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[K(18-crown-6)][FeCp*(CO)$_2$]

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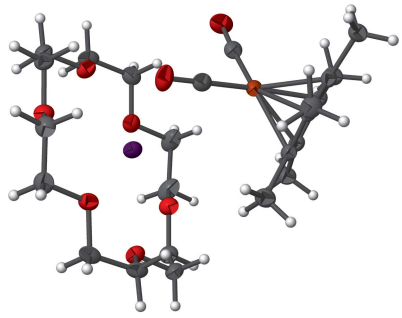
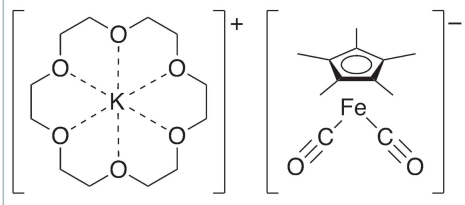
The title compound, (1,4,7,10,13,16-hexaoxacyclooctadecane-κ^6O)potassium dicarbonyl(η^5-pentamethylcyclopentadienyl)ferrate(II), [K($C_{12}H_{24}O_6$)][Fe($C_{10}H_{15}$)(CO)$_2$], consists of K^+ cations embedded in 18-crown-6 molecules and [FeCp*(CO)$_2$]$^-$ anions. Cations and anions form ion pairs which are linked by weak C—H···O interactions.

3D view



Chemical scheme



Structure description

Alkali metal salts of transition-metal carbonyl anions are important reagents in both organic and organometallic chemistry. In particular, K[FeCp(CO)$_2$] (Cp = cyclopentadienyl) and its solvated derivatives are often used for this purpose and have been structurally characterized (Hey-Hawkins & von Schnering, 1991; Sänger *et al.*, 2012). Nevertheless, the Cp* analogue (Cp* is pentamethylcyclopentadienyl) is usually prepared and reacted *in situ* (Catheline & Astruc, 1984; Barras *et al.*, 1993; Sazonov *et al.*, 2014), but details of its crystal structure remain unknown so far. We synthesized K[FeCp*(CO)$_2$] by the reduction of [FeCp*(CO)$_2$]$_2$ with potassium graphite. Although the recrystallization of the pure compound at this stage was unsuccessful, we were able to isolate the title compound by adding 18-crown-6.

The crystal contains a [K(18-crown-6)]$^+$ cation and a [FeCp*(CO)$_2$]$^-$ anion (Fig. 1). The cationic and anionic moieties are linked by the coordination of one of the carbonyl groups (C1≡O1) to K1. The C1—K and O1—K distances are 3.074 (3) and 2.994 (3) Å, respectively, which are apparently shorter than those of the Cp-analogue, [K(18-crown-6)]$^+$·[FeCp(CO)$_2$]$^-$ [C—K: 3.288 (2); O—K: 3.558 (2) Å; Sänger *et al.*, 2012]. Intramolecular C21—H33···O2 and C24—H38···O1 hydrogen bonds affect the overall molecular conformation. In the crystal, [K(18-crown-6)]$^+$·[FeCp*(CO)$_2$]$^-$ pairs are linked by additional C—H···O interactions (Table 1 and Fig. 2).

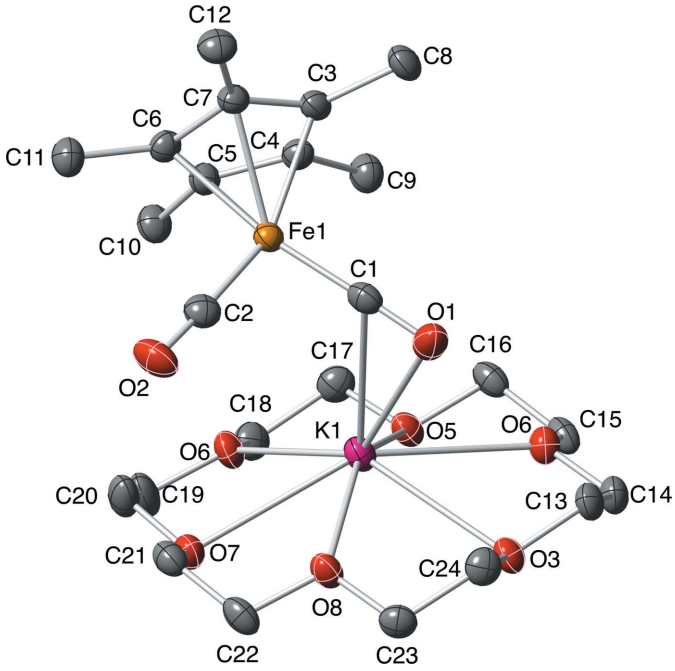


Figure 1
The structure of the molecular entities in the title compound, showing displacement ellipsoids at the 50% probability level. Hydrogen atoms are omitted for clarity.

