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Tricarbonyl(η^6 -4',7-dimethoxyisoflavone)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 ^bDepartment of Pharmacology, University of Pretoria, PO Box 2034, Pretoria 0001, South Africa
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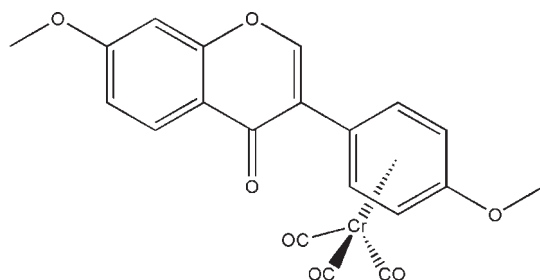
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.044; wR factor = 0.117; data-to-parameter ratio = 16.3.

The metal atom of the $\text{Cr}(\text{CO})_3$ unit of the title compound, $[\text{Cr}(\text{C}_{17}\text{H}_{14}\text{O}_4)(\text{CO})_3]$, is coordinated to the methoxyphenyl ring of the isoflavone ligand; the $\text{Cr}(\text{CO})_3$ unit exhibits a three-legged piano-stool conformation. The aromatic ring of the methoxyphenyl group is twisted by 42.49 (9) $^\circ$ with respect to the γ -pyrone ring. In the fused-ring, the dihedral angle between the phenylene and γ -pyrone rings is 3.08 (13) $^\circ$.

Related literature

For tricarbonyl(arene)chromium complexes in regioselective reactions, see: Dominique *et al.* (1999). For their photochromic properties, see: Hanneschlager *et al.* (1999). For $\text{Cr}(\text{CO})_3$ complexation to the aromatic ring of flavanone, see: Dominique *et al.* (1999). For $\text{Cr}(\text{CO})_3$ complexation to (1,3-dimethoxybenzene), see: Zeller *et al.* (2004). For comparison bond distances, see: Allen (2002). For the synthesis of 4',7-dimethoxyisoflavone, see: Thakkar & Cushman (1995).



Experimental

Crystal data

 $[\text{Cr}(\text{C}_{17}\text{H}_{14}\text{O}_4)(\text{CO})_3]$ $M_r = 418.31$ Monoclinic, $P2_1/c$
 $a = 12.3454$ (7) Å
 $b = 17.9984$ (8) Å
 $c = 7.9988$ (4) Å
 $\beta = 103.733$ (2) $^\circ$
 $V = 1726.50$ (15) Å³ $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.71$ mm⁻¹
 $T = 173$ K
 $0.43 \times 0.23 \times 0.10$ mm

Data collection

Bruker APEXII CCD
diffractometer
Absorption correction: multi-scan
(SADABS; Bruker, 2004)
 $T_{\min} = 0.751$, $T_{\max} = 0.933$ 9320 measured reflections
4145 independent reflections
3393 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.117$
 $S = 1.08$
4145 reflections255 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.86$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.41$ e Å⁻³

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT-Plus (Bruker, 2004); data reduction: SAINT-Plus; 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2656).

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Acta Cryst. (2009). E65, m1343 [doi:10.1107/S1600536809040537]

Tricarbonyl(η^6 -4',7-dimethoxyisoflavone)chromium(0)

J. H. van Tonder, B. C. B. Bezuidenhoudt and J. M. Janse van Rensburg

Comment

Tricarbonyl(arene)chromium complexes have received much attention due to their use as intermediates in regioselective reactions (Dominique *et al.*, 1999), as well as for their photochromic properties (Hanneschlager *et al.*, 1999).

