

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Racemic tricarbonyl(η^6 -7-methoxyflavan)chromium(0)

 Johannes. H. van Tonder,^a Barend C. B. Bezuidenhoudt^{a*} and J. Marthinus Janse van Rensburg^b
^aDepartment of Chemistry, University of the Free State, PO Box 339, Bloemfontein 9300, South Africa, and ^bOrganic Chemistry, Department of Chemistry, Lund University, PO Box 124, S-221 00, Lund, Sweden

Correspondence e-mail: bezuidbc.sci@ufs.ac.za

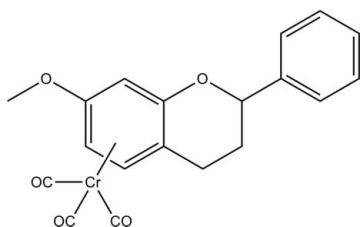
Received 15 February 2010; accepted 25 June 2010

 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.030; wR factor = 0.085; data-to-parameter ratio = 17.6.

In the title compound [systematic name: tricarbonyl(η^6 -7-methoxy-2-phenyl-3,4-dihydro-2H-1-benzopyran)chromium(0)], $[\text{Cr}(\text{C}_{16}\text{H}_{16}\text{O}_2)(\text{CO})_3]$, the $\text{Cr}(\text{CO})_3$ unit is coordinated by the phenylene ring of the flavan ligand, exhibiting a three-legged piano-stool conformation, with a point to plane distance of 1.750 (1) Å. The phenyl ring is twisted away from the fused ring system by 36.49 (5)° (r.m.s. deviation = 0.027 Å; fitted atoms are the C_6 ring and the attached fused-ring C and O atoms). The dihydropyran ring displays a distorted envelope configuration by displacement of the phenyl-bearing and the adjacent ring C atoms from the fused-ring system plane by 0.356 (2) and 0.402 (2) Å, respectively.

Related literature

7-Methoxyflavan was synthesized *via* hydrogenation from 7-methoxyflavanone, as described by Sato *et al.* (2006). For coordination of 7-methoxyflavan to chromium, see: Müller *et al.* (1999). For the importance of flavonoids in biological investigations, see: Rice-Evans & Packer (2003). For $\text{Cr}(\text{CO})_3$ coordination to the phenylene ring of a flavanone compound, see: Dominique *et al.* (1999). For comparison bond distances, see: Allen *et al.* (1987). For related structures, see: van Tonder *et al.* (2009a,b). For the use of tricarbonyl(arene)chromium complexes in regioselective organic synthesis, see: Muschalek *et al.* (2007).



Experimental

Crystal data

$[\text{Cr}(\text{C}_{16}\text{H}_{16}\text{O}_2)(\text{CO})_3]$
 $M_r = 376.32$
 Monoclinic, $P2_1/c$
 $a = 9.8422$ (2) Å
 $b = 12.3850$ (3) Å
 $c = 15.0146$ (3) Å
 $\beta = 115.171$ (1)°

$V = 1656.42$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.72$ mm⁻¹
 $T = 173$ K
 $0.41 \times 0.34 \times 0.24$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.757$, $T_{\max} = 0.847$

12982 measured reflections
 3985 independent reflections
 3224 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.085$
 $S = 1.07$
 3985 reflections

227 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.40$ e Å⁻³

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT-Plus (Bruker, 2004); data reduction: SAINT-Plus and XPREP (Bruker, 2004); program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 2005); software used to prepare material for publication: WinGX (Farrugia, 1999).

Financial assistance from the University of the Free State and SASOL to JHvanT is gratefully acknowledged. Opinions, findings, conclusions or recommendations expressed in this material are those of the authors and do not necessarily reflect the views of SASOL. We would like to express our gratitude to the School of Chemistry at the University of the Witwatersrand for the use of the diffractometer. Special thanks are due to Dr M. A. Fernandes.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZB2005).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Altomare, A., Burla, M. C., Camalli, M., Casciarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2004). *SAINTE-Plus*, *XPREP* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dominique, S., Lepoivre, A., Lemièrre, G., Rapatopoulou, C. P. & Klouras, N. D. (1999). *Monatsh. Chem.* **130**, 305–311.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Müller, T. J. J., Ansorge, M. & Polburn, K. (1999). *J. Organomet. Chem.* **578**, 252–259.
- Muschalek, B., Weidner, I. & Butenschön, H. (2007). *J. Organomet. Chem.* **692**, 2415–2424.
- Rice-Evans, C. A. & Packer, L. (2003). *Flavonoids in Health and Disease*, 2nd ed. New York: Marcel Dekker Inc.

metal-organic compounds

Sato, S., Hiroe, K., Kumazawa, T. & Jun-ichi, O. (2006). *Carbohydr. Res.* **341**, 1091–1095.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

Tonder, J. H. van, Bezuidenhout, B. C. B. & Janse van Rensburg, J. M. (2009a). *Acta Cryst.* **E65**, m1343.
Tonder, J. H. van, Bezuidenhout, B. C. B. & Janse van Rensburg, J. M. (2009b). *Acta Cryst.* **E65**, m1346.