Synthesis and crystallization

To a THF (3 ml) solution of $[FeCp^*(CO)_2]_2$ (301 mg, 0.608 mmol) was added freshly prepared KC_8 (345 mg, 2.55 mmol) at room temperature. The mixture was stirred for 3 h at the same temperature, and hexane (5 ml) was added. Graphite and the remaining KC_8 were removed by filtration with a Celite pad, and the filtrate was concentrated. Subsequently, to a THF (3 ml) solution of 18-crown-6 (242 mg, 0.915 mmol) was added the concentrated material at room temperature. The mixture was stirred for 1 h at the same temperature, and hexane (3 ml) was added. Storing the solution at 238 K gave a brown solid, which was washed with hexane to give the title compound (390 mg, 0.708 mmol) in 78% yield. Single crystals suitable for X-ray crystallographic analysis were also obtained under these conditions.

IR spectra were recorded on a Nicolet iS5 FT–IR spectrometer with a Golden Gate Single Reflection ATR unit. The melting point of 418 K was determined on a Yanaco micro melting point apparatus.

IR (ATR): 1838, 1760 cm^{-1} [ν(CO)].

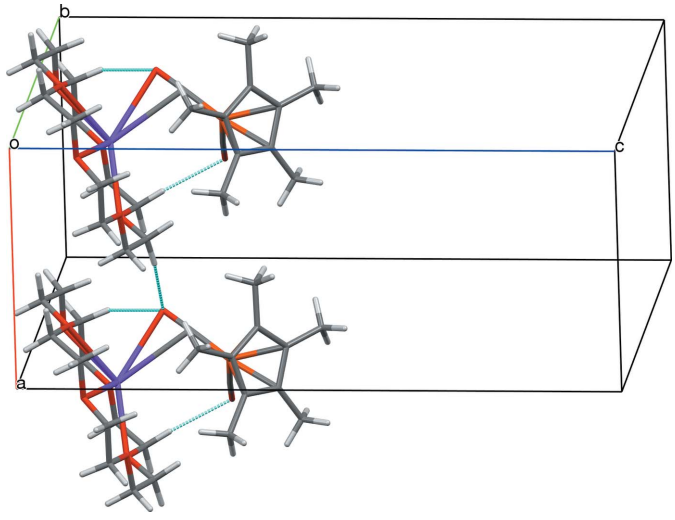


Figure 2
Parts of the crystal packing of the title compound, emphasizing intra- and intermolecular C—H···O interactions (light-blue dotted lines).

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C20—H31···O1^{i}	0.99	2.62	3.553 (4)	157
C21—H33···O2	0.99	2.40	3.379 (4)	172
C24—H38···O1	0.99	2.65	3.429 (4)	136

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$[K(C_{12}H_{24}O_6)][Fe(C_{10}H_{15})(CO)_2]$
M_r	550.50
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	103
a, b, c (Å)	8.4761 (2), 15.2842 (2), 20.5734 (3)
V (Å^3)	2665.29 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.77
Crystal size (mm)	0.18 × 0.07 × 0.03
Data collection	
Diffractometer	Rigaku Saturn
Absorption correction	Multi-scan (*MULABS*; Blessing, 1995)
T_{min}, T_{max}	0.870, 0.946
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21591, 4911, 4837
R_{int}	0.038
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.606
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.026, 0.067, 1.11
No. of reflections	4911
No. of parameters	313
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.36, −0.23
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.104 (16)

Computer programs: *CrystalClear* (Rigaku, 1999), *HKL-2000* (Otwinowski & Minor, 1997), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *Yadokari-XG* (Wakita, 2001; Kabuto *et al.*, 2009), *Mercury* (Macrae *et al.*, 2008), *CrystalMaker* (Palmer, 2007) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

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[K(18-crown-6)][FeCp*(CO)$_2$]

Yoshiyuki Mizuhata, Tatsuya Yanagisawa, Takahiro Sasamori and Norihiro Tokitoh

(1,4,7,10,13,16-Hexaoxacyclooctadecane-κ^6O)potassium dicarbonyl(η^5-pentamethylcyclopentadienyl)ferrate(II)