The title compound, (I), $[\text{Cr}(\text{CO})_3(\text{C}_{17}\text{H}_{14}\text{O}_4)]$, where $(\text{C}_{17}\text{H}_{14}\text{O}_4) = 4',7\text{-dimethoxyisoflavone}$, crystallized in the monoclinic space group $P2_1/c$, with $Z = 4$ (Fig. 1). For the title compound the molecular structure displays the $\text{Cr}(\text{CO})_3$ moiety complexation to the phenyl ring, exhibiting the known three-legged piano-stool conformation. This conformation is expected for a tricarbonyl-metal with an η^6 -coordinated arene. The $\text{Cr}-\text{C}(\text{arene})$ distances range from 2.188 (2) to 2.262 (2) Å. The longest $\text{Cr}-\text{C}(\text{arene})$ bond is $\text{Cr}-\text{C4}'$, that in turn is bonded to the $\text{O4}'-\text{C41}'$ methoxy group. This bond elongation is probably due to the methoxy group that weakens the π -interaction ability of $\text{C4}'$ towards the chromium metal centre. The Cr -arene(centroid) distance is 1.7205 (4) Å. The $\text{Cr}-\text{C}(\text{carbonyl})$ bond distances range from 1.827 (3) to 1.855 (3) Å and the carbonyl distances of $\text{C11}-\text{O1}$, $\text{C12}-\text{O2}$ and $\text{C13}-\text{O3}$ are 1.158 (3), 1.154 (3) and 1.150 (3) Å respectively. These distances are within the normal range, see Allen (2002). The phenyl ring is essentially planar (r.m.s of fitted atoms $\text{C1}'-\text{C6}' = 0.0119$ Å). Slight molecular disorder is displayed by a twist in the isoflavone backbone, that forms a dihedral angle of 42.49 (9)° between the phenyl and γ -pyrone ring and a dihedral angle of 41.1 (1)° between the phenyl and the benzopyrone ring system. A dihedral angle of 3.08 (13)° is also present between the benzene and the γ -pyrone ring, with a r.m.s of fitted atoms $\text{C2}-\text{C10}$ and O5 of 0.0387 Å. The $\text{O4}'-\text{C41}'$ methoxy group on the phenyl ring bends towards the $\text{Cr}(\text{CO})_3$ moiety, forming the $\text{C5}'-\text{C4}'-\text{O4}'-\text{C41}'$ torsion angle of 15.9 (4)°. The $\text{O7}-\text{C71}$ methoxy group on the benzene ring is also slightly displaced from the benzene ring plane, shown by the $\text{C8}-\text{C7}-\text{O7}-\text{C71}$ torsion angle of 175.0 (3)°. Other molecular geometrical parameters is in good agreement with literature values, see Allen (2002). Selected geometrical parameters is presented in Table 1.

As illustrated in Fig. 2 the molecular packing is such that a benzene ring of one molecule is above the γ -pyrone ring of a neighbouring molecule, separated by a plane to plane distance of 3.369 Å and a centroid to centroid distance of 4.281 Å.

Experimental

4',7-Dimethoxyisoflavone was prepared as previously described by Thakkar & Cushman (1995). A solution of 4',7-Dimethoxyisoflavone (1.28 g, 4.5 mmol) and $\text{Cr}(\text{CO})_6$ (1.00 g, 4.6 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(η^6 -4',7-dimethoxyisoflavone)-chromium(0) (0.48 g; 25.0%) as a yellow solid. Recrystallization from diethyl ether yielded yellow cuboidal crystals.

R_f 0.18 (Hexane: Acetone; 8:2); Mp 127.0 °C; Note: A, B and C-ring labelling refers to the benzene, phenyl and γ -pyrone rings respectively. ^1H NMR (600 MHz, CDCl_3) δ p.p.m. 8.15 (1H, d, $J = 9.04$ Hz, H-5), 8.09 (1H, s, H-2), 7.01 (1H, dd, J

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= 1.88, 9.04 Hz, H-6), 6.86 (1H, d, J = 1.88 Hz, H-8), 5.85 (2H, d, J = 6.78 Hz, H-2' and H-6'), 5.21 (2H, d, J = 6.78 Hz, H-3' and H-5'), 3.92 (3H, s, -OCH₃), 3.75 (3H, s, -OCH₃); ¹³C NMR (151 MHz, CDCl₃) δ p.p.m. 55.88 (-OCH₃), 56.06 (-OCH₃), 77.37 (C-3' and C-5'), 94.71, 97.63 (C-2' and C-6'), 100.45 (C-8), 115.32 (C-6), 117.67, 121.16, 127.71 (C-5), 143.39 (C(i)-OCH₃ B-ring), 154.78 (C-2), 158.07, 164.60, 175.26 (C-4), 232.89 (Cr—CO); MS (MS Scheme 3) m/z 362 (M⁺-2CO, 0.5%), 343 (2.1), 282 (100.0), 267 (20.8), 252 (3.0), 239 (10.9), 224 (3.7), 211 (3.8), 196 (3.5), 183 (1.2), 168 (2.9), 150 (12.9), 141 (6.1), 131 (69.5), 122 (10.7), 107 (7.9), 103 (2.4).

Refinement

The H atoms were positioned geometrically and refined using a riding model with fixed C—H distances of 0.93 Å (CH) [*U*_{iso}(H) = 1.2*U*_{eq}] and 0.96 Å (CH₃) [*U*_{iso}(H) = 1.5*U*_{eq}] respectively. Initial positions of methyl H-atoms were obtained from fourier difference and refined as a fixed rotor.

The highest density peak is 0.86 located 0.96 Å from O1 and the deepest hole is -0.41 located at 0.54 Å from Cr.

