976); (c) Jost *et al.* (1975); (d) this work. Symmetry code: (i)  $x, \frac{1}{2} - y, z$ .

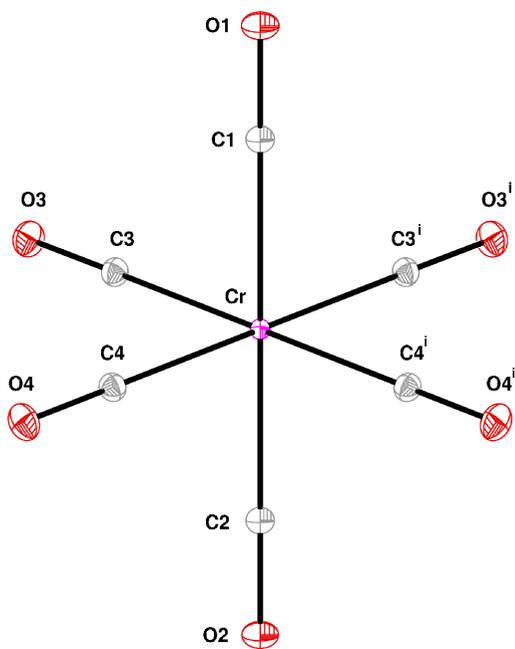


Figure 1  
 The  $\text{Cr}(\text{CO})_6$  molecule at 11 K. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code: (i)  $x, \frac{1}{2} - y, z$ .]

Data collection: Local diffractometer control software; cell refinement: Local diffractometer control software; data reduction: *PROFIT* (Streltsov & Zavodnik, 1989); program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Bruker (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Hall, S. R., du Boulay, D. J. & Olthof-Hazekamp, R. (2000). Editors. *Xtal3.7 System*. University of Western Australia, Australia.  
 Henriksen, K., Larsen, F. K. & Rasmussen, S. E. (1986). *J. Appl. Cryst.* **19**, 390–394.  
 Jost, A., Rees, B. & Yelon, W. B. (1975). *Acta Cryst.* **B31**, 2649–2658.  
 Larsen, F. K. (1995). *Acta Cryst.* **B51**, 468–482.  
 Rees, B. & Mitschler, A. (1976). *J. Am. Chem. Soc.* **98**, 7918–7924.  
 Streltsov, V. A. & Zavodnik, V. E. (1989). *Sov. Phys. Crystallogr.* **34**, 824–828.  
 Whitaker, A. & Jeffery, J. W. (1967). *Acta Cryst.* **23**, 977–984.