Crystal data

$[K(C_{12}H_{24}O_6)][Fe(C_{10}H_{15})(CO)_2]$
M_r = 550.50
Orthorhombic, $P2_12_12_1$
a = 8.4761 (2) Å
b = 15.2842 (2) Å
c = 20.5734 (3) Å
V = 2665.29 (8) Å^3
Z = 4
F(000) = 1168
D_x = 1.372 Mg m^{-3}
Melting point: 418 K
Mo $K\alpha$ radiation, λ = 0.71075 Å
Cell parameters from 21591 reflections
θ = 2.8–25.5°
μ = 0.77 mm^{-1}
T = 103 K
Prism, brown
0.18 × 0.07 × 0.03 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 28.5714 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (MULABS; Blessing, 1995)
T_{min} = 0.870, T_{max} = 0.946
21591 measured reflections
4911 independent reflections
4837 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 25.5°, θ_{min} = 2.8°
h = −9→10
k = −18→18
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.067
S = 1.11
4911 reflections
313 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2 + 0.9299P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.104 (16)

Special details

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.

Refinement. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.44214 (4)	0.66136 (2)	0.82314 (2)	0.02116 (11)
C1	0.5419 (3)	0.62203 (18)	0.75673 (14)	0.0268 (6)
O1	0.6076 (3)	0.59478 (16)	0.71033 (11)	0.0381 (5)
C2	0.2988 (3)	0.58171 (19)	0.81589 (14)	0.0280 (6)
O2	0.2025 (3)	0.52649 (16)	0.81386 (12)	0.0398 (5)
C3	0.6137 (3)	0.73000 (18)	0.87713 (13)	0.0242 (6)
C4	0.5228 (3)	0.79010 (18)	0.83896 (12)	0.0235 (6)
C5	0.3621 (3)	0.78327 (17)	0.85829 (13)	0.0234 (5)
C6	0.3541 (3)	0.71748 (17)	0.90776 (13)	0.0235 (6)
C7	0.5103 (3)	0.68568 (18)	0.92040 (13)	0.0234 (6)
C8	0.7898 (3)	0.7198 (2)	0.87451 (15)	0.0308 (6)
H1	0.8387	0.7591	0.9063	0.046*
H2	0.8275	0.7346	0.8308	0.046*
H3	0.8181	0.6592	0.8847	0.046*
C9	0.5885 (4)	0.8520 (2)	0.78864 (15)	0.0320 (7)
H4	0.5055	0.8674	0.7575	0.048*
H5	0.6759	0.8237	0.7656	0.048*
H6	0.6269	0.9052	0.8101	0.048*
C10	0.2273 (3)	0.8401 (2)	0.83546 (14)	0.0295 (6)
H7	0.1286	0.8067	0.8375	0.044*
H8	0.2465	0.8586	0.7905	0.044*
H9	0.2191	0.8917	0.8635	0.044*
C11	0.2080 (4)	0.6868 (2)	0.94250 (15)	0.0310 (6)
H10	0.2060	0.7115	0.9865	0.046*
H11	0.2085	0.6228	0.9451	0.046*
H12	0.1144	0.7063	0.9186	0.046*
C12	0.5547 (4)	0.62323 (19)	0.97377 (14)	0.0305 (6)
H13	0.6536	0.5937	0.9624	0.046*
H14	0.4709	0.5797	0.9792	0.046*
H15	0.5687	0.6556	1.0145	0.046*
K1	0.36441 (7)	0.70131 (4)	0.64053 (3)	0.02530 (14)
O3	0.5288 (2)	0.58827 (13)	0.54428 (9)	0.0260 (4)
C13	0.6919 (3)	0.6124 (2)	0.54491 (15)	0.0285 (6)
H16	0.7531	0.5725	0.5165	0.034*
H17	0.7344	0.6080	0.5896	0.034*
C14	0.7056 (3)	0.7044 (2)	0.52085 (15)	0.0300 (6)
H18	0.8182	0.7206	0.5164	0.036*
H19	0.6548	0.7098	0.4777	0.036*
O4	0.6300 (2)	0.76098 (12)	0.56636 (9)	0.0265 (4)
C15	0.6397 (3)	0.85028 (19)	0.54705 (14)	0.0281 (6)
H20	0.5840	0.8588	0.5052	0.034*
H21	0.7515	0.8673	0.5411	0.034*
C16	0.5660 (3)	0.90531 (18)	0.59826 (14)	0.0275 (6)