Figures

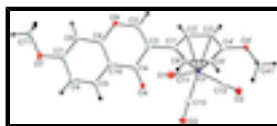


Fig. 1. A view of (I) showing the atom-numbering scheme with displacement ellipsoids at the 30% probability level.

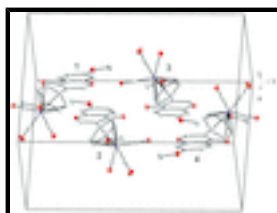


Fig. 2. Indication of molecular packing in the unit-cell. Symmetry operators 1) *x*; *y*; *z*. 2) *x*; 0.5 - *y*; -1/2 + *z*. 3) 1 - *x*; 1/2 + *y*; 1.5 - *z*. 4) 1 - *x*; 1 - *y*; 1 - *z*.

Tricarbonyl(η⁶-4',7-dimethoxyisoflavone)chromium(0)

Crystal data

[Cr(C₁₇H₁₄O₄)(CO)₃]

M_r = 418.31

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

a = 12.3454 (7) Å

b = 17.9984 (8) Å

c = 7.9988 (4) Å

β = 103.733 (2)°

V = 1726.50 (15) Å³

Z = 4

*F*₀₀₀ = 856

D_x = 1.609 Mg m⁻³

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 3261 reflections

θ = 2.8–28.3°

μ = 0.71 mm⁻¹

T = 173 K

Block, yellow

0.43 × 0.23 × 0.1 mm

Data collection

Bruker APEXII CCD diffractometer	3393 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.027$
$T = 173$ K	$\theta_{\text{max}} = 28^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.7^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$h = -16 \rightarrow 9$
$T_{\text{min}} = 0.751$, $T_{\text{max}} = 0.933$	$k = -23 \rightarrow 21$
9320 measured reflections	$l = -10 \rightarrow 9$
4145 independent reflections	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 2.2801P]$
$R[F^2 > 2\sigma(F^2)] = 0.044$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.117$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.08$	$\Delta\rho_{\text{max}} = 0.86 \text{ e } \text{\AA}^{-3}$
4145 reflections	$\Delta\rho_{\text{min}} = -0.41 \text{ e } \text{\AA}^{-3}$
255 parameters	Extinction correction: none

Special details

Experimental. The intensity data was collected on a Bruker Apex II CCD diffractometer using an exposure time of 10 s/frame. The 509 frames were collected with a frame width of 0.5° covering up to $\theta = 28^\circ$ with 99.4% completeness accomplished.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cr	0.67866 (3)	0.08506 (2)	0.83271 (5)	0.01520 (12)
C71	-0.0137 (3)	0.34474 (18)	0.1743 (5)	0.0415 (8)
H71A	-0.0177	0.3606	0.29	0.062*
H71B	-0.0845	0.3559	0.0926	0.062*
H71C	0.0469	0.3713	0.1402	0.062*
C1'	0.5514 (2)	0.16695 (13)	0.8698 (3)	0.0170 (5)
C2	0.4317 (2)	0.26583 (14)	0.7185 (3)	0.0210 (5)
H2	0.4859	0.2986	0.7838	0.025*
C2'	0.6553 (2)	0.20306 (14)	0.8800 (3)	0.0198 (5)
H2'	0.6591	0.2432	0.8046	0.024*

