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Tris(*O*-methyldithiocarbonato)chromium(III)

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(O–C) = 0.005 Å
R factor = 0.033
wR factor = 0.022
Data-to-parameter ratio = 14.1

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

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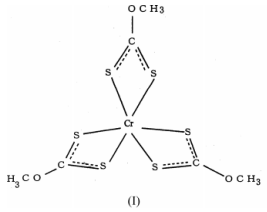
In the title complex, $[Cr(C_2H_3OS_2)_3]$, the Cr atom is coordinated by six S atoms in a distorted octahedral arrangement. The six Cr—S bond distances are in the range 2.391 (1)–2.406 (1) Å, with an average of 2.397 (2) Å.

Comment

As part of a study of metal xanthates and dialkyl dithiophosphates (Ito, 2002*a*,*b*), the crystal and molecular structure of the title complex, (I), has been determined. A displacement ellipsoid plot of (I) is shown in Fig. 1. The average Cr—S distance of 2.397 (2) Å is 0.032 (7) Å shorter than that in tris(*O*,*O*′-dimethyldithiophosphato)chromium(III), (II) (Ito, 2002*b*), which shows that the Cr—S bonds in (I) are stronger than those in (II). On the other hand, distortions of S atoms around the Cr atom from octahedral coordination in (I) are larger than those in (II). For example, average S—Cr—S chelate angles in (I) and (II) are 74.5 (2)° and 81.8°, respectively.



(I)

The structures of xanthate ligands in (I) are very similar to those in iron methylxanthate, (III) (Ito, 2002*a*). Average S—C, S_2C—O and O—CH_3 bond distances of 1.688 (4), 1.318 (4) and 1.446 (4) Å, respectively, are in agreement with the corresponding distances in (III), within standard uncertainties.

Experimental

Potassium methylxanthate (2.0 g) and hexaaquachromium(III) chloride (4.0 g) were each dissolved in pure water (40 ml and 80 ml, respectively), and a powder of (I) was precipitated by combining the two solutions. Recrystallization from an ether solution at room temperature gave dark-blue plate-shaped crystals of (I).

Crystal data

$[Cr(C_2H_3OS_2)_3]$
M_r = 373.55
Monoclinic, $P2_1/n$
a = 9.633 (4) Å
b = 13.852 (4) Å
c = 11.301 (3) Å
β = 106.94 (2)°
V = 1442.5 (8) Å^3
Z = 4
D_x = 1.720 Mg m^{-3}
D_m = 1.715 Mg m^{-3}
D_m measured by flotation in zinc iodide (aq.)
Mo Kα radiation
Cell parameters from 23 reflections
θ = 15.2–16.3°
μ = 1.65 mm^{-1}
T = 296 K
Plate, dark blue
0.45 × 0.35 × 0.10 mm

Data collection

Rigaku AFC-5*S* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.510, T_{max} = 0.848
3640 measured reflections
3311 independent reflections
2041 reflections with $I > 3\sigma(I)$
R_{int} = 0.029
θ_{max} = 27.5°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 17$
$l = -14 \rightarrow 14$
3 standard reflections
every 150 reflections
intensity decay: 0.1%

Refinement

Refinement on F
R = 0.033
wR = 0.022
S = 1.66
2041 reflections
145 parameters
H-atom parameters constrained
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cr—S1	2.406 (1)	S4—C2	1.693 (4)
Cr—S2	2.394 (1)	S5—C3	1.700 (4)
Cr—S3	2.391 (1)	S6—C3	1.676 (4)
Cr—S4	2.394 (1)	O1—C1	1.322 (4)
Cr—S5	2.403 (1)	O1—C4	1.444 (4)
Cr—S6	2.394 (1)	O2—C2	1.316 (4)
S1—C1	1.688 (4)	O2—C5	1.445 (4)
S2—C1	1.690 (4)	O3—C3	1.317 (4)
S3—C2	1.682 (4)	O3—C6	1.448 (4)
S1—Cr—S2	74.35 (4)	S2—Cr—S6	94.15 (5)
S1—Cr—S3	93.98 (5)	S3—Cr—S4	74.44 (4)
S1—Cr—S4	164.34 (5)	S3—Cr—S5	96.62 (5)
S1—Cr—S5	94.76 (5)	S3—Cr—S6	165.89 (4)
S1—Cr—S6	97.47 (5)	S4—Cr—S5	97.01 (5)
S2—Cr—S3	96.85 (5)	S4—Cr—S6	95.64 (5)
S2—Cr—S4	96.23 (5)	S5—Cr—S6	74.31 (4)
S2—Cr—S5	163.21 (5)		

H atoms were placed in geometrically calculated positions and made to ride on their parent atoms, with U_{iso} parameters equal to 1.2 times the U_{eq} parameters of their parent atoms.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *CrystalStructure* (Molecular Structure Corporation and Rigaku Corporation, 2001); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *CrystalStructure*; molecular graphics: *ORTEP*-III (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

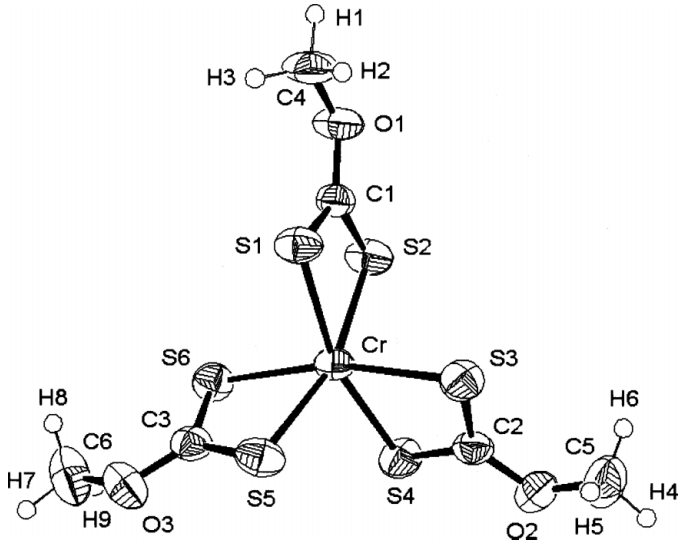


Figure 1
ORTEP-III (Burnett & Johnson, 1996) drawing of the title chromium methylxanthate complex. Displacement ellipsoids are drawn at the 50% probability level.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.

Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*-III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.

Ito, T. (2002*a*). *Acta Cryst.* E**58**, m265–m266.

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

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.

Molecular Structure Corporation & Rigaku Corporation. (2001). *CrystalStructure*. Version 2.00. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA, and Rigaku Corporation, Akishima, Tokyo 196-8666, Japan.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.