H22	0.6145	0.8925	0.6410	0.033*
H23	0.5826	0.9680	0.5884	0.033*
O5	0.4003 (2)	0.88612 (13)	0.60013 (10)	0.0263 (4)
C17	0.3181 (4)	0.94237 (19)	0.64366 (15)	0.0299 (6)
H24	0.3318	1.0041	0.6302	0.036*
H25	0.3605	0.9357	0.6882	0.036*
C18	0.1460 (4)	0.91822 (19)	0.64240 (15)	0.0303 (6)
H26	0.0855	0.9574	0.6715	0.036*
H27	0.1037	0.9243	0.5978	0.036*
O6	0.1320 (2)	0.83001 (13)	0.66363 (9)	0.0276 (4)
C19	−0.0283 (3)	0.8041 (2)	0.67082 (19)	0.0385 (7)
H28	−0.0828	0.8067	0.6283	0.046*
H29	−0.0831	0.8440	0.7012	0.046*
C20	−0.0323 (4)	0.7123 (2)	0.69673 (17)	0.0381 (8)
H30	0.0310	0.7084	0.7371	0.046*
H31	−0.1422	0.6953	0.7070	0.046*
O7	0.0309 (2)	0.65546 (13)	0.64870 (9)	0.0285 (4)
C21	0.0245 (3)	0.56674 (19)	0.67000 (15)	0.0306 (6)
H32	−0.0865	0.5494	0.6778	0.037*
H33	0.0835	0.5601	0.7112	0.037*
C22	0.0953 (3)	0.51010 (19)	0.61891 (15)	0.0302 (6)
H34	0.0837	0.4478	0.6311	0.036*
H35	0.0406	0.5196	0.5770	0.036*
O8	0.2589 (2)	0.53168 (13)	0.61259 (10)	0.0277 (4)
C23	0.3347 (3)	0.47949 (19)	0.56470 (14)	0.0276 (6)
H36	0.2897	0.4922	0.5213	0.033*
H37	0.3180	0.4167	0.5743	0.033*
C24	0.5071 (4)	0.49987 (18)	0.56509 (15)	0.0291 (6)
H38	0.5502	0.4922	0.6095	0.035*
H39	0.5638	0.4596	0.5355	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.01871 (18)	0.02217 (19)	0.02260 (18)	0.00073 (15)	−0.00130 (15)	−0.00015 (15)
C1	0.0228 (14)	0.0275 (13)	0.0302 (15)	−0.0057 (12)	−0.0040 (12)	0.0006 (11)
O1	0.0318 (12)	0.0487 (13)	0.0340 (11)	−0.0049 (10)	0.0087 (10)	−0.0149 (10)
C2	0.0269 (14)	0.0288 (14)	0.0283 (14)	0.0024 (12)	−0.0043 (12)	0.0011 (12)
O2	0.0324 (11)	0.0403 (12)	0.0468 (14)	−0.0129 (10)	−0.0106 (10)	0.0065 (11)
C3	0.0202 (13)	0.0261 (13)	0.0263 (13)	0.0014 (11)	−0.0015 (10)	−0.0059 (11)
C4	0.0243 (13)	0.0228 (13)	0.0233 (13)	−0.0018 (11)	0.0007 (10)	−0.0026 (10)
C5	0.0231 (13)	0.0219 (13)	0.0252 (13)	0.0026 (11)	0.0001 (12)	−0.0027 (10)
C6	0.0236 (13)	0.0232 (13)	0.0235 (13)	0.0021 (11)	0.0015 (11)	−0.0035 (10)
C7	0.0240 (13)	0.0247 (14)	0.0217 (13)	0.0022 (11)	−0.0029 (10)	−0.0026 (10)
C8	0.0185 (13)	0.0354 (16)	0.0385 (16)	−0.0003 (11)	−0.0024 (12)	−0.0045 (13)
C9	0.0320 (16)	0.0315 (15)	0.0326 (15)	−0.0015 (12)	0.0042 (12)	0.0040 (13)
C10	0.0259 (14)	0.0295 (14)	0.0332 (15)	0.0052 (13)	0.0000 (11)	0.0029 (13)
C11	0.0281 (15)	0.0329 (16)	0.0320 (15)	−0.0011 (12)	0.0049 (12)	0.0028 (12)

