

C(2)	-0.6323 (8)	-0.1141 (3)	-1.0136 (7)	0.026 (1)
C(3)	-0.6705 (9)	-0.0758 (4)	-0.7531 (8)	0.030 (2)
C(4)	-0.8304 (9)	-0.0694 (4)	-0.6715 (8)	0.031 (2)
C(5)	-0.5502 (7)	-0.2050 (3)	-0.7901 (7)	0.023 (1)
C(6)	-0.5220 (7)	-0.2799 (3)	-0.8867 (7)	0.024 (1)
C(7)	-0.6237 (8)	-0.3797 (3)	-0.7157 (6)	0.023 (1)
C(8)	-0.7758 (9)	-0.4366 (3)	-0.6999 (7)	0.026 (1)
C(9)	-0.5885 (8)	-0.4137 (3)	-0.9782 (7)	0.024 (1)
C(10)	-0.7176 (8)	-0.4266 (3)	-1.1329 (7)	0.025 (1)
Na(1)	-0.4750 (4)	-0.5115 (2)	-1.4014 (3)	0.0376 (9)
Na(2)	-1.0541 (4)	-0.0066 (2)	-1.3683 (4)	0.044 (1)
O(1W)	-0.3542 (11)	-0.3696 (4)	-1.3551 (8)	0.058 (2)
O(2W)	-0.5370 (2)	-0.6567 (6)	-1.4927 (1)	0.094 (5)
O(3W)	-1.1051 (1)	-0.1491 (5)	-1.4268 (2)	0.097 (5)
O(4W)	-0.9460 (1)	0.1317 (5)	-1.3636 (1)	0.101 (5)

Table 2. Selected geometric parameters (Å, °)

Mo(1)—Mo(2)	2.554 (1)	O(10)—C(4)	1.252 (9)
Mo(1)—O(1)	1.926 (4)	O(11)—C(10)	1.195 (8)
Mo(1)—O(2)	1.941 (4)	O(11)···Na(1)	2.357 (7)
Mo(1)—O(3)	1.691 (5)	O(12)—C(8)	1.218 (8)
Mo(1)—O(5)	2.113 (5)	N(1)—C(2)	1.488 (8)
Mo(1)—O(6)	2.101 (6)	N(1)—C(3)	1.474 (8)
Mo(1)—N(1)	2.419 (5)	N(1)—C(5)	1.485 (7)
Mo(2)—O(1)	1.932 (4)	N(2)—C(6)	1.502 (6)
Mo(2)—O(2)	1.934 (4)	N(2)—C(7)	1.483 (7)
Mo(2)—O(4)	1.696 (4)	N(2)—C(9)	1.487 (7)
Mo(2)—O(7)	2.113 (4)	C(1)—C(2)	1.522 (8)
Mo(2)—O(8)	2.098 (5)	C(3)—C(4)	1.50 (1)
Mo(2)—N(2)	2.397 (4)	C(5)—C(6)	1.542 (8)
O(5)—C(1)	1.277 (9)	C(7)—C(8)	1.515 (8)
O(6)—C(4)	1.277 (8)	C(9)—C(10)	1.534 (8)
O(7)—C(8)	1.302 (7)	Na(1)···O(1W)	2.554 (8)
O(8)—C(10)	1.279 (7)	Na(1)···O(2W)	2.58 (1)
O(9)—C(1)	1.203 (9)	Na(2)···O(3W)	2.458 (9)
O(9)···Na(2)	2.344 (9)	Na(2)···O(4W)	2.455 (9)
O(6)—Mo(1)—N(1)	74.4 (2)	Mo(1)—O(1)—Mo(2)	82.9 (2)
O(5)—Mo(1)—N(1)	69.8 (2)	Mo(1)—O(2)—Mo(2)	82.5 (1)
Mo(1)—O(5)—C(1)	122.2 (4)	Mo(1)—O(6)—C(4)	122.6 (5)
O(3)—Mo(1)—O(6)	94.1 (3)	Mo(2)—O(7)—C(8)	123.3 (3)
O(3)—Mo(1)—O(5)	91.5 (2)	Mo(2)—O(8)—C(10)	124.5 (4)
Mo(1)—N(1)—C(5)	117.5 (3)	O(2)—Mo(1)—O(3)	109.4 (2)
Mo(1)—N(1)—C(3)	106.3 (4)	Mo(1)—N(1)—C(2)	104.7 (3)
O(1)—Mo(1)—O(3)	109.6 (2)	O(1)—Mo(1)—O(2)	92.3 (2)
Mo(2)—N(2)—C(9)	106.9 (3)	Mo(2)—N(2)—C(7)	106.1 (3)
Mo(2)—N(2)—C(6)	119.0 (3)	O(9)—C(1)—C(2)	120.1 (6)
O(5)—C(1)—O(9)	123.0 (7)	O(5)—C(1)—C(2)	116.0 (6)
N(1)—C(2)—C(1)	109.0 (5)	N(1)—C(3)—C(4)	113.9 (5)
O(10)—C(4)—C(3)	119.0 (6)	O(6)—C(4)—C(3)	119.0 (6)
O(8)—Mo(2)—N(2)	74.7 (2)	O(6)—C(4)—O(10)	122.0 (7)
O(7)—Mo(2)—N(2)	70.7 (2)	N(1)—C(5)—C(6)	118.3 (5)
N(2)—C(6)—C(5)	117.6 (4)	N(2)—C(7)—C(8)	109.5 (4)
O(4)—Mo(2)—O(8)	92.1 (2)	O(12)—C(8)—C(7)	119.9 (5)
O(4)—Mo(2)—O(7)	92.0 (2)	O(7)—C(8)—C(7)	115.3 (5)
O(7)—C(8)—O(12)	124.8 (6)	N(2)—C(9)—C(10)	113.7 (4)
O(11)—C(10)—C(9)	120.5 (6)	O(2)—Mo(2)—O(4)	109.6 (2)
O(8)—C(10)—C(9)	116.3 (5)	O(8)—C(10)—O(11)	123.2 (6)
O(1)—Mo(2)—O(4)	109.1 (2)	O(1)—Mo(2)—O(2)	92.4 (2)