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C3'	0.7524 (2)	0.18039 (14)	0.9997 (3)	0.0212 (5)
H3'	0.8208	0.2058	1.0066	0.025*
C3	0.4495 (2)	0.19286 (14)	0.7448 (3)	0.0185 (5)
C4	0.3661 (2)	0.13983 (14)	0.6510 (3)	0.0192 (5)
C4'	0.7479 (2)	0.11970 (14)	1.1093 (3)	0.0201 (5)
C5	0.1963 (2)	0.13098 (15)	0.4082 (4)	0.0248 (6)
H5	0.2022	0.0784	0.4126	0.03*
C5'	0.6471 (2)	0.08121 (14)	1.0971 (3)	0.0182 (5)
H5'	0.6442	0.0395	1.1688	0.022*
C6	0.1094 (2)	0.16347 (16)	0.2927 (4)	0.0272 (6)
H6	0.0567	0.1336	0.2156	0.033*
C6'	0.5506 (2)	0.10521 (14)	0.9775 (3)	0.0185 (5)
H6'	0.4828	0.0789	0.9692	0.022*
C7	0.0986 (2)	0.24107 (16)	0.2887 (4)	0.0246 (6)
C8	0.1774 (2)	0.28543 (15)	0.3938 (4)	0.0241 (6)
H8	0.1713	0.338	0.3893	0.029*
C9	0.2667 (2)	0.25058 (14)	0.5070 (3)	0.0196 (5)
C10	0.2769 (2)	0.17415 (14)	0.5204 (3)	0.0196 (5)
C11	0.6772 (2)	0.11149 (14)	0.6116 (4)	0.0224 (5)
C12	0.8108 (2)	0.03640 (15)	0.8470 (4)	0.0252 (6)
C13	0.6030 (2)	-0.00087 (14)	0.7453 (3)	0.0213 (5)
C41'	0.8552 (3)	0.03005 (17)	1.2974 (4)	0.0326 (7)
H41A	0.8072	0.0277	1.3788	0.049*
H41B	0.9328	0.0212	1.3587	0.049*
H41C	0.8319	-0.0079	1.2082	0.049*
O1	0.6770 (2)	0.12958 (12)	0.4726 (3)	0.0362 (5)
O2	0.89489 (18)	0.00706 (14)	0.8540 (3)	0.0461 (6)
O3	0.55781 (18)	-0.05454 (11)	0.6900 (3)	0.0318 (5)
O4'	0.84603 (16)	0.10203 (11)	1.2190 (2)	0.0260 (4)
O4	0.36907 (17)	0.07292 (10)	0.6784 (3)	0.0271 (4)
O5	0.34364 (15)	0.29706 (10)	0.6077 (2)	0.0227 (4)
O7	0.00685 (17)	0.26705 (12)	0.1746 (3)	0.0347 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cr	0.0162 (2)	0.01354 (19)	0.0156 (2)	-0.00044 (15)	0.00332 (14)	0.00014 (15)
C71	0.0309 (16)	0.0381 (18)	0.050 (2)	0.0135 (14)	-0.0004 (15)	0.0107 (15)
C1'	0.0199 (12)	0.0159 (11)	0.0154 (11)	0.0018 (9)	0.0048 (9)	-0.0032 (9)
C2	0.0205 (12)	0.0201 (12)	0.0211 (12)	0.0020 (10)	0.0024 (10)	0.0009 (10)
C2'	0.0242 (13)	0.0139 (11)	0.0208 (12)	-0.0011 (9)	0.0047 (10)	-0.0009 (9)
C3'	0.0204 (12)	0.0188 (12)	0.0228 (13)	-0.0029 (10)	0.0020 (10)	-0.0026 (10)
C3	0.0185 (12)	0.0199 (12)	0.0177 (12)	0.0012 (9)	0.0058 (10)	0.0018 (10)
C4	0.0187 (12)	0.0188 (12)	0.0203 (12)	0.0014 (9)	0.0054 (10)	0.0015 (10)
C4'	0.0218 (12)	0.0192 (12)	0.0173 (12)	0.0002 (10)	0.0007 (10)	-0.0037 (10)
C5	0.0249 (13)	0.0200 (13)	0.0277 (14)	-0.0016 (10)	0.0028 (11)	0.0021 (11)
C5'	0.0272 (13)	0.0156 (11)	0.0124 (11)	0.0003 (10)	0.0060 (9)	0.0014 (9)
C6	0.0227 (13)	0.0291 (14)	0.0268 (14)	-0.0044 (11)	0.0002 (11)	0.0029 (12)

C6'	0.0191 (12)	0.0193 (12)	0.0179 (12)	-0.0008 (9)	0.0059 (10)	-0.0012 (9)
C7	0.0173 (12)	0.0293 (14)	0.0269 (14)	0.0042 (10)	0.0049 (10)	0.0086 (11)
C8	0.0241 (13)	0.0209 (13)	0.0268 (14)	0.0044 (10)	0.0049 (11)	0.0062 (11)
C9	0.0187 (12)	0.0215 (12)	0.0190 (12)	0.0012 (10)	0.0051 (10)	0.0006 (10)
C10	0.0187 (12)	0.0198 (12)	0.0209 (12)	0.0004 (9)	0.0056 (10)	0.0027 (10)
C11	0.0237 (13)	0.0178 (12)	0.0247 (14)	-0.0014 (10)	0.0038 (10)	-0.0025 (10)
C12	0.0245 (13)	0.0227 (13)	0.0260 (14)	0.0002 (11)	0.0013 (11)	-0.0061 (11)
C13	0.0242 (13)	0.0210 (13)	0.0199 (12)	-0.0013 (10)	0.0073 (10)	0.0001 (10)
C41'	0.0289 (15)	0.0312 (15)	0.0324 (16)	0.0028 (12)	-0.0033 (12)	0.0096 (13)
O1	0.0576 (15)	0.0321 (11)	0.0212 (10)	-0.0032 (10)	0.0140 (10)	0.0037 (9)
O2	0.0268 (12)	0.0449 (14)	0.0615 (16)	0.0122 (10)	0.0002 (11)	-0.0138 (12)
O3	0.0364 (12)	0.0229 (10)	0.0373 (12)	-0.0090 (9)	0.0110 (9)	-0.0063 (9)
O4'	0.0216 (9)	0.0258 (10)	0.0255 (10)	-0.0009 (7)	-0.0041 (8)	0.0029 (8)
O4	0.0275 (10)	0.0177 (9)	0.0312 (11)	-0.0030 (7)	-0.0030 (8)	0.0048 (8)
O5	0.0232 (9)	0.0172 (9)	0.0253 (10)	0.0024 (7)	0.0009 (8)	0.0008 (7)
O7	0.0233 (10)	0.0340 (12)	0.0410 (13)	0.0039 (8)	-0.0038 (9)	0.0103 (10)