C12	0.0337 (15)	0.0290 (14)	0.0287 (14)	0.0015 (13)	−0.0049 (13)	0.0029 (12)
K1	0.0201 (3)	0.0236 (3)	0.0322 (3)	0.0000 (2)	0.0000 (2)	0.0000 (2)
O3	0.0193 (10)	0.0252 (9)	0.0334 (10)	0.0009 (8)	−0.0003 (8)	0.0023 (8)
C13	0.0183 (13)	0.0341 (15)	0.0332 (15)	0.0035 (12)	0.0034 (12)	−0.0022 (12)
C14	0.0220 (13)	0.0364 (16)	0.0315 (15)	−0.0013 (13)	0.0064 (12)	−0.0012 (13)
O4	0.0243 (10)	0.0250 (10)	0.0302 (10)	−0.0001 (9)	0.0038 (8)	0.0032 (8)
C15	0.0214 (13)	0.0300 (14)	0.0329 (14)	−0.0057 (12)	−0.0014 (12)	0.0095 (12)
C16	0.0234 (13)	0.0239 (13)	0.0352 (15)	−0.0055 (12)	−0.0046 (12)	0.0070 (11)
O5	0.0199 (10)	0.0248 (10)	0.0342 (10)	0.0000 (8)	−0.0015 (8)	−0.0032 (8)
C17	0.0345 (16)	0.0227 (13)	0.0324 (15)	−0.0010 (12)	0.0009 (13)	−0.0027 (12)
C18	0.0279 (15)	0.0283 (14)	0.0346 (15)	0.0067 (12)	0.0003 (13)	−0.0017 (12)
O6	0.0177 (9)	0.0282 (10)	0.0369 (11)	0.0011 (8)	0.0017 (8)	−0.0005 (8)
C19	0.0188 (14)	0.0393 (16)	0.057 (2)	−0.0006 (12)	0.0072 (14)	−0.0147 (16)
C20	0.0251 (15)	0.0421 (18)	0.0472 (18)	−0.0090 (14)	0.0119 (13)	−0.0127 (14)
O7	0.0232 (10)	0.0317 (10)	0.0305 (10)	−0.0050 (9)	0.0030 (8)	−0.0025 (9)
C21	0.0232 (14)	0.0360 (15)	0.0325 (15)	−0.0043 (11)	−0.0028 (12)	0.0045 (13)
C22	0.0228 (14)	0.0258 (14)	0.0419 (16)	−0.0064 (11)	−0.0038 (12)	0.0046 (12)
O8	0.0218 (10)	0.0256 (10)	0.0358 (11)	−0.0040 (8)	−0.0001 (8)	−0.0036 (8)
C23	0.0315 (15)	0.0208 (13)	0.0306 (14)	−0.0012 (12)	−0.0025 (12)	−0.0026 (11)
C24	0.0309 (15)	0.0232 (14)	0.0332 (15)	0.0047 (12)	0.0012 (12)	−0.0005 (11)

Geometric parameters (Å, º)

Fe1—C1	1.715 (3)	O3—C24	1.429 (3)
Fe1—C2	1.726 (3)	O3—C13	1.431 (3)
Fe1—C6	2.079 (3)	C13—C14	1.496 (4)
Fe1—C4	2.108 (3)	C13—H16	0.9900
Fe1—C3	2.109 (3)	C13—H17	0.9900
Fe1—C5	2.111 (3)	C14—O4	1.427 (4)
Fe1—C7	2.116 (3)	C14—H18	0.9900
Fe1—K1	3.8627 (7)	C14—H19	0.9900
C1—O1	1.181 (4)	O4—C15	1.424 (3)
C1—K1	3.074 (3)	C15—C16	1.486 (4)
O1—K1	2.994 (3)	C15—H20	0.9900
C2—O2	1.175 (4)	C15—H21	0.9900
C3—C7	1.421 (4)	C16—O5	1.436 (3)
C3—C4	1.433 (4)	C16—H22	0.9900
C3—C8	1.502 (4)	C16—H23	0.9900
C4—C5	1.423 (4)	O5—C17	1.423 (4)
C4—C9	1.509 (4)	C17—C18	1.505 (4)
C5—C6	1.432 (4)	C17—H24	0.9900
C5—C10	1.510 (4)	C17—H25	0.9900
C6—C7	1.434 (4)	C18—O6	1.422 (4)
C6—C11	1.504 (4)	C18—H26	0.9900
C7—C12	1.503 (4)	C18—H27	0.9900
C8—H1	0.9800	O6—C19	1.423 (3)
C8—H2	0.9800	C19—C20	1.501 (5)
C8—H3	0.9800	C19—H28	0.9900