Data were collected by the rotating-anode method. No absorption correction was necessary on account of the very small crystal size. The two Mo atoms were obtained from a Patterson map. Subsequent Fourier maps based on these two atoms located the rest of the non-H atoms of the asymmetric unit. All the H-atom positions were generated. The structure was refined by full-matrix least-squares methods using *SHELX76* (Sheldrick, 1976). The *x* and *z* coordinates of Mo(1) were fixed to define the origin. The absolute structure of the individual crystal was checked by refinement with the opposite hand, resulting in *R* = 0.045.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AL1041). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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[[μ -C(OC₂H₅)](CO)₄Fe₂C{(OC₂H₅)(p-CF₃C₆H₄)(η -C₇H₇)}]

JIAN CHEN AND PEI-JU ZHENG*

Research Center of Analysis and Measurement,
Fudan University, Shanghai 200433,
People's Republic of China

YONG YU AND JIA-BI CHEN

Shanghai Institute of Organic Chemistry,
Chinese Academy of Sciences, Shanghai 200032,
People's Republic of China

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Abstract

The crystal structure of tetracarbonyl-1 κ^2 C,2 κ^2 C- μ -{2-[α -ethoxy-4-(trifluoromethyl)benzylidene-1 κ^2 C ^{α}]-1(1,2- η):2(3,4,5,6- η)-1,3,5-cycloheptatriene]- μ -ethoxymethylidyne-1:2 κ^2 C-diiron has been determined by X-ray diffraction analysis. The Fe atoms coordinated by ironyl and cycloheptatriene groups are unsymmetrically bridged by a carbyne ligand. The extensively delocalized bonding may be found over the entire metal–ligand framework.

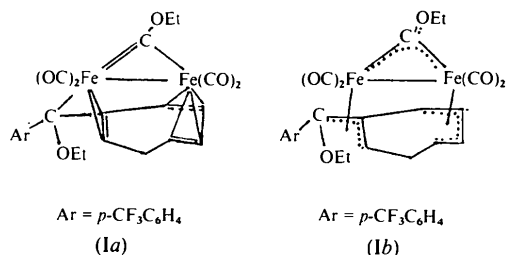
Comment

The structural study of the title compound is one of a series of studies of olefin-coordinated transition metal