Geometric parameters (Å, °)

Cr—C11	1.827 (3)	C4—C10	1.463 (3)
Cr—C12	1.831 (3)	C4'—O4'	1.354 (3)
Cr—C13	1.855 (3)	C4'—C5'	1.407 (4)
Cr—C2'	2.188 (2)	C5—C6	1.369 (4)
Cr—C6'	2.200 (2)	C5—C10	1.405 (4)
Cr—C1'	2.225 (2)	C5—H5	0.95
Cr—C3'	2.231 (3)	C5'—C6'	1.407 (3)
Cr—C5'	2.241 (2)	C5'—H5'	0.95
Cr—C4'	2.262 (2)	C6—C7	1.403 (4)
C71—O7	1.421 (4)	C6—H6	0.95
C71—H71A	0.98	C6'—H6'	0.95
C71—H71B	0.98	C7—O7	1.358 (3)
C71—H71C	0.98	C7—C8	1.379 (4)
C1'—C6'	1.407 (3)	C8—C9	1.398 (4)
C1'—C2'	1.423 (3)	C8—H8	0.95
C1'—C3	1.484 (3)	C9—O5	1.374 (3)
C2—C3	1.340 (4)	C9—C10	1.383 (4)
C2—O5	1.351 (3)	C11—O1	1.158 (3)
C2—H2	0.95	C12—O2	1.154 (3)
C2'—C3'	1.406 (4)	C13—O3	1.150 (3)
C2'—H2'	0.95	C41'—O4'	1.432 (3)
C3'—C4'	1.410 (4)	C41'—H41A	0.98
C3'—H3'	0.95	C41'—H41B	0.98
C3—C4	1.472 (3)	C41'—H41C	0.98
C4—O4	1.223 (3)		
C11—Cr—C12	89.38 (12)	C4'—C3'—Cr	72.89 (14)
C11—Cr—C13	87.92 (12)	C2'—C3'—H3'	120.2
C12—Cr—C13	89.19 (12)	C4'—C3'—H3'	120.2
C11—Cr—C2'	86.74 (11)	Cr—C3'—H3'	129.5
C12—Cr—C2'	127.26 (11)	C2—C3—C4	119.1 (2)