C9—H4	0.9800	C19—H29	0.9900
C9—H5	0.9800	C20—O7	1.421 (4)
C9—H6	0.9800	C20—H30	0.9900
C10—H7	0.9800	C20—H31	0.9900
C10—H8	0.9800	O7—C21	1.426 (3)
C10—H9	0.9800	C21—C22	1.488 (4)
C11—H10	0.9800	C21—H32	0.9900
C11—H11	0.9800	C21—H33	0.9900
C11—H12	0.9800	C22—O8	1.431 (3)
C12—H13	0.9800	C22—H34	0.9900
C12—H14	0.9800	C22—H35	0.9900
C12—H15	0.9800	O8—C23	1.421 (3)
K1—O8	2.802 (2)	C23—C24	1.494 (4)
K1—O6	2.825 (2)	C23—H36	0.9900
K1—O4	2.869 (2)	C23—H37	0.9900
K1—O7	2.917 (2)	C24—H38	0.9900
K1—O5	2.960 (2)	C24—H39	0.9900
K1—O3	2.974 (2)		

C1—Fe1—C2	91.77 (14)	O4—K1—O1	83.55 (6)
C1—Fe1—C6	171.21 (13)	O7—K1—O1	120.60 (6)
C2—Fe1—C6	96.35 (13)	O5—K1—O1	125.66 (6)
C1—Fe1—C4	106.86 (12)	O3—K1—O1	71.39 (6)
C2—Fe1—C4	154.13 (12)	O8—K1—C1	87.19 (7)
C6—Fe1—C4	66.56 (11)	O6—K1—C1	119.02 (7)
C1—Fe1—C3	104.71 (12)	O4—K1—C1	98.92 (7)
C2—Fe1—C3	151.81 (12)	O7—K1—C1	109.56 (7)
C6—Fe1—C3	66.51 (11)	O5—K1—C1	122.98 (7)
C4—Fe1—C3	39.72 (11)	O3—K1—C1	93.42 (7)
C1—Fe1—C5	137.79 (12)	O1—K1—C1	22.40 (7)
C2—Fe1—C5	115.22 (12)	O8—K1—Fe1	96.19 (5)
C6—Fe1—C5	39.97 (11)	O6—K1—Fe1	93.75 (4)
C4—Fe1—C5	39.42 (10)	O4—K1—Fe1	115.71 (4)
C3—Fe1—C5	66.54 (11)	O7—K1—Fe1	94.09 (4)
C1—Fe1—C7	132.86 (12)	O5—K1—Fe1	113.98 (4)
C2—Fe1—C7	113.43 (12)	O3—K1—Fe1	118.41 (4)
C6—Fe1—C7	39.97 (11)	O1—K1—Fe1	47.94 (4)
C4—Fe1—C7	66.51 (10)	C1—K1—Fe1	25.54 (6)
C3—Fe1—C7	39.32 (11)	C24—O3—C13	111.4 (2)
C5—Fe1—C7	66.96 (10)	C24—O3—K1	106.83 (16)
C1—Fe1—K1	50.58 (10)	C13—O3—K1	107.29 (16)
C2—Fe1—K1	84.69 (10)	O3—C13—C14	108.3 (2)
C6—Fe1—K1	133.47 (8)	O3—C13—H16	110.0
C4—Fe1—K1	93.32 (7)	C14—C13—H16	110.0
C3—Fe1—K1	123.45 (8)	O3—C13—H17	110.0
C5—Fe1—K1	97.99 (8)	C14—C13—H17	110.0
C7—Fe1—K1	159.84 (8)	H16—C13—H17	108.4
O1—C1—Fe1	178.6 (3)	O4—C14—C13	108.6 (2)

