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ISSN 2414-3146

Tetracarbonyldi-μ-chlorido-dichloridobis(η^5-cyclopentadienyl)diirondigallium(2 *Fe*—*Ga*)

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Received 19 August 2022
Accepted 20 August 2022

Edited by M. Bolte, Goethe-Universität Frankfurt, Germany

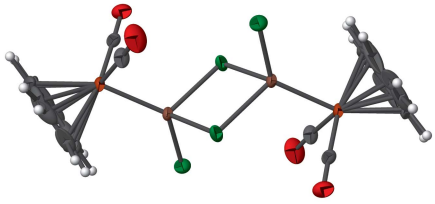
Keywords: crystal structure; iron; gallium.

CCDC reference: 2202575

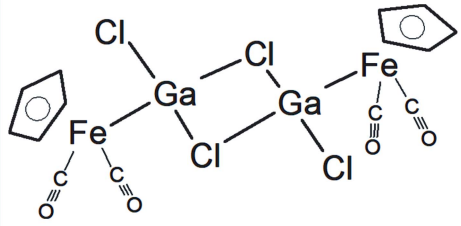
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $[Fe_2Ga_2(C_5H_5)_2Cl_4(CO)_4]$, has an iron–gallium bond distance of 2.3028 (3) Å. The gallium atoms are connected by two bridging chlorine atoms, each gallium also has one terminal chlorine. The molecule has an inversion center located between the gallium atoms. The cyclopentadienyl ligand is disordered over two sites with an occupancy of 0.57 (2) for the major occupied site.

3D view



Chemical scheme



Structure description

Digallium(II) dichloride has been used in the synthesis of two gallium–ruthenium metal clusters (Harakas & Whittlesey, 1997). The reaction of Ga_2Cl_4·1,4 dioxane with $[CpFe(CO)_2]_2$ in toluene, followed by work-up with a THF, diethyl ether and pentane solution resulted in the isolation of η^5-$CpFeGaCl_2L$, L = 1,4-dioxane or THF (Linti *et al.*, 2001). The reaction of $GaCl_3$ and $K[CpFe(CO)_2]$ in toluene produced $[\{CpFe(CO)_2\}(Ga(Cl{\cdot}GaCl_3)(\mu\text{-}Cl)]_2$ (Borovik *et al.*, 1999). In the absence of ether solvents, the reaction of Ga_2Cl_4 with $[CpFe(CO)_2]_2$ in toluene produced the title compound, which is a dimeric analog to the compounds isolated by Linti *et al.* (2001).

The Fe1—Ga1 bond distance of 2.3028 (3) Å in the title compound (Fig. 1) is similar to the 2.317 and 2.316 Å distances found for the etherate compounds (Linti *et al.*, 2001) but longer than the 2.286 Å value in $[\{CpFe(CO)_2\}(Ga(Cl{\cdot}GaCl_3)(\mu\text{-}Cl)]_2$ (Borovik *et al.*, 1999). The gallium–gallium distance of 3.4603 (3) Å is much greater than 2.406 Å for Ga_2Cl_4·2 (1,4-dioxane) (Beamish *et al.*, 1979), indicating there are no metal–metal bonding interactions between the gallium atoms.

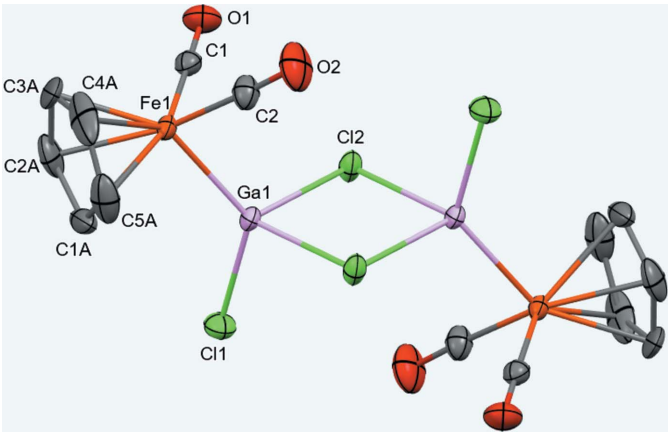


Figure 1
The title compound with 50% displacement ellipsoids. The H atoms and the minor occupied sites of the disordered atoms have been omitted for clarity. Unlabeled atoms are generated by an inversion center.

Synthesis and crystallization

All manipulations were conducted using inert atmosphere techniques. A stock solution of Ga_2Cl_4 was produced by the reaction of Ga (5.496 g, 78.83 mmol) with $GaCl_3$ (5.01 g, 28.4 mmol) in 150 ml of toluene. The mixture was heated to reflux for 24 h then cooled to 25°C. In a 150 ml Schlenk flask, $[CpFe(CO)_2]_2$ (1.107 g, 3.128 mmol) in 25 ml of toluene was combined with 25 ml of the Ga_2Cl_4 stock solution. The reaction flask was refluxed for 1 h. The mixture was cooled to room temperature, and the solution was decanted away from the residue into a new Schlenk flask. Crystals suitable for X-ray analysis formed after 24 h at 25°C. A single crystal was coated with NVH oil and mounted on a MiTeGen loop under a stream of argon gas then cooled to −75°C for data collection.