supporting information

Acta Cryst. (2010). E66, m907–m908 [https://doi.org/10.1107/S1600536810024992]

Racemic tricarbonyl(η^6 -7-methoxyflavan)chromium(0)

Johannes. H. van Tonder, Barend C. B. Bezuidenhout and J. Marthinus Janse van Rensburg

S1. Comment

The title compound, (I), $[\text{Cr}(\text{C}_{16}\text{H}_{16}\text{O}_2)(\text{CO})_3]$, where $(\text{C}_{16}\text{H}_{16}\text{O}_2) = 7\text{-methoxyflavan}$, has been examined due to the general biological activity of flavanoids (Rice-Evans & Packer, 2003) and the use of tricarbonyl(arene)chromium complexes in regioselective organic synthesis (Muschalek *et al.*, 2007).

As with the tricarbonyl(η^6 -flavanone)chromium(0) complex reported by Dominique *et al.* (1999), the $\text{Cr}(\text{CO})_3$ unit of the title compound is coordinated by the phenylene ring of the flavanoid backbone (Fig.1). The chromium metal centre is displaced by 1.750 (1) Å from the A- η^6 -coordinated arene ring centre. The dihydropyran ring displays a distorted envelope configuration by displacement of atoms C2 and C3 from the fused ring system plane, with distances of 0.356 (2) and 0.402 (2) Å respectively (r.m.s. of fitted atoms C4, C10, C5, C6, C7, C8, C9 and O5 = 0.027 °). Further molecular disorder is displayed by the phenyl ring twist away from the fused ring system plane, by 36.49 (5)°.

The molecular packing displays two types of soft intermolecular contacts, this between O2...H8 [2.682 (1) Å] forming a O2...H8—C8 angle of 152.5 (1)° and O1...H6 [2.459 (1) Å] forming a O1...H6—C6 angle of 125.9 (1)° (Fig.2).

S2. Experimental

7-Methoxyflavan was synthesised via H_2SO_4 catalyzed hydrogenation (5 bar) over 10% Pd/C from 7-methoxyflavanone, as described by Sato *et al.* (2006). 7-Methoxyflavan-4-one (1.00 g; 3.9 mmol), 10 % Pd/C (0.10 g), 3 M H_2SO_4 (aq.) (1 ml), EtOH (30 ml). Purification by means of flash column-chromatography yielded 7-methoxyflavan (0.67 g; 70.6%) as a colourless oil.

R_f 0.65 (H:DCM:EtOAc; 50:50:1); ^1H NMR (600 MHz, CDCl_3) δ ppm 7.44 – 7.41 (2H, m, H-2' and H-6'), 7.40 – 7.37 (2H, m, H-3' and H-5'), 7.34 – 7.31 (1H, m, H-4'), 6.99 – 6.97 (1H, m, H-5), 6.50 – 6.47 (2H, m, H-6 and H-8), 5.05 (1H, dd, $J = 2.37, 10.19$ Hz, H-2), 3.77 (3H, s, $-\text{OCH}_3$), 2.92 (1H, ddd, $J = 6.02, 10.92, 16.08$ Hz, H-4(a)), 2.74 (1H, ddd, $J = 3.40, 5.12, 16.08$ Hz, H-4(e)), 2.22 – 2.18 (1H, m, H-3), 2.11– 2.04 (1H, m, H-3); ^{13}C NMR (600 MHz, CDCl_3) δ ppm 24.47 (C-4), 30.19 (C-3), 55.38 ($-\text{OCH}_3$), 77.98 (C-2), 101.71 (C-6/8), 107.54 (C-6/8), 114.01, 126.11, 127.93, 128.61, 130.05, 141.79, 155.91, 155.91, 159.23

Preparation of the title compound, tricarbonyl(A- η^6 -7-methoxyflavane)chromium(0), was based on a method described by Müller *et al.* (1999). A solution of 7-Methoxyflavane (0.27 g, 1.1 mmol) and $\text{Cr}(\text{CO})_6$ (0.25 g, 1.1 mmol: 1 eq.) in $\text{Bu}_2\text{O}:\text{THF}$ (9:1; 10 ml per 100 mg $\text{Cr}(\text{CO})_6$) was degassed with argon, using standard Schlenk techniques, and refluxed (48 h) under an oxygen free atmosphere. The reaction mixture was cooled to room temperature and the solvent evaporated in vacuo. Purification through flash column-chromatography yielded tricarbonyl(A- η^6 -7-methoxyflavane)chromium(0) (0.07 g; 16.6.0%) as a yellow solid. Recrystallization from diethyl ether yielded yellow cuboidal crystals.