O1—C1—K1	75.01 (18)	O4—C14—H18	110.0
Fe1—C1—K1	103.88 (12)	C13—C14—H18	110.0
C1—O1—K1	82.59 (18)	O4—C14—H19	110.0
O2—C2—Fe1	176.9 (3)	C13—C14—H19	110.0
C7—C3—C4	108.5 (2)	H18—C14—H19	108.4
C7—C3—C8	125.9 (3)	C15—O4—C14	111.9 (2)
C4—C3—C8	125.5 (3)	C15—O4—K1	119.89 (16)
C7—C3—Fe1	70.59 (15)	C14—O4—K1	120.58 (16)
C4—C3—Fe1	70.11 (15)	O4—C15—C16	108.7 (2)
C8—C3—Fe1	127.9 (2)	O4—C15—H20	109.9
C5—C4—C3	108.3 (2)	C16—C15—H20	109.9
C5—C4—C9	126.2 (3)	O4—C15—H21	109.9
C3—C4—C9	125.4 (3)	C16—C15—H21	109.9
C5—C4—Fe1	70.37 (16)	H20—C15—H21	108.3
C3—C4—Fe1	70.17 (16)	O5—C16—C15	108.4 (2)
C9—C4—Fe1	126.8 (2)	O5—C16—H22	110.0
C4—C5—C6	107.2 (2)	C15—C16—H22	110.0
C4—C5—C10	126.6 (3)	O5—C16—H23	110.0
C6—C5—C10	126.0 (3)	C15—C16—H23	110.0
C4—C5—Fe1	70.21 (16)	H22—C16—H23	108.4
C6—C5—Fe1	68.84 (14)	C17—O5—C16	111.9 (2)
C10—C5—Fe1	130.1 (2)	C17—O5—K1	110.46 (16)
C5—C6—C7	108.8 (2)	C16—O5—K1	107.63 (15)
C5—C6—C11	126.5 (3)	O5—C17—C18	108.4 (2)
C7—C6—C11	124.6 (2)	O5—C17—H24	110.0
C5—C6—Fe1	71.19 (15)	C18—C17—H24	110.0
C7—C6—Fe1	71.38 (15)	O5—C17—H25	110.0
C11—C6—Fe1	124.4 (2)	C18—C17—H25	110.0
C3—C7—C6	107.1 (2)	H24—C17—H25	108.4
C3—C7—C12	127.3 (3)	O6—C18—C17	108.0 (2)
C6—C7—C12	125.4 (3)	O6—C18—H26	110.1
C3—C7—Fe1	70.10 (15)	C17—C18—H26	110.1
C6—C7—Fe1	68.65 (15)	O6—C18—H27	110.1
C12—C7—Fe1	130.4 (2)	C17—C18—H27	110.1
C3—C8—H1	109.5	H26—C18—H27	108.4
C3—C8—H2	109.5	C18—O6—C19	112.1 (2)
H1—C8—H2	109.5	C18—O6—K1	123.37 (16)
C3—C8—H3	109.5	C19—O6—K1	119.32 (17)
H1—C8—H3	109.5	O6—C19—C20	108.6 (2)
H2—C8—H3	109.5	O6—C19—H28	110.0
C4—C9—H4	109.5	C20—C19—H28	110.0
C4—C9—H5	109.5	O6—C19—H29	110.0
H4—C9—H5	109.5	C20—C19—H29	110.0
C4—C9—H6	109.5	H28—C19—H29	108.4
H4—C9—H6	109.5	O7—C20—C19	108.4 (3)
H5—C9—H6	109.5	O7—C20—H30	110.0
C5—C10—H7	109.5	C19—C20—H30	110.0
C5—C10—H8	109.5	O7—C20—H31	110.0

H7—C10—H8	109.5	C19—C20—H31	110.0
C5—C10—H9	109.5	H30—C20—H31	108.4
H7—C10—H9	109.5	C20—O7—C21	110.7 (2)
H8—C10—H9	109.5	C20—O7—K1	104.97 (16)
C6—C11—H10	109.5	C21—O7—K1	106.46 (15)
C6—C11—H11	109.5	O7—C21—C22	108.7 (2)
H10—C11—H11	109.5	O7—C21—H32	110.0
C6—C11—H12	109.5	C22—C21—H32	110.0
H10—C11—H12	109.5	O7—C21—H33	110.0
H11—C11—H12	109.5	C22—C21—H33	110.0
C7—C12—H13	109.5	H32—C21—H33	108.3
C7—C12—H14	109.5	O8—C22—C21	108.7 (2)
H13—C12—H14	109.5	O8—C22—H34	109.9
C7—C12—H15	109.5	C21—C22—H34	109.9
H13—C12—H15	109.5	O8—C22—H35	109.9
H14—C12—H15	109.5	C21—C22—H35	109.9
O8—K1—O6	117.15 (6)	H34—C22—H35	108.3
O8—K1—O4	115.82 (6)	C23—O8—C22	111.8 (2)
O6—K1—O4	114.55 (6)	C23—O8—K1	121.15 (16)
O8—K1—O7	58.69 (6)	C22—O8—K1	120.25 (16)
O6—K1—O7	58.78 (6)	O8—C23—C24	108.8 (2)
O4—K1—O7	150.17 (6)	O8—C23—H36	109.9
O8—K1—O5	149.10 (6)	C24—C23—H36	109.9
O6—K1—O5	56.92 (6)	O8—C23—H37	109.9
O4—K1—O5	57.77 (6)	C24—C23—H37	109.9
O7—K1—O5	110.17 (6)	H36—C23—H37	108.3
O8—K1—O3	58.36 (6)	O3—C24—C23	108.7 (2)
O6—K1—O3	147.49 (6)	O3—C24—H38	109.9
O4—K1—O3	57.52 (5)	C23—C24—H38	109.9
O7—K1—O3	110.65 (6)	O3—C24—H39	109.9
O5—K1—O3	108.62 (6)	C23—C24—H39	109.9
O8—K1—O1	79.34 (6)	H38—C24—H39	108.3
O6—K1—O1	141.12 (6)		