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C13—Cr—C2'	143.03 (11)	C2—C3—C1'	119.7 (2)
C11—Cr—C6'	128.55 (11)	C4—C3—C1'	121.2 (2)
C12—Cr—C6'	141.88 (11)	O4—C4—C10	121.9 (2)
C13—Cr—C6'	88.62 (10)	O4—C4—C3	124.0 (2)
C2'—Cr—C6'	66.94 (10)	C10—C4—C3	114.0 (2)
C11—Cr—C1'	96.39 (11)	O4'—C4'—C5'	124.7 (2)
C12—Cr—C1'	162.83 (11)	O4'—C4'—C3'	115.1 (2)
C13—Cr—C1'	107.12 (10)	C5'—C4'—C3'	120.2 (2)
C2'—Cr—C1'	37.61 (9)	O4'—C4'—Cr	129.89 (18)
C6'—Cr—C1'	37.09 (9)	C5'—C4'—Cr	70.96 (14)
C11—Cr—C3'	106.73 (11)	C3'—C4'—Cr	70.53 (15)
C12—Cr—C3'	95.71 (11)	C6—C5—C10	121.1 (3)
C13—Cr—C3'	164.55 (11)	C6—C5—H5	119.4
C2'—Cr—C3'	37.07 (9)	C10—C5—H5	119.4
C6'—Cr—C3'	78.48 (10)	C6'—C5'—C4'	119.2 (2)
C1'—Cr—C3'	67.15 (9)	C6'—C5'—Cr	69.97 (14)
C11—Cr—C5'	163.24 (11)	C4'—C5'—Cr	72.62 (14)
C12—Cr—C5'	106.15 (11)	C6'—C5'—H5'	120.4
C13—Cr—C5'	98.36 (10)	C4'—C5'—H5'	120.4
C2'—Cr—C5'	78.88 (9)	Cr—C5'—H5'	129.3
C6'—Cr—C5'	36.93 (9)	C5—C6—C7	119.7 (3)
C1'—Cr—C5'	66.93 (9)	C5—C6—H6	120.2
C3'—Cr—C5'	66.21 (9)	C7—C6—H6	120.2
C11—Cr—C4'	142.14 (11)	C5'—C6'—C1'	122.1 (2)
C12—Cr—C4'	86.93 (11)	C5'—C6'—Cr	73.10 (14)
C13—Cr—C4'	129.64 (11)	C1'—C6'—Cr	72.42 (14)
C2'—Cr—C4'	66.30 (9)	C5'—C6'—H6'	119
C6'—Cr—C4'	65.90 (9)	C1'—C6'—H6'	119
C1'—Cr—C4'	78.58 (9)	Cr—C6'—H6'	127.7
C3'—Cr—C4'	36.57 (9)	O7—C7—C8	124.4 (3)
C5'—Cr—C4'	36.43 (9)	O7—C7—C6	114.7 (3)
O7—C71—H71A	109.5	C8—C7—C6	120.9 (2)
O7—C71—H71B	109.5	C7—C8—C9	117.9 (2)
H71A—C71—H71B	109.5	C7—C8—H8	121
O7—C71—H71C	109.5	C9—C8—H8	121
H71A—C71—H71C	109.5	O5—C9—C10	121.5 (2)
H71B—C71—H71C	109.5	O5—C9—C8	115.8 (2)
C6'—C1'—C2'	117.5 (2)	C10—C9—C8	122.7 (2)
C6'—C1'—C3	122.2 (2)	C9—C10—C5	117.6 (2)
C2'—C1'—C3	120.2 (2)	C9—C10—C4	121.0 (2)
C6'—C1'—Cr	70.49 (14)	C5—C10—C4	121.4 (2)
C2'—C1'—Cr	69.80 (14)	O1—C11—Cr	178.7 (2)
C3—C1'—Cr	129.11 (17)	O2—C12—Cr	178.4 (3)
C3—C2—O5	126.0 (2)	O3—C13—Cr	178.8 (2)
C3—C2—H2	117	O4'—C41'—H41A	109.5
O5—C2—H2	117	O4'—C41'—H41B	109.5
C3'—C2'—C1'	121.2 (2)	H41A—C41'—H41B	109.5
C3'—C2'—Cr	73.13 (15)	O4'—C41'—H41C	109.5
C1'—C2'—Cr	72.59 (14)	H41A—C41'—H41C	109.5