R_f 0.23 (Hexane: Acetone; 8:2); Mp 148.4 °C; Note: A, B and C-ring labelling refers to the benzene, phenyl and dihydropyran rings respectively. ^1H NMR (600 MHz, CDCl_3) δ ppm 7.49 (2H, d, $J = 7.15$ Hz, H-2' and H-6'), 7.41 (2H,

dd, $J = 7.15, 8.66$ Hz, H-3' and H-5'), 7.39 – 7.35 (1H, m, H-4'), 5.65 (1H, d, $J = 6.61$ Hz, H-5), 5.15 (1H, s, H-8), 4.90 – 4.86 (2H, m, H-2 and H-6), 3.72 (3H, s, -OCH₃), 2.93 (1H, ddd, $J = 4.89, 12.43, 15.65$ Hz, H-4(a)), 2.54 (1H, dd, $J = 4.14, 15.65$ Hz, H-4(e)), 2.31 (1H, ddd, $J = 4.14, 12.43, 13.68$ Hz, H-3(a)), 2.13 (1H, dd, $J = 4.89, 13.68$ Hz, H-3(e)); ¹³C NMR (600 MHz, CDCl₃) δ ppm 25.52 (C-4), 29.52 (C-3), 55.86 (-OCH₃), 68.31 (C-8), 74.42 (C-2/6), 80.56 (C-2/6), 89.11, 94.55 (C-5), 126.61, 128.83, 128.89, 139.62, 140.30, 143.33, 234.44 (-Cr(CO)₃); MS m/z 376 (M⁺, 13.0), 344 (0.2), 320 (0.1), 292 (70.7), 277 (0.2), 256 (0.1), 240 (5.8), 225 (0.5), 209 (0.3), 188 (100.0), 173 (0.4), 146 (10.0), 137 (2.1), 121 (2.0), 104 (5.1).

S3. Refinement

The H atoms were positioned geometrically and refined using a riding model with fixed C—H distances of 0.93 Å (ArH) [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$], 1.00 Å (CH) [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$], 0.99 Å (CH₂) [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$] and 0.96 Å (CH₃) [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}$]. Initial positions of methyl H-atoms were obtained from fourier difference and refined as a fixed rotor.

The highest density peak is 0.25 located 0.66 Å from C1' and the deepest hole is -0.40 located at 0.50 Å from Cr.

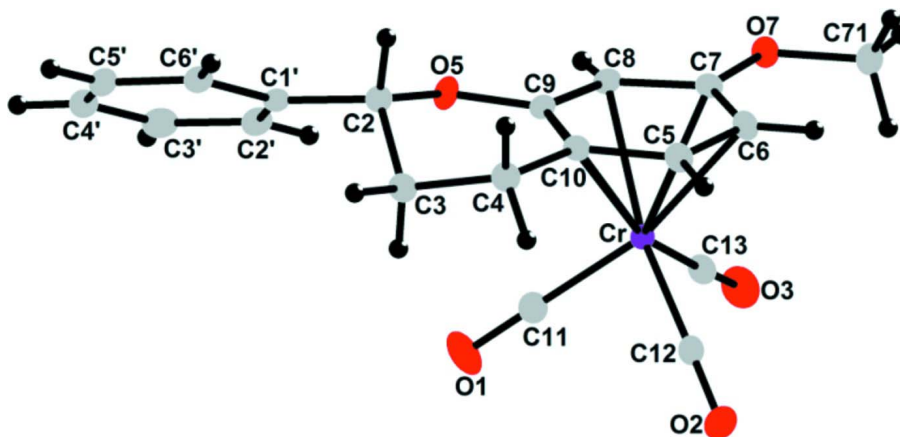


Figure 1

A view of (I) showing the atom-numbering scheme with displacement ellipsoids at the 30% probability level.