carbene complexes (Chen, Lei, Xu & Tang, 1985; Chen, Lei & Tang, 1987; Chen, Yin & Shao, 1987; Chen, Lei & Zhang, 1988; Chen, Yin, Fan & Xu, 1988; Chen, Yin, Lei, Jin & Lin, 1988; Chen, Yin, Lei & Lin, 1989). It is the first bi-metal complex with a bridging carbyne ligand synthesized by the reaction of an olefin-ligated metal carbonyl compound with nucleophiles followed by alkylation with Et_3OBF_4 in a one-pot reaction (Chen, Yu, Chen & Zhang, 1994). As shown in Fig. 1, the title compound is an olefin-ligated diiron complex with a bridging carbyne group. The Fe—Fe single bond distance of 2.671 (1) Å is between those in $[\{\mu\text{-EtOC}(\text{=CH}_2)\text{C}(\text{=O})\}(\mu\text{-}^t\text{BuS})\text{Fe}_2(\text{CO})_6]$ [2.565 (1) Å; Hoke, Dewan & Seyferth, 1987] and (hexacarbonyl)-(cycloheptatriene)diiron [2.866 (1) Å; Cotton, 1971]. Moreover, it is comparable with those in $[\{\mu\text{-C}(\text{OEt})\text{-C}(\text{C}_6\text{H}_5)\text{H}\}\text{Fe}_2(\text{CO})_6]$ [2.635 (1) Å; Ros & Mathieu, 1984] and in $[\mu\text{-C}(\text{OEt})(p\text{-CF}_3\text{C}_6\text{H}_4)\text{Fe}_2(\eta\text{-C}_8\text{H}_8)(\text{CO})_4]$ [2.686 (1) Å; Li, 1990].

The metal—metal bond is unsymmetrically bridged by the atom C(10) with distances of 1.857 (7) and 1.804 (6) Å for Fe(1)—C(10) and Fe(2)—C(10), respectively. It may be compared with the asymmetric carbyne bridges in $[\{\mu\text{-MeOCH}(\text{=CHC})(\mu\text{-}^t\text{BuS})\}\text{Fe}_2(\text{CO})_6]$ (average Fe=C_{carbyne} = 1.825 and Fe—C_{carbyne} = 1.88 Å; Seyferth, Ruschke & Davis, 1989). In addition, the Fe(2)—C(10) bond length is comparable to the Mo=C_{carbyne} distance in $[\text{FeMo}\{\mu\text{-C}(4\text{-MeC}_6\text{H}_4)\}(\eta\text{-C}_5\text{H}_5)(\text{CO})_6]$ [1.921 (5) Å; Garcia, Jeffery, Sherwood & Stone, 1987] taking into account the different metal atom radii. These values infer a multiple bond character for Fe(2)—C(10). Thus, each Fe atom may be considered to have 18 valence electrons including the metal—metal bond. With the significantly asymmetric metal—carbyne linkage in the title compound, the metal—C_{carbyne} bonding could be described in localized valence—bond terms [(Ib) in scheme below]. However, the observed Fe(1)—C(10) bond distance is significantly shorter than those for a single Fe—C bridging bond, which

range from 1.930 (3) to 1.97 (1) Å in $[\{\mu\text{-EtOC}(\text{=CH}_2)\text{-C}(\text{=O})\}(\mu\text{-}^t\text{BuS})\text{Fe}_2(\text{CO})_6]$ (Hoke *et al.*, 1987) and $[\text{Fe}_2(\text{CO})_6\{\mu\text{-C}[\text{C}(\text{O})\text{OMe}]\text{C}[\text{C}(\text{O})\text{OMe}]\text{C}(\text{O})\text{C}(\text{C}_6\text{H}_5)\text{-C}(\text{C}_6\text{H}_5)\text{H}\}]\text{[P}(\text{C}_6\text{H}_5)_4]$ (Ros, Solans, Font-Altaba & Mathieu, 1984). Furthermore, the length of C(10)—O(10) [1.320 (9) Å] indicates double-bond nature, which is in accord with the knowledge that related Fischer alkoxy and acyloxy carbene complexes display considerable carbon—oxygen double-bond character (Fischer *et al.*, 1983). Hence, the metal—C_{carbyne} bonding seems more likely to be described by extensive delocalization (Ib).



The atom C(60) of a carbonyl ligand in the title compound is connected with C(71) of the cycloheptatriene ligand and C(61) of the aryl group. That the sum of C(61)—C(60)—C(71), C(61)—C(60)—O(60) and O(60)—C(60)—C(71) is very close to 360° demonstrates that the C(60) atom is bonded to three adjacent atoms [O(60), C(61) and C(71)] in a trigonal planar geometry.