C2—Fe1—C1—K1	81.64 (12)	C11—C6—C7—C3	179.3 (3)
C4—Fe1—C1—K1	−80.17 (12)	Fe1—C6—C7—C3	59.82 (18)
C3—Fe1—C1—K1	−121.46 (10)	C5—C6—C7—C12	173.2 (2)
C5—Fe1—C1—K1	−50.3 (2)	C11—C6—C7—C12	−5.7 (4)
C7—Fe1—C1—K1	−153.50 (11)	Fe1—C6—C7—C12	−125.1 (3)
C7—C3—C4—C5	−0.1 (3)	C5—C6—C7—Fe1	−61.68 (18)
C8—C3—C4—C5	−176.7 (3)	C11—C6—C7—Fe1	119.5 (3)
Fe1—C3—C4—C5	60.29 (19)	C24—O3—C13—C14	178.4 (2)
C7—C3—C4—C9	177.9 (3)	K1—O3—C13—C14	−65.0 (2)
C8—C3—C4—C9	1.4 (4)	O3—C13—C14—O4	65.6 (3)
Fe1—C3—C4—C9	−121.7 (3)	C13—C14—O4—C15	179.4 (2)
C7—C3—C4—Fe1	−60.41 (19)	C13—C14—O4—K1	−31.2 (3)
C8—C3—C4—Fe1	123.0 (3)	C14—O4—C15—C16	−177.1 (2)
C3—C4—C5—C6	−1.0 (3)	K1—O4—C15—C16	33.2 (3)

C9—C4—C5—C6	−179.1 (3)	O4—C15—C16—O5	−66.4 (3)
Fe1—C4—C5—C6	59.13 (18)	C15—C16—O5—C17	−174.3 (2)
C3—C4—C5—C10	174.0 (3)	C15—C16—O5—K1	64.2 (2)
C9—C4—C5—C10	−4.0 (5)	C16—O5—C17—C18	179.7 (2)
Fe1—C4—C5—C10	−125.8 (3)	K1—O5—C17—C18	−60.4 (2)
C3—C4—C5—Fe1	−60.17 (19)	O5—C17—C18—O6	61.2 (3)
C9—C4—C5—Fe1	121.8 (3)	C17—C18—O6—C19	173.6 (3)
C4—C5—C6—C7	1.8 (3)	C17—C18—O6—K1	−32.2 (3)
C10—C5—C6—C7	−173.3 (3)	C18—O6—C19—C20	−176.9 (2)
Fe1—C5—C6—C7	61.80 (19)	K1—O6—C19—C20	27.7 (3)
C4—C5—C6—C11	−179.4 (3)	O6—C19—C20—O7	−66.2 (3)
C10—C5—C6—C11	5.6 (4)	C19—C20—O7—C21	−177.8 (2)
Fe1—C5—C6—C11	−119.4 (3)	C19—C20—O7—K1	67.7 (2)
C4—C5—C6—Fe1	−60.01 (18)	C20—O7—C21—C22	−178.6 (2)
C10—C5—C6—Fe1	124.9 (3)	K1—O7—C21—C22	−65.1 (2)
C4—C3—C7—C6	1.2 (3)	O7—C21—C22—O8	64.4 (3)
C8—C3—C7—C6	177.8 (3)	C21—C22—O8—C23	179.6 (2)
Fe1—C3—C7—C6	−58.90 (18)	C21—C22—O8—K1	−28.9 (3)
C4—C3—C7—C12	−173.7 (3)	C22—O8—C23—C24	−174.9 (2)
C8—C3—C7—C12	2.8 (4)	K1—O8—C23—C24	33.9 (3)
Fe1—C3—C7—C12	126.2 (3)	C13—O3—C24—C23	178.8 (2)
C4—C3—C7—Fe1	60.11 (18)	K1—O3—C24—C23	61.9 (2)
C8—C3—C7—Fe1	−123.3 (3)	O8—C23—C24—O3	−65.8 (3)
C5—C6—C7—C3	−1.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C20—H31···O1^{i}	0.99	2.62	3.553 (4)	157
C21—H33···O2	0.99	2.40	3.379 (4)	172
C24—H38···O1	0.99	2.65	3.429 (4)	136

Symmetry code: (i) *x*−1, *y*, *z*.