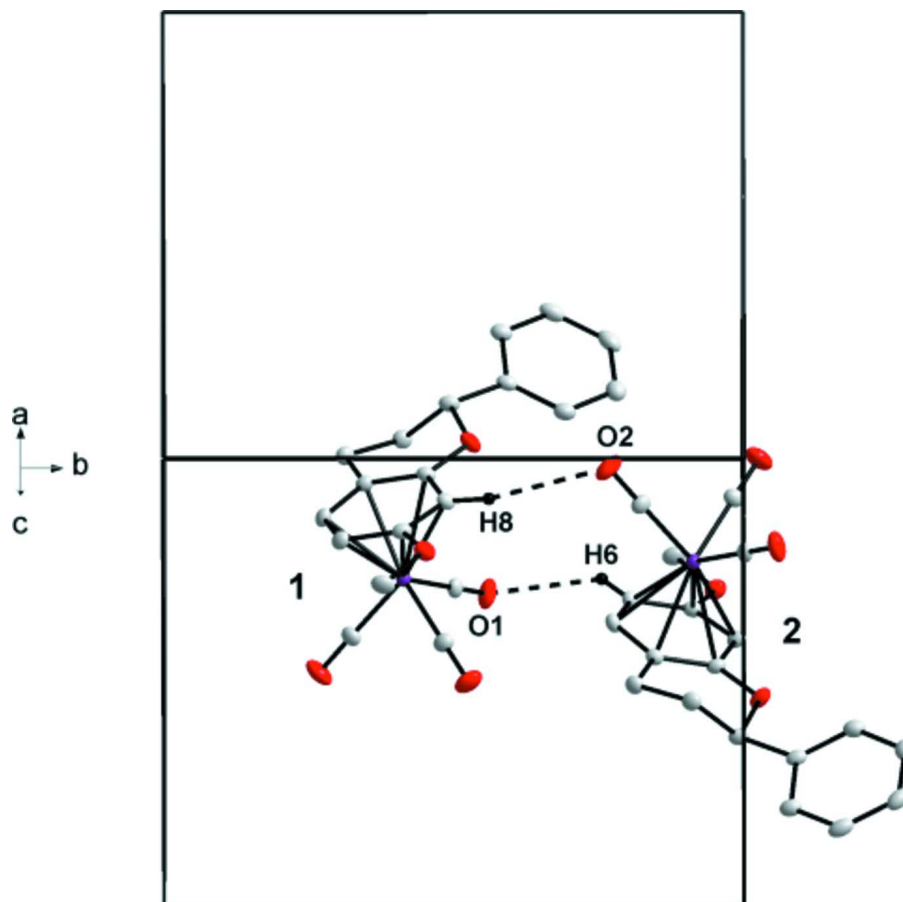


Figure 2

Indication of molecular packing in the unit-cell. Symmetry operators 1) $x; y; z$. 2) $1 - x; 0.5 + y; 1.5 - z$.

tricarbonyl(η^6 -7-methoxy-2-phenyl-3,4-dihydro-2H-1-benzopyran)chromium(0)

Crystal data

$[\text{Cr}(\text{C}_{16}\text{H}_{16}\text{O}_2)(\text{CO})_3]$

$M_r = 376.32$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 9.8422(2)\ \text{\AA}$

$b = 12.3850(3)\ \text{\AA}$

$c = 15.0146(3)\ \text{\AA}$

$\beta = 115.171(1)^\circ$

$V = 1656.42(6)\ \text{\AA}^3$

$Z = 4$

$F(000) = 776$

$D_x = 1.509\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 5285 reflections

$\theta = 2.2\text{--}28.3^\circ$

$\mu = 0.72\ \text{mm}^{-1}$

$T = 173\ \text{K}$

Prism, yellow

$0.41 \times 0.34 \times 0.24\ \text{mm}$

Data collection

Bruker APEXII CCD

diffractometer

φ and ω scans

Absorption correction: multi-scan

(Bruker, 2004)

$T_{\min} = 0.757$, $T_{\max} = 0.847$

12982 measured reflections

3985 independent reflections

3224 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$

$\theta_{\max} = 28^\circ$, $\theta_{\min} = 2.2^\circ$

$h = -11 \rightarrow 12$

$k = -16 \rightarrow 16$

$l = -19 \rightarrow 19$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.085$ $S = 1.07$

3985 reflections

227 parameters

0 restraints

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.0884P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.40 \text{ e } \text{\AA}^{-3}$ *Special details*

Experimental. The intensity data was collected on a Bruker Apex II CCD diffractometer using a frame width of 0.5° covering up to $\theta = 28^\circ$ with 100 % completeness accomplished.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1'	1.01976 (18)	0.59116 (14)	0.84890 (12)	0.0260 (3)
C2'	0.9566 (2)	0.69062 (15)	0.85055 (13)	0.0325 (4)
H2'	0.8506	0.6988	0.8213	0.039*
C2	0.92726 (18)	0.49179 (14)	0.80339 (12)	0.0268 (4)
H2	0.9703	0.4553	0.7618	0.032*
C3'	1.0487 (2)	0.77884 (16)	0.89526 (14)	0.0385 (4)
H3'	1.0052	0.8471	0.8958	0.046*
C3	0.92236 (18)	0.41073 (13)	0.87814 (12)	0.0276 (4)
H3A	0.8755	0.4443	0.9181	0.033*
H3B	1.0257	0.3886	0.923	0.033*
C4	0.83175 (18)	0.31200 (14)	0.82448 (12)	0.0272 (4)
H4A	0.8908	0.2687	0.798	0.033*
H4B	0.8107	0.2663	0.8713	0.033*
C4'	1.2029 (2)	0.76723 (16)	0.93867 (13)	0.0380 (4)
H4'	1.2654	0.8272	0.9696	0.046*
C5'	1.2657 (2)	0.66846 (16)	0.93694 (13)	0.0360 (4)
H5'	1.3717	0.6603	0.967	0.043*
C5	0.56441 (18)	0.27492 (13)	0.69612 (12)	0.0260 (4)
H5	0.5723	0.2037	0.7214	0.031*
C6	0.43161 (18)	0.30503 (13)	0.61485 (12)	0.0263 (3)
H6	0.3532	0.2541	0.5842	0.032*
C6'	1.17553 (18)	0.58135 (15)	0.89185 (12)	0.0295 (4)
H6'	1.2201	0.5138	0.89	0.035*
C7	0.41667 (18)	0.41122 (13)	0.57975 (11)	0.0247 (3)
C8	0.53223 (18)	0.48677 (13)	0.62755 (12)	0.0251 (3)
H8	0.5183	0.5606	0.6085	0.03*
C9	0.66817 (17)	0.45213 (14)	0.70352 (12)	0.0242 (3)
C10	0.68615 (17)	0.34612 (13)	0.74146 (12)	0.0237 (3)
C11	0.56076 (19)	0.50205 (13)	0.85013 (13)	0.0269 (4)