Examination of the distances between C(60), C(71) and C(72) [C(60)—C(71) = 1.417 (8) and C(71)—C(72) = 1.42 (1) Å] indicates extensive electron delocalization (Ib). The three-carbon unit is best described as an allyl group η-bonded to Fe(2) in side-on mode. The slightly longer Fe(2)—C(60) distance may result from the steric effect of the bulky groups around it. The phenomenon of delocalization also explains the partial double-bond linkages in the C(74)—C(77) chain. The similar π₄-bonding mode (π₄^Δ is defined as the delocalized π-bonding involving four π electrons and the *p*_z orbitals of four atoms) has been observed in $[(\text{C}_8\text{H}_8)(\text{CO})_2\text{Fe}\{\mu\text{-C}(\text{OEt})(p\text{-MeC}_6\text{H}_4)\}]$ (Chen, Lei & Tang, 1987).

Fe(1) is surrounded by two carbonyl groups and atoms C(10), C(74), C(75), C(76) and C(77), thus defining a distorted monocapped trigonal prism with C(76) at the cap. On the other hand, the ligands [atoms C(3), C(4), C(10), C(60), C(71) and C(72)] around Fe(2) create a pseudo-octahedral geometry. Both the Fe(1)—(CO)₂ and Fe(2)—(CO)₂ groups have normal iron—carbon distances and iron—carbon—oxygen angles.

Experimental

Crystal data

$[\text{Fe}_2(\text{C}_{17}\text{H}_{16}\text{F}_3\text{O})(\text{C}_3\text{H}_5\text{O})\text{-}(\text{CO})_4]$

Mo K α radiation
 $\lambda = 0.71073$ Å

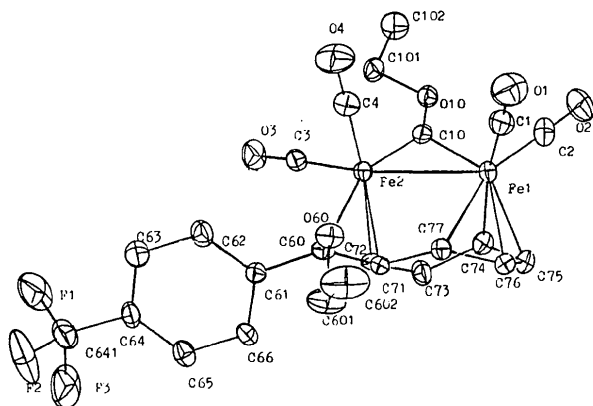


Fig. 1. The molecular structure of the title compound drawn using ORTEP (Johnson, 1965). The displacement ellipsoids are drawn at the 23% probability level.

$M_r = 574.12$
 Monoclinic
 $C2/c$
 $a = 24.725 (8) \text{ \AA}$
 $b = 14.544 (2) \text{ \AA}$
 $c = 13.950 (1) \text{ \AA}$
 $\beta = 103.10 (6)^\circ$
 $V = 4886.2 (2) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.561 \text{ Mg m}^{-3}$

Cell parameters from 25 reflections
 $\theta = 10.8\text{--}16.3^\circ$
 $\mu = 1.2447 \text{ mm}^{-1}$
 $T = 289 \text{ K}$
 $0.4 \times 0.3 \times 0.3 \text{ mm}$
 Brown

C(73)	0.5404 (3)	0.1839 (6)	0.1184 (5)	4.6 (2)
C(74)	0.5638 (3)	0.2103 (6)	0.0315 (5)	5.1 (2)
C(75)	0.6056 (4)	0.1600 (6)	-0.0020 (5)	5.5 (2)
C(76)	0.6534 (3)	0.1370 (5)	0.0641 (5)	4.9 (2)
C(77)	0.6691 (3)	0.1569 (5)	0.1677 (5)	4.7 (2)
C(101)	0.5635 (3)	0.4966 (5)	0.1729 (5)	4.7 (2)
C(102)	0.5412 (4)	0.5806 (6)	0.1181 (7)	6.8 (2)
C(601)	0.7394 (3)	0.0662 (6)	0.3834 (6)	5.6 (2)
C(602)	0.8006 (4)	0.0617 (7)	0.3867 (9)	9.1 (3)
C(641)	0.5815 (4)	0.1180 (6)	0.6857 (6)	6.7 (2)