C3'—C2'—H2'	119.4	H41B—C41'—H41C	109.5
C1'—C2'—H2'	119.4	C4'—O4'—C41'	117.5 (2)
Cr—C2'—H2'	126.9	C2—O5—C9	117.9 (2)
C2'—C3'—C4'	119.7 (2)	C7—O7—C71	117.4 (2)
C2'—C3'—Cr	69.80 (14)		
C11—Cr—C1'—C6'	153.08 (16)	C5'—Cr—C4'—O4'	119.8 (3)
C12—Cr—C1'—C6'	-97.9 (4)	C11—Cr—C4'—C5'	152.16 (18)
C13—Cr—C1'—C6'	63.30 (16)	C12—Cr—C4'—C5'	-122.82 (17)
C2'—Cr—C1'—C6'	-130.8 (2)	C13—Cr—C4'—C5'	-36.4 (2)
C3'—Cr—C1'—C6'	-101.42 (16)	C2'—Cr—C4'—C5'	103.89 (16)
C5'—Cr—C1'—C6'	-28.66 (14)	C6'—Cr—C4'—C5'	29.69 (14)
C4'—Cr—C1'—C6'	-64.94 (15)	C1'—Cr—C4'—C5'	66.45 (15)
C11—Cr—C1'—C2'	-76.17 (16)	C3'—Cr—C4'—C5'	133.3 (2)
C12—Cr—C1'—C2'	32.8 (4)	C11—Cr—C4'—C3'	18.8 (2)
C13—Cr—C1'—C2'	-165.95 (15)	C12—Cr—C4'—C3'	103.85 (17)
C6'—Cr—C1'—C2'	130.8 (2)	C13—Cr—C4'—C3'	-169.74 (16)
C3'—Cr—C1'—C2'	29.33 (15)	C2'—Cr—C4'—C3'	-29.44 (15)
C5'—Cr—C1'—C2'	102.09 (16)	C6'—Cr—C4'—C3'	-103.64 (17)
C4'—Cr—C1'—C2'	65.82 (15)	C1'—Cr—C4'—C3'	-66.88 (16)
C11—Cr—C1'—C3	36.9 (2)	C5'—Cr—C4'—C3'	-133.3 (2)
C12—Cr—C1'—C3	145.9 (4)	O4'—C4'—C5'—C6'	-180.0 (2)
C13—Cr—C1'—C3	-52.9 (2)	C3'—C4'—C5'—C6'	-1.6 (4)
C2'—Cr—C1'—C3	113.1 (3)	Cr—C4'—C5'—C6'	-54.1 (2)
C6'—Cr—C1'—C3	-116.1 (3)	O4'—C4'—C5'—Cr	-125.9 (3)
C3'—Cr—C1'—C3	142.4 (2)	C3'—C4'—C5'—Cr	52.5 (2)
C5'—Cr—C1'—C3	-144.8 (2)	C11—Cr—C5'—C6'	34.8 (4)
C4'—Cr—C1'—C3	178.9 (2)	C12—Cr—C5'—C6'	-167.93 (16)
C6'—C1'—C2'—C3'	-3.3 (4)	C13—Cr—C5'—C6'	-76.34 (16)
C3—C1'—C2'—C3'	178.8 (2)	C2'—Cr—C5'—C6'	66.23 (15)
Cr—C1'—C2'—C3'	-56.9 (2)	C1'—Cr—C5'—C6'	28.78 (14)
C6'—C1'—C2'—Cr	53.6 (2)	C3'—Cr—C5'—C6'	102.91 (16)
C3—C1'—C2'—Cr	-124.3 (2)	C4'—Cr—C5'—C6'	131.2 (2)
C11—Cr—C2'—C3'	-123.62 (17)	C11—Cr—C5'—C4'	-96.4 (4)
C12—Cr—C2'—C3'	-36.9 (2)	C12—Cr—C5'—C4'	60.89 (17)
C13—Cr—C2'—C3'	154.20 (18)	C13—Cr—C5'—C4'	152.48 (16)
C6'—Cr—C2'—C3'	101.74 (17)	C2'—Cr—C5'—C4'	-64.95 (15)
C1'—Cr—C2'—C3'	131.5 (2)	C6'—Cr—C5'—C4'	-131.2 (2)
C5'—Cr—C2'—C3'	65.04 (16)	C1'—Cr—C5'—C4'	-102.40 (16)
C4'—Cr—C2'—C3'	29.07 (15)	C3'—Cr—C5'—C4'	-28.27 (14)
C11—Cr—C2'—C1'	104.87 (16)	C10—C5—C6—C7	-1.7 (4)
C12—Cr—C2'—C1'	-168.40 (16)	C4'—C5'—C6'—C1'	-0.4 (4)
C13—Cr—C2'—C1'	22.7 (2)	Cr—C5'—C6'—C1'	-55.7 (2)
C6'—Cr—C2'—C1'	-29.77 (14)	C4'—C5'—C6'—Cr	55.4 (2)
C3'—Cr—C2'—C1'	-131.5 (2)	C2'—C1'—C6'—C5'	2.8 (4)
C5'—Cr—C2'—C1'	-66.47 (15)	C3—C1'—C6'—C5'	-179.4 (2)
C4'—Cr—C2'—C1'	-102.44 (16)	Cr—C1'—C6'—C5'	56.1 (2)
C1'—C2'—C3'—C4'	1.5 (4)	C2'—C1'—C6'—Cr	-53.3 (2)
Cr—C2'—C3'—C4'	-55.2 (2)	C3—C1'—C6'—Cr	124.6 (2)
C1'—C2'—C3'—Cr	56.7 (2)	C11—Cr—C6'—C5'	-167.85 (15)