C12	0.42375 (19)	0.32289 (14)	0.82151 (13)	0.0295 (4)
C13	0.29284 (19)	0.48252 (14)	0.70530 (13)	0.0298 (4)
C71	0.17072 (19)	0.37892 (16)	0.45371 (13)	0.0335 (4)
H71A	0.2029	0.3181	0.4255	0.05*
H71B	0.0894	0.4179	0.401	0.05*
H71C	0.1349	0.3518	0.5013	0.05*
O1	0.61634 (16)	0.55726 (10)	0.91861 (10)	0.0436 (4)
O2	0.39278 (16)	0.26735 (12)	0.87217 (10)	0.0486 (4)
O3	0.17872 (15)	0.52563 (12)	0.67906 (11)	0.0482 (4)
O5	0.77684 (12)	0.52853 (9)	0.73998 (9)	0.0305 (3)
O7	0.29490 (12)	0.45045 (10)	0.50235 (8)	0.0303 (3)
Cr	0.47389 (3)	0.413031 (19)	0.743218 (18)	0.01990 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1'	0.0229 (8)	0.0363 (9)	0.0193 (8)	-0.0040 (7)	0.0096 (7)	0.0017 (7)
C2'	0.0282 (9)	0.0401 (10)	0.0317 (10)	-0.0012 (8)	0.0152 (8)	0.0017 (8)
C2	0.0198 (8)	0.0364 (9)	0.0228 (8)	-0.0006 (7)	0.0077 (7)	0.0013 (7)
C3'	0.0494 (11)	0.0344 (10)	0.0391 (11)	-0.0015 (9)	0.0259 (10)	0.0000 (8)
C3	0.0239 (8)	0.0319 (9)	0.0230 (8)	0.0030 (7)	0.0062 (7)	0.0046 (7)
C4	0.0241 (8)	0.0281 (8)	0.0265 (9)	0.0044 (7)	0.0079 (7)	0.0039 (7)
C4'	0.0444 (11)	0.0425 (11)	0.0286 (10)	-0.0176 (9)	0.0170 (9)	-0.0023 (8)
C5'	0.0278 (9)	0.0524 (12)	0.0263 (9)	-0.0110 (9)	0.0100 (8)	0.0034 (8)
C5	0.0286 (8)	0.0203 (8)	0.0280 (9)	0.0025 (7)	0.0110 (7)	-0.0027 (6)
C6	0.0261 (8)	0.0254 (8)	0.0256 (8)	-0.0011 (7)	0.0092 (7)	-0.0063 (7)
C6'	0.0248 (8)	0.0389 (10)	0.0247 (9)	-0.0013 (7)	0.0105 (7)	0.0037 (7)
C7	0.0209 (8)	0.0331 (9)	0.0195 (8)	0.0020 (7)	0.0081 (6)	0.0009 (7)
C8	0.0243 (8)	0.0279 (8)	0.0232 (8)	-0.0002 (7)	0.0102 (7)	0.0061 (7)
C9	0.0211 (8)	0.0306 (8)	0.0216 (8)	-0.0023 (7)	0.0098 (7)	0.0017 (7)
C10	0.0229 (8)	0.0258 (8)	0.0227 (8)	0.0021 (6)	0.0101 (7)	-0.0003 (6)
C11	0.0281 (9)	0.0203 (8)	0.0292 (9)	0.0040 (7)	0.0090 (7)	0.0041 (7)
C12	0.0291 (9)	0.0308 (9)	0.0269 (9)	-0.0074 (7)	0.0104 (7)	-0.0030 (7)
C13	0.0274 (9)	0.0331 (9)	0.0280 (9)	-0.0003 (7)	0.0108 (7)	-0.0054 (7)
C71	0.0206 (8)	0.0466 (11)	0.0286 (9)	-0.0041 (8)	0.0058 (7)	0.0003 (8)
O1	0.0546 (9)	0.0264 (6)	0.0327 (7)	0.0063 (6)	0.0020 (7)	-0.0075 (6)
O2	0.0537 (9)	0.0537 (9)	0.0381 (8)	-0.0232 (7)	0.0195 (7)	0.0062 (7)
O3	0.0302 (7)	0.0592 (9)	0.0495 (9)	0.0148 (7)	0.0115 (7)	-0.0084 (7)
O5	0.0203 (6)	0.0333 (7)	0.0295 (6)	-0.0058 (5)	0.0024 (5)	0.0110 (5)
O7	0.0231 (6)	0.0369 (7)	0.0243 (6)	-0.0018 (5)	0.0037 (5)	0.0050 (5)
Cr	0.02079 (14)	0.01811 (14)	0.02113 (14)	-0.00032 (10)	0.00923 (11)	0.00022 (10)

Geometric parameters (Å, °)

C1'—C2'	1.385 (3)	C6—C7	1.401 (2)
C1'—C6'	1.393 (2)	C6—Cr	2.2350 (16)
C1'—C2	1.510 (2)	C6—H6	0.95
C2'—C3'	1.396 (3)	C6'—H6'	0.95