Data collection

Enraf-Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: empirical
 $T_{\min} = 0.8642$, $T_{\max} = 0.9981$
 5618 measured reflections
 5224 independent reflections
 2094 observed reflections
 $[I > 3\sigma(I)]$

$R_{\text{int}} = 0.06$
 $\theta_{\text{max}} = 25^\circ$
 $h = -29 \rightarrow 29$
 $k = 0 \rightarrow 17$
 $l = 0 \rightarrow 16$
 3 standard reflections
 frequency: 60 min
 intensity variation: 3%

Refinement

Refinement on F
 $R = 0.045$
 $wR = 0.047$
 $S = 4.26$
 2094 reflections
 316 parameters
 Some coordinates of H atoms refined, other H-atom parameters not refined

Unit weights applied
 $(\Delta/\sigma)_{\text{max}} = 0.02$
 $\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.11 \text{ e \AA}^{-3}$
 Extinction correction: none
 Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$B_{\text{eq}} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B_{eq}
Fe(1)	0.64254 (4)	0.27746 (7)	0.06476 (7)	4.34 (2)
Fe(2)	0.63480 (4)	0.29513 (7)	0.25173 (6)	3.22 (2)
F(1)	0.5652 (3)	0.1891 (4)	0.7209 (4)	14.1 (2)
F(2)	0.6153 (3)	0.0778 (6)	0.7565 (4)	13.1 (2)
F(3)	0.5388 (3)	0.0605 (6)	0.6752 (4)	14.1 (2)
O(1)	0.7600 (2)	0.3300 (5)	0.1018 (5)	9.4 (2)
O(2)	0.6187 (4)	0.3846 (5)	-0.1129 (5)	11.1 (3)
O(3)	0.5687 (2)	0.3679 (4)	0.3792 (4)	6.7 (1)
O(4)	0.7330 (3)	0.4065 (5)	0.3335 (5)	7.8 (2)
O(10)	0.5841 (2)	0.4351 (3)	0.1064 (3)	4.1 (1)
O(60)	0.7225 (2)	0.1610 (3)	0.3600 (4)	4.2 (1)
C(1)	0.7135 (3)	0.3099 (5)	0.0857 (6)	5.8 (2)
C(2)	0.6278 (4)	0.3407 (7)	-0.0429 (6)	6.5 (2)
C(3)	0.5953 (3)	0.3409 (5)	0.3291 (5)	4.4 (2)
C(4)	0.6957 (3)	0.3621 (5)	0.3017 (6)	4.7 (2)
C(10)	0.6117 (3)	0.3595 (5)	0.1394 (5)	3.8 (2)
C(60)	0.6645 (3)	0.1705 (4)	0.3387 (5)	3.4 (1)
C(61)	0.6411 (3)	0.1560 (4)	0.4271 (4)	3.2 (1)
C(62)	0.6627 (3)	0.2045 (6)	0.5111 (5)	4.5 (2)
C(63)	0.6439 (3)	0.1925 (6)	0.5964 (5)	5.3 (2)
C(64)	0.6019 (3)	0.1282 (5)	0.5965 (5)	4.3 (2)
C(65)	0.5819 (3)	0.0778 (5)	0.5144 (5)	4.2 (2)
C(66)	0.6013 (3)	0.0902 (5)	0.4294 (5)	3.8 (2)
C(71)	0.6345 (3)	0.1573 (4)	0.2403 (4)	3.3 (1)
C(72)	0.5783 (3)	0.1872 (5)	0.2179 (5)	3.6 (1)

Table 2. Selected geometric parameters (\AA , $^\circ$)