Refinement

Crystal data, data collection, and structure refinement details are summarized in Table 1. The cyclopentadienyl rings were modeled for disorder with two offset ring orientations (C1*A*—C5*A* and C1*B*—C5*B*) at 0.57 (2):0.43 (2) occupancy, respectively.

Table 1
Experimental details.

Crystal data	
Chemical formula	$[Fe_2Ga_2(C_5H_5)_2Cl_4(CO)_4]$
M_r	635.16
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	198
a, b, c (Å)	8.3567 (3), 7.0331 (2), 16.5792 (6)
β (°)	91.218 (1)
V (Å^3)	974.20 (6)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	4.76
Crystal size (mm)	0.24 × 0.22 × 0.12
Data collection	
Diffractometer	Bruker D8 Quest Eco, Photon II 7
Absorption correction	Multi-scan (Krause *et al.*, 2015)
T_{min}, T_{max}	0.36, 0.60
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	100078, 7242, 5892
R_{int}	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.962
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.086, 1.21
No. of reflections	7242
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.93, −0.99

Computer programs: *APEX3* and *SAINT* (Bruker, 2019), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*), and *ShelXle* (Hübschle *et al.*, 2011).

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full crystallographic data

IUCrData (2022). **7**, x220832 [https://doi.org/10.1107/S241431462200832X]

Tetracarbonyldi-μ-chlorido-dichloridobis(η^5-cyclopentadienyl)diirondigallium(2 *Fe—Ga*)

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Tetracarbonyldi-μ-chlorido-dichloridobis(η^5-cyclopentadienyl)diirondigallium(2 *Fe—Ga*)

Crystal data

$[Fe_2Ga_2(C_5H_5)_2Cl_4(CO)_4]$
M_r = 635.16
Monoclinic, $P2_1/c$
a = 8.3567 (3) Å
b = 7.0331 (2) Å
c = 16.5792 (6) Å
β = 91.218 (1)°
V = 974.20 (6) Å^3
Z = 2
$F(000)$ = 616
D_x = 2.165 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9746 reflections
θ = 2.9–42.0°
μ = 4.76 mm^{-1}
T = 198 K
Cube, orange
0.24 × 0.22 × 0.12 mm