C2'—H2'	0.95	C7—O7	1.3558 (19)
C2—O5	1.4513 (19)	C7—C8	1.411 (2)
C2—C3	1.522 (2)	C7—Cr	2.2729 (16)
C2—H2	1	C8—C9	1.406 (2)
C3'—C4'	1.381 (3)	C8—Cr	2.2424 (16)
C3'—H3'	0.95	C8—H8	0.95
C3—C4	1.526 (2)	C9—O5	1.3568 (19)
C3—H3A	0.99	C9—C10	1.412 (2)
C3—H3B	0.99	C9—Cr	2.2838 (16)
C4—C10	1.505 (2)	C10—Cr	2.2580 (16)
C4—H4A	0.99	C11—O1	1.160 (2)
C4—H4B	0.99	C11—Cr	1.8319 (17)
C4'—C5'	1.375 (3)	C12—O2	1.159 (2)
C4'—H4'	0.95	C12—Cr	1.8344 (18)
C5'—C6'	1.378 (2)	C13—O3	1.151 (2)
C5'—H5'	0.95	C13—Cr	1.8376 (18)
C5—C6	1.407 (2)	C71—O7	1.433 (2)
C5—C10	1.408 (2)	C71—H71A	0.98
C5—Cr	2.1808 (16)	C71—H71B	0.98
C5—H5	0.95	C71—H71C	0.98
C2'—C1'—C6'	118.91 (16)	O5—C9—C8	115.42 (14)
C2'—C1'—C2	122.96 (15)	O5—C9—C10	122.93 (14)
C6'—C1'—C2	118.12 (15)	C8—C9—C10	121.58 (15)
C1'—C2'—C3'	120.00 (16)	O5—C9—Cr	130.27 (11)
C1'—C2'—H2'	120	C8—C9—Cr	70.31 (9)
C3'—C2'—H2'	120	C10—C9—Cr	70.90 (9)
O5—C2—C1'	106.96 (13)	C5—C10—C9	116.89 (14)
O5—C2—C3	110.32 (13)	C5—C10—C4	122.62 (14)
C1'—C2—C3	113.95 (14)	C9—C10—C4	120.46 (14)
O5—C2—H2	108.5	C5—C10—Cr	68.55 (9)
C1'—C2—H2	108.5	C9—C10—Cr	72.88 (9)
C3—C2—H2	108.5	C4—C10—Cr	130.77 (11)
C4'—C3'—C2'	120.27 (18)	O1—C11—Cr	179.12 (15)
C4'—C3'—H3'	119.9	O2—C12—Cr	178.82 (16)
C2'—C3'—H3'	119.9	O3—C13—Cr	178.24 (16)
C2—C3—C4	109.51 (14)	O7—C71—H71A	109.5
C2—C3—H3A	109.8	O7—C71—H71B	109.5
C4—C3—H3A	109.8	H71A—C71—H71B	109.5
C2—C3—H3B	109.8	O7—C71—H71C	109.5
C4—C3—H3B	109.8	H71A—C71—H71C	109.5
H3A—C3—H3B	108.2	H71B—C71—H71C	109.5
C10—C4—C3	110.43 (13)	C9—O5—C2	117.07 (12)
C10—C4—H4A	109.6	C7—O7—C71	117.83 (13)
C3—C4—H4A	109.6	C11—Cr—C12	87.55 (7)
C10—C4—H4B	109.6	C11—Cr—C13	91.11 (7)
C3—C4—H4B	109.6	C12—Cr—C13	89.65 (8)
H4A—C4—H4B	108.1	C11—Cr—C5	130.80 (7)