Fe(1)—Fe(2)	2.671 (1)	O(10)—C(101)	1.460 (9)
Fe(1)—C(1)	1.776 (9)	O(60)—C(60)	1.404 (8)
Fe(1)—C(2)	1.729 (8)	O(60)—C(601)	1.456 (9)
Fe(1)—C(10)	1.857 (7)	C(60)—C(61)	1.49 (1)
Fe(1)—C(74)	2.133 (8)	C(60)—C(71)	1.417 (8)
Fe(1)—C(75)	2.059 (8)	C(61)—C(62)	1.368 (9)
Fe(1)—C(76)	2.061 (8)	C(61)—C(66)	1.38 (1)
Fe(1)—C(77)	2.267 (7)	C(62)—C(63)	1.38 (1)
Fe(2)—C(3)	1.743 (8)	C(63)—C(64)	1.40 (1)
Fe(2)—C(4)	1.797 (7)	C(64)—C(641)	1.45 (1)
Fe(2)—C(10)	1.804 (6)	C(71)—C(72)	1.42 (1)
Fe(2)—C(60)	2.212 (6)	C(72)—C(73)	1.490 (9)
Fe(2)—C(71)	2.011 (6)	C(74)—C(75)	1.43 (1)
Fe(2)—C(72)	2.084 (6)	C(76)—C(77)	1.438 (9)
F(1)—C(641)	1.26 (1)	C(601)—C(602)	1.50 (1)
F(2)—C(641)	1.28 (2)	C(64)—C(65)	1.355 (9)
F(3)—C(641)	1.33 (1)	C(65)—C(66)	1.39 (2)
O(1)—C(1)	1.16 (1)	C(71)—C(77)	1.47 (2)
O(2)—C(2)	1.15 (2)	C(73)—C(74)	1.51 (1)
O(3)—C(3)	1.13 (1)	C(75)—C(76)	1.37 (2)
O(4)—C(4)	1.14 (1)	C(101)—C(102)	1.49 (1)
O(10)—C(10)	1.320 (9)		
Fe(2)—Fe(1)—C(1)	95.8 (1)	C(10)—O(10)—C(101)	121.1 (5)
Fe(2)—Fe(1)—C(10)	42.4 (1)	O(60)—C(60)—C(61)	112.3 (5)
Fe(2)—Fe(1)—C(75)	113.5 (2)	C(61)—C(60)—C(71)	124.4 (6)
Fe(2)—Fe(1)—C(77)	61.99 (2)	C(60)—C(71)—C(77)	114.2 (6)
Fe(2)—Fe(1)—C(2)	138.5 (2)	C(71)—C(72)—C(73)	124.9 (6)
Fe(2)—Fe(1)—C(74)	89.6 (2)	C(73)—C(74)—C(75)	124.8 (7)
Fe(2)—Fe(1)—C(76)	97.9 (1)	C(75)—C(76)—C(77)	128.6 (7)
Fe(1)—Fe(2)—C(3)	144.6 (1)	O(10)—C(101)—C(102)	108.7 (6)
Fe(1)—Fe(2)—C(10)	43.9 (1)	C(60)—O(60)—C(601)	111.6 (5)
Fe(1)—Fe(2)—C(71)	80.0 (1)	O(60)—C(60)—C(71)	118.5 (6)
Fe(1)—Fe(2)—C(4)	101.1 (1)	C(60)—C(71)—C(72)	116.3 (7)
Fe(1)—Fe(2)—C(60)	111.62 (9)	C(72)—C(71)—C(77)	122.9 (6)
Fe(1)—Fe(2)—C(72)	84.2 (1)	C(72)—C(73)—C(74)	117.8 (6)
C(1)—Fe(1)—C(2)	90.5 (5)	C(74)—C(75)—C(76)	118.9 (6)
C(1)—Fe(1)—C(10)	104.7 (3)	C(71)—C(77)—C(76)	128.9 (6)
C(1)—Fe(1)—C(74)	167.5 (3)	O(60)—C(601)—C(602)	106.5 (7)
C(1)—Fe(1)—C(75)	128.1 (4)	Fe(1)—C(10)—Fe(2)	93.7 (2)
C(1)—Fe(1)—C(76)	98.0 (3)	Fe(2)—C(10)—O(10)	140.5 (3)
C(1)—Fe(1)—C(77)	88.1 (3)	Fe(2)—C(60)—C(61)	115.3 (2)
C(2)—Fe(1)—C(10)	96.4 (4)	Fe(2)—C(71)—C(60)	78.2 (2)
C(2)—Fe(1)—C(74)	93.1 (4)	Fe(1)—C(10)—O(10)	125.9 (3)
C(2)—Fe(1)—C(75)	93.7 (4)	Fe(2)—C(60)—O(60)	113.1 (2)
C(2)—Fe(1)—C(76)	121.8 (4)	Fe(2)—C(60)—C(71)	62.9 (2)
C(2)—Fe(1)—C(77)	159.5 (4)	C(3)—Fe(2)—C(60)	97.8 (3)
C(10)—Fe(1)—C(74)	86.9 (4)	C(3)—Fe(2)—C(71)	115.9 (3)
C(10)—Fe(1)—C(75)	126.2 (3)	C(3)—Fe(2)—C(72)	89.2 (4)
C(10)—Fe(1)—C(76)	135.2 (3)	C(4)—Fe(2)—C(10)	97.5 (3)
C(10)—Fe(1)—C(77)	103.8 (3)	C(4)—Fe(2)—C(60)	95.0 (3)
C(74)—Fe(1)—C(75)	39.8 (3)	C(4)—Fe(2)—C(71)	124.0 (4)
C(74)—Fe(1)—C(76)	70.1 (4)	C(4)—Fe(2)—C(72)	163.1 (3)
C(74)—Fe(1)—C(77)	84.5 (3)	C(10)—Fe(2)—C(60)	154.5 (3)
C(75)—Fe(1)—C(76)	38.7 (3)	C(10)—Fe(2)—C(71)	117.0 (3)
C(75)—Fe(1)—C(77)	71.3 (3)	C(10)—Fe(2)—C(72)	97.5 (3)
C(76)—Fe(1)—C(77)	38.4 (3)	C(60)—Fe(2)—C(71)	38.9 (2)
C(3)—Fe(2)—C(4)	95.2 (4)	C(60)—Fe(2)—C(72)	68.2 (2)
C(3)—Fe(2)—C(10)	103.3 (3)	C(71)—Fe(2)—C(72)	40.6 (3)