supplementary materials

C11—Cr—C3'—C2'	60.24 (18)	C12—Cr—C6'—C5'	19.0 (2)
C12—Cr—C3'—C2'	151.31 (17)	C13—Cr—C6'—C5'	105.92 (16)
C13—Cr—C3'—C2'	-100.7 (4)	C2'—Cr—C6'—C5'	-102.57 (16)
C6'—Cr—C3'—C2'	-66.83 (16)	C1'—Cr—C6'—C5'	-132.7 (2)
C1'—Cr—C3'—C2'	-29.73 (15)	C3'—Cr—C6'—C5'	-65.54 (15)
C5'—Cr—C3'—C2'	-103.53 (17)	C4'—Cr—C6'—C5'	-29.31 (14)
C4'—Cr—C3'—C2'	-131.7 (2)	C11—Cr—C6'—C1'	-35.1 (2)
C11—Cr—C3'—C4'	-168.06 (16)	C12—Cr—C6'—C1'	151.72 (18)
C12—Cr—C3'—C4'	-76.99 (17)	C13—Cr—C6'—C1'	-121.35 (16)
C13—Cr—C3'—C4'	31.0 (5)	C2'—Cr—C6'—C1'	30.16 (14)
C2'—Cr—C3'—C4'	131.7 (2)	C3'—Cr—C6'—C1'	67.20 (15)
C6'—Cr—C3'—C4'	64.87 (16)	C5'—Cr—C6'—C1'	132.7 (2)
C1'—Cr—C3'—C4'	101.97 (17)	C4'—Cr—C6'—C1'	103.42 (16)
C5'—Cr—C3'—C4'	28.17 (15)	C5—C6—C7—O7	-177.2 (3)
O5—C2—C3—C4	-2.0 (4)	C5—C6—C7—C8	3.3 (4)
O5—C2—C3—C1'	179.2 (2)	O7—C7—C8—C9	179.0 (3)
C6'—C1'—C3—C2	142.3 (3)	C6—C7—C8—C9	-1.6 (4)
C2'—C1'—C3—C2	-39.9 (4)	C7—C8—C9—O5	179.3 (2)
Cr—C1'—C3—C2	-127.5 (2)	C7—C8—C9—C10	-1.8 (4)
C6'—C1'—C3—C4	-36.6 (3)	O5—C9—C10—C5	-177.9 (2)
C2'—C1'—C3—C4	141.3 (2)	C8—C9—C10—C5	3.3 (4)
Cr—C1'—C3—C4	53.6 (3)	O5—C9—C10—C4	4.3 (4)
C2—C3—C4—O4	-172.4 (3)	C8—C9—C10—C4	-174.6 (2)
C1'—C3—C4—O4	6.4 (4)	C6—C5—C10—C9	-1.5 (4)
C2—C3—C4—C10	7.1 (4)	C6—C5—C10—C4	176.3 (3)
C1'—C3—C4—C10	-174.1 (2)	O4—C4—C10—C9	171.3 (3)
C2'—C3'—C4'—O4'	179.6 (2)	C3—C4—C10—C9	-8.3 (4)
Cr—C3'—C4'—O4'	125.8 (2)	O4—C4—C10—C5	-6.5 (4)
C2'—C3'—C4'—C5'	1.1 (4)	C3—C4—C10—C5	174.0 (2)
Cr—C3'—C4'—C5'	-52.7 (2)	C5'—C4'—O4'—C41'	15.9 (4)
C2'—C3'—C4'—Cr	53.8 (2)	C3'—C4'—O4'—C41'	-162.6 (2)
C11—Cr—C4'—O4'	-88.0 (3)	Cr—C4'—O4'—C41'	-77.7 (3)
C12—Cr—C4'—O4'	-3.0 (2)	C3—C2—O5—C9	-2.6 (4)
C13—Cr—C4'—O4'	83.4 (3)	C10—C9—O5—C2	1.4 (4)
C2'—Cr—C4'—O4'	-136.3 (3)	C8—C9—O5—C2	-179.7 (2)
C6'—Cr—C4'—O4'	149.5 (3)	C8—C7—O7—C71	-5.5 (4)
C1'—Cr—C4'—O4'	-173.8 (2)	C6—C7—O7—C71	175.0 (3)
C3'—Cr—C4'—O4'	-106.9 (3)		

Fig. 1

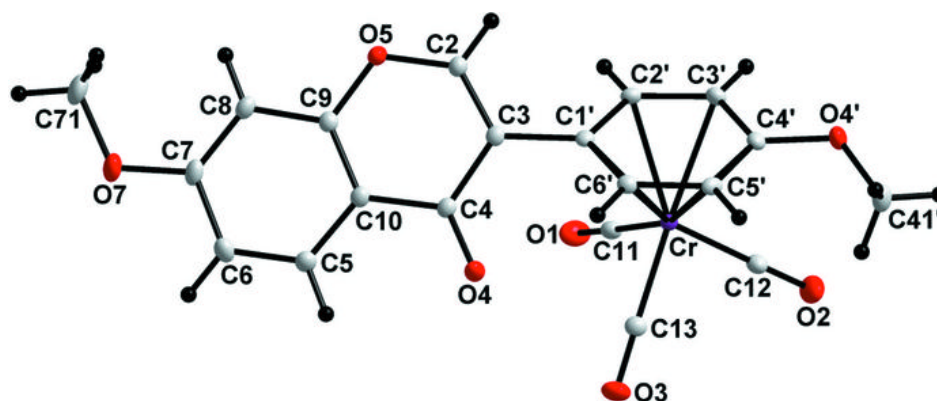


Fig. 2