C5'—C4'—C3'	119.72 (18)	C12—Cr—C5	89.36 (7)
C5'—C4'—H4'	120.1	C13—Cr—C5	137.98 (7)
C3'—C4'—H4'	120.1	C11—Cr—C6	164.20 (7)
C4'—C5'—C6'	120.35 (17)	C12—Cr—C6	100.73 (7)
C4'—C5'—H5'	119.8	C13—Cr—C6	102.28 (7)
C6'—C5'—H5'	119.8	C5—Cr—C6	37.14 (6)
C6—C5—C10	122.61 (15)	C11—Cr—C8	104.82 (7)
C6—C5—Cr	73.52 (9)	C12—Cr—C8	166.28 (7)
C10—C5—Cr	74.51 (9)	C13—Cr—C8	95.93 (7)
C6—C5—H5	118.7	C5—Cr—C8	78.10 (6)
C10—C5—H5	118.7	C6—Cr—C8	65.85 (6)
Cr—C5—H5	125	C11—Cr—C10	98.15 (7)
C7—C6—C5	119.02 (15)	C12—Cr—C10	106.65 (7)
C7—C6—Cr	73.38 (9)	C13—Cr—C10	161.49 (7)
C5—C6—Cr	69.34 (9)	C5—Cr—C10	36.94 (6)
C7—C6—H6	120.5	C6—Cr—C10	66.68 (6)
C5—C6—H6	120.5	C8—Cr—C10	66.26 (6)
Cr—C6—H6	129	C11—Cr—C7	139.56 (7)
C5'—C6'—C1'	120.73 (17)	C12—Cr—C7	132.48 (7)
C5'—C6'—H6'	119.6	C13—Cr—C7	84.71 (7)
C1'—C6'—H6'	119.6	C5—Cr—C7	65.78 (6)
O7—C7—C6	124.88 (15)	C6—Cr—C7	36.20 (6)
O7—C7—C8	115.21 (14)	C8—Cr—C7	36.40 (6)
C6—C7—C8	119.89 (14)	C10—Cr—C7	77.84 (6)
O7—C7—Cr	130.14 (11)	C11—Cr—C9	88.31 (7)
C6—C7—Cr	70.42 (9)	C12—Cr—C9	141.22 (7)
C8—C7—Cr	70.62 (9)	C13—Cr—C9	128.98 (7)
C9—C8—C7	119.56 (15)	C5—Cr—C9	65.09 (6)
C9—C8—Cr	73.51 (9)	C6—Cr—C9	76.78 (6)
C7—C8—Cr	72.98 (9)	C8—Cr—C9	36.18 (6)
C9—C8—H8	120.2	C10—Cr—C9	36.22 (6)
C7—C8—H8	120.2	C7—Cr—C9	64.57 (6)
Cr—C8—H8	124.9		
C6'—C1'—C2'—C3'	-0.4 (3)	C7—C6—Cr—C9	-64.80 (10)
C2—C1'—C2'—C3'	178.97 (16)	C5—C6—Cr—C9	66.02 (10)
C2'—C1'—C2—O5	17.3 (2)	C9—C8—Cr—C11	65.59 (11)
C6'—C1'—C2—O5	-163.39 (14)	C7—C8—Cr—C11	-165.45 (10)
C2'—C1'—C2—C3	-104.93 (18)	C9—C8—Cr—C12	-88.2 (3)
C6'—C1'—C2—C3	74.42 (19)	C7—C8—Cr—C12	40.8 (3)
C1'—C2'—C3'—C4'	-0.5 (3)	C9—C8—Cr—C13	158.32 (10)
O5—C2—C3—C4	61.99 (17)	C7—C8—Cr—C13	-72.72 (10)
C1'—C2—C3—C4	-177.69 (14)	C9—C8—Cr—C5	-63.83 (10)
C2—C3—C4—C10	-47.68 (18)	C7—C8—Cr—C5	65.13 (10)
C2'—C3'—C4'—C5'	0.6 (3)	C9—C8—Cr—C6	-100.86 (11)
C3'—C4'—C5'—C6'	0.3 (3)	C7—C8—Cr—C6	28.10 (9)
C10—C5—C6—C7	-2.7 (2)	C9—C8—Cr—C10	-26.92 (10)
Cr—C5—C6—C7	56.02 (14)	C7—C8—Cr—C10	102.04 (10)