The structure was solved using *MULTAN*11/82 (Main *et al.*, 1982). All calculations were performed on a MicroVAX II using *SDP-Plus* (B. A. Frenz & Associates, Inc., 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HL1042). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Hexaaquanickel(II) D-Camphor-10-sulfonate

WILLIAM HENDERSON AND BRIAN K. NICHOLSON

Department of Chemistry, The University of Waikato, Private Bag 3105, Hamilton, New Zealand.

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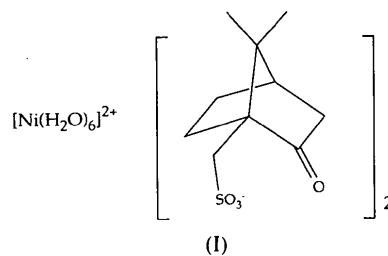
Abstract

The structure of the title complex, $[\text{Ni}(\text{H}_2\text{O})_6] \cdot 2\text{C}_{10}\text{H}_{15}\text{O}_4\text{S}$, consists of D-camphor-10-sulfonate

anions, together with essentially regular octahedral $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ cations. An extensive hydrogen-bonded network results in an overall lattice structure in which alternating layers of oppositely charged ions are stacked perpendicular to the *c* axis. The structure contains two independent D-camphor-10-sulfonate anions, with conformations related by rotation about the C(1)—C(10) bond.

Comment

Metal salts of both phosphonic and sulfonic acids with layered structures have potential applications for ion exchange and for the separation of organic compounds by selective intercalation (Suib, 1993; Cao, Hong & Mallouk, 1992; Zhang, Scott & Clearfield, 1993). Organic derivatives are used to pillar and open-out the inorganic-based layers, and the physical properties of the layered solid can readily be varied by modification of these organic groups (Burwell & Thompson, 1991). Chiral layered materials of this type (Cao, Garcia, Alcalá, Burgess & Mallouk, 1992) offer potential for enantiomeric separations by intercalation and we are investigating layered materials derived from chiral sulfonic and phosphonic acids. Only one simple metal salt (copper) of camphor-10-sulfonic acid has been structurally characterized previously (Couldwell, Prout, Robey, Taylor & Rossotti, 1978). Here we present the crystal structure of $[\text{Ni}(\text{H}_2\text{O})_6][\text{D-camphor-10-SO}_3]_2$, (1).



The crystal lattice consists of $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ cations and two crystallographically independent D-camphor-10-sulfonate anions. The $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ ion is essentially octahedral with Ni—O distances ranging from 2.029 (3) to 2.073 (5) Å (average 2.051 Å) and O—Ni—O angles between 82.7 (2) and 96.9 (2)°. These Ni—O distances are very similar to those of nickel(II) phenylsulfonate [2.036 (5)–2.073 (5) Å], in which the hexaaquanickel ion is a more regular octahedron with O—Ni—O angles in the range 87.3 (2)–92.7 (2)° (Kosnic, McClymont, Hodder & Squattrito, 1992). There is no coordination of either the sulfonate or the ketone O atoms of the anion, in contrast to observations for related phosphonate salts. This is not unexpected as these ligands show relatively poor coordinating ability when compared