C10—C5—C6—Cr	-58.68 (14)	C9—C8—Cr—C7	-128.96 (15)
C4'—C5'—C6'—C1'	-1.2 (3)	C7—C8—Cr—C9	128.96 (15)
C2'—C1'—C6'—C5'	1.2 (3)	C5—C10—Cr—C11	155.21 (10)
C2—C1'—C6'—C5'	-178.16 (16)	C9—C10—Cr—C11	-75.77 (10)
C5—C6—C7—O7	-179.96 (15)	C4—C10—Cr—C11	40.00 (16)
Cr—C6—C7—O7	-125.88 (16)	C5—C10—Cr—C12	65.34 (11)
C5—C6—C7—C8	-1.7 (2)	C9—C10—Cr—C12	-165.64 (10)
Cr—C6—C7—C8	52.36 (14)	C4—C10—Cr—C12	-49.87 (16)
C5—C6—C7—Cr	-54.07 (13)	C5—C10—Cr—C13	-85.5 (2)
O7—C7—C8—C9	-174.85 (14)	C9—C10—Cr—C13	43.5 (3)
C6—C7—C8—C9	6.7 (2)	C4—C10—Cr—C13	159.3 (2)
Cr—C7—C8—C9	59.01 (14)	C9—C10—Cr—C5	129.02 (14)
O7—C7—C8—Cr	126.14 (13)	C4—C10—Cr—C5	-115.21 (18)
C6—C7—C8—Cr	-52.27 (14)	C5—C10—Cr—C6	-29.40 (9)
C7—C8—C9—O5	175.10 (14)	C9—C10—Cr—C6	99.61 (10)
Cr—C8—C9—O5	-126.16 (14)	C4—C10—Cr—C6	-144.62 (16)
C7—C8—C9—C10	-7.7 (2)	C5—C10—Cr—C8	-102.12 (10)
Cr—C8—C9—C10	51.09 (14)	C9—C10—Cr—C8	26.90 (9)
C7—C8—C9—Cr	-58.74 (14)	C4—C10—Cr—C8	142.67 (16)
C6—C5—C10—C9	1.9 (2)	C5—C10—Cr—C7	-65.70 (10)
Cr—C5—C10—C9	-56.37 (13)	C9—C10—Cr—C7	63.32 (10)
C6—C5—C10—C4	-176.23 (15)	C4—C10—Cr—C7	179.09 (16)
Cr—C5—C10—C4	125.55 (15)	C5—C10—Cr—C9	-129.02 (14)
C6—C5—C10—Cr	58.22 (14)	C4—C10—Cr—C9	115.77 (18)
O5—C9—C10—C5	-179.62 (15)	O7—C7—Cr—C11	-85.11 (17)
C8—C9—C10—C5	3.3 (2)	C6—C7—Cr—C11	155.29 (11)
Cr—C9—C10—C5	54.18 (13)	C8—C7—Cr—C11	21.99 (14)
O5—C9—C10—C4	-1.5 (2)	O7—C7—Cr—C12	85.04 (17)
C8—C9—C10—C4	-178.53 (15)	C6—C7—Cr—C12	-34.57 (13)
Cr—C9—C10—C4	-127.70 (15)	C8—C7—Cr—C12	-167.87 (11)
O5—C9—C10—Cr	126.20 (16)	O7—C7—Cr—C13	0.38 (15)
C8—C9—C10—Cr	-50.83 (14)	C6—C7—Cr—C13	-119.22 (11)
C3—C4—C10—C5	-163.05 (15)	C8—C7—Cr—C13	107.48 (11)
C3—C4—C10—C9	18.9 (2)	O7—C7—Cr—C5	149.67 (16)
C3—C4—C10—Cr	-74.28 (18)	C6—C7—Cr—C5	30.06 (9)
C8—C9—O5—C2	-167.67 (14)	C8—C7—Cr—C5	-103.24 (10)
C10—C9—O5—C2	15.1 (2)	O7—C7—Cr—C6	119.60 (19)
Cr—C9—O5—C2	107.28 (16)	C8—C7—Cr—C6	-133.30 (14)
C1'—C2—O5—C9	-169.74 (13)	O7—C7—Cr—C8	-107.10 (18)
C3—C2—O5—C9	-45.31 (19)	C6—C7—Cr—C8	133.30 (14)
C6—C7—O7—C71	1.2 (2)	O7—C7—Cr—C10	-173.42 (16)
C8—C7—O7—C71	-177.07 (14)	C6—C7—Cr—C10	66.97 (10)
Cr—C7—O7—C71	-91.82 (17)	C8—C7—Cr—C10	-66.33 (10)
C6—C5—Cr—C11	-164.93 (10)	O7—C7—Cr—C9	-137.65 (16)
C10—C5—Cr—C11	-33.24 (13)	C6—C7—Cr—C9	102.75 (10)
C6—C5—Cr—C12	108.86 (11)	C8—C7—Cr—C9	-30.55 (9)
C10—C5—Cr—C12	-119.45 (10)	O5—C9—Cr—C11	-11.15 (14)
C6—C5—Cr—C13	20.10 (15)	C8—C9—Cr—C11	-118.27 (11)

C10—C5—Cr—C13	151.78 (11)	C10—C9—Cr—C11	106.27 (10)
C10—C5—Cr—C6	131.69 (15)	O5—C9—Cr—C12	-95.13 (17)
C6—C5—Cr—C8	-65.53 (10)	C8—C9—Cr—C12	157.75 (12)
C10—C5—Cr—C8	66.16 (10)	C10—C9—Cr—C12	22.30 (15)
C6—C5—Cr—C10	-131.69 (15)	O5—C9—Cr—C13	78.91 (16)
C6—C5—Cr—C7	-29.35 (10)	C8—C9—Cr—C13	-28.21 (13)
C10—C5—Cr—C7	102.34 (10)	C10—C9—Cr—C13	-163.67 (10)
C6—C5—Cr—C9	-101.28 (11)	O5—C9—Cr—C5	-148.41 (16)
C10—C5—Cr—C9	30.41 (9)	C8—C9—Cr—C5	104.47 (11)
C7—C6—Cr—C11	-84.6 (3)	C10—C9—Cr—C5	-30.99 (9)
C5—C6—Cr—C11	46.3 (3)	O5—C9—Cr—C6	174.13 (15)
C7—C6—Cr—C12	154.79 (10)	C8—C9—Cr—C6	67.01 (10)
C5—C6—Cr—C12	-74.38 (11)	C10—C9—Cr—C6	-68.45 (10)
C7—C6—Cr—C13	62.79 (11)	O5—C9—Cr—C8	107.12 (18)
C5—C6—Cr—C13	-166.38 (11)	C10—C9—Cr—C8	-135.45 (15)
C7—C6—Cr—C5	-130.82 (15)	O5—C9—Cr—C10	-117.42 (18)
C7—C6—Cr—C8	-28.25 (9)	C8—C9—Cr—C10	135.45 (15)
C5—C6—Cr—C8	102.57 (11)	O5—C9—Cr—C7	137.85 (16)
C7—C6—Cr—C10	-101.56 (10)	C8—C9—Cr—C7	30.73 (10)
C5—C6—Cr—C10	29.26 (9)	C10—C9—Cr—C7	-104.73 (10)
C5—C6—Cr—C7	130.82 (15)		