Data collection

Bruker D8 Quest Eco, Photon II 7 diffractometer
Detector resolution: 7.3910 pixels mm^{-1}
phi and ω scans
Absorption correction: multi-scan (Krause *et al.*, 2015)
T_{min} = 0.36, T_{max} = 0.60
100078 measured reflections
7242 independent reflections
5892 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 43.1°, θ_{min} = 2.5°
h = −15→16
k = −13→13
l = −29→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.086
S = 1.21
7242 reflections
164 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0255P)^2 + 0.9013P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.93 e Å^{-3}
$\Delta\rho_{min}$ = −0.99 e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ga1	0.85697 (2)	0.66995 (2)	0.47616 (2)	0.01798 (4)	
Fe1	0.69410 (2)	0.68540 (3)	0.36255 (2)	0.01656 (4)	
Cl1	0.86625 (6)	0.86249 (6)	0.57979 (3)	0.02919 (8)	
Cl2	0.86216 (4)	0.37209 (6)	0.54493 (3)	0.02429 (7)	
O1	0.50545 (19)	0.3758 (2)	0.42638 (10)	0.0335 (3)	
O2	0.9025 (2)	0.4232 (3)	0.28009 (11)	0.0501 (5)	
C1	0.58075 (19)	0.4990 (2)	0.40250 (10)	0.0221 (3)	
C2	0.8235 (2)	0.5277 (3)	0.31382 (11)	0.0281 (3)	
C1A	0.6807 (11)	0.9801 (13)	0.3870 (7)	0.0346 (17)	0.57 (2)
H1A	0.725236	1.046554	0.435902	0.041000*	0.57 (2)
C2A	0.5295 (13)	0.9041 (14)	0.3781 (7)	0.0381 (18)	0.57 (2)
H2A	0.445268	0.906758	0.420085	0.046000*	0.57 (2)
C3A	0.5114 (15)	0.8261 (14)	0.3027 (10)	0.057 (4)	0.57 (2)
H3A	0.411632	0.767194	0.279609	0.068000*	0.57 (2)
C4A	0.653 (2)	0.8541 (18)	0.2625 (4)	0.056 (3)	0.57 (2)
H4A	0.673376	0.817989	0.205359	0.067000*	0.57 (2)
C5A	0.7600 (10)	0.9482 (15)	0.3150 (7)	0.043 (2)	0.57 (2)
H5A	0.871108	0.989832	0.302262	0.052000*	0.57 (2)
C1B	0.627 (4)	0.956 (3)	0.3914 (8)	0.077 (6)	0.43 (2)
H1B	0.628153	1.011381	0.447019	0.092000*	0.43 (2)
C2B	0.4986 (19)	0.865 (3)	0.3545 (15)	0.064 (6)	0.43 (2)
H2B	0.392019	0.842282	0.379064	0.076000*	0.43 (2)
C3B	0.540 (2)	0.8158 (16)	0.2786 (11)	0.050 (4)	0.43 (2)
H3B	0.471033	0.750474	0.237288	0.060000*	0.43 (2)
C4B	0.7000 (17)	0.878 (2)	0.2685 (9)	0.043 (3)	0.43 (2)
H4B	0.763455	0.864982	0.218325	0.052000*	0.43 (2)
C5B	0.7506 (15)	0.9653 (16)	0.3384 (12)	0.052 (4)	0.43 (2)
H5B	0.856690	1.027701	0.348301	0.062000*	0.43 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ga1	0.01699 (6)	0.01791 (7)	0.01880 (7)	0.00328 (5)	−0.00553 (5)	0.00044 (5)
Fe1	0.01504 (7)	0.01978 (9)	0.01474 (8)	0.00420 (6)	−0.00213 (6)	0.00057 (6)
Cl1	0.0355 (2)	0.02587 (18)	0.02603 (18)	0.00044 (15)	−0.00416 (15)	−0.00649 (14)
Cl2	0.01591 (12)	0.02142 (15)	0.03549 (19)	0.00060 (11)	−0.00080 (12)	0.01075 (14)
O1	0.0346 (7)	0.0321 (7)	0.0339 (7)	−0.0102 (6)	0.0019 (5)	−0.0029 (6)
O2	0.0548 (11)	0.0556 (11)	0.0403 (9)	0.0282 (9)	0.0118 (8)	−0.0069 (8)
C1	0.0212 (6)	0.0243 (6)	0.0206 (6)	0.0006 (5)	−0.0028 (5)	−0.0032 (5)
C2	0.0291 (7)	0.0332 (8)	0.0219 (7)	0.0095 (6)	0.0018 (6)	−0.0005 (6)
C1A	0.048 (3)	0.0189 (15)	0.036 (4)	0.005 (2)	−0.022 (3)	−0.0042 (17)
C2A	0.036 (4)	0.027 (3)	0.052 (4)	0.016 (2)	0.017 (3)	0.003 (2)
C3A	0.047 (4)	0.036 (3)	0.085 (9)	0.018 (3)	−0.051 (5)	−0.012 (4)
C4A	0.101 (9)	0.051 (6)	0.017 (2)	0.043 (6)	−0.009 (4)	0.006 (2)
C5A	0.036 (3)	0.033 (4)	0.062 (4)	0.008 (2)	0.020 (3)	0.026 (3)

C1B	0.154 (18)	0.049 (9)	0.026 (3)	0.066 (10)	−0.003 (9)	−0.003 (5)
C2B	0.033 (5)	0.051 (9)	0.108 (15)	0.025 (5)	0.031 (8)	0.050 (8)
C3B	0.067 (8)	0.023 (3)	0.058 (7)	−0.013 (4)	−0.048 (6)	0.017 (4)
C4B	0.050 (5)	0.030 (3)	0.051 (7)	0.011 (3)	0.029 (5)	0.019 (4)
C5B	0.042 (6)	0.022 (2)	0.089 (9)	−0.006 (3)	−0.045 (6)	0.001 (5)

Geometric parameters (Å, º)

Ga1—Cl1	2.1877 (5)	Fe1—C5A	2.088 (8)
Ga1—Fe1	2.3028 (3)	Fe1—C1A	2.116 (9)
Ga1—Cl2	2.3850 (4)	O1—C1	1.146 (2)
Ga1—Cl2^{i}	2.3987 (4)	O2—C2	1.142 (2)
Fe1—C1	1.7555 (17)	C1A—C2A	1.377 (11)
Fe1—C2	1.7578 (18)	C1A—C5A	1.397 (13)
Fe1—C1B	2.045 (12)	C2A—C3A	1.372 (13)
Fe1—C3A	2.057 (8)	C3A—C4A	1.382 (16)
Fe1—C4A	2.062 (8)	C4A—C5A	1.401 (13)
Fe1—C5B	2.066 (11)	C1B—C5B	1.37 (2)
Fe1—C2B	2.068 (11)	C1B—C2B	1.38 (2)
Fe1—C4B	2.069 (11)	C2B—C3B	1.36 (2)
Fe1—C2A	2.083 (9)	C3B—C4B	1.417 (17)
Fe1—C3B	2.086 (11)	C4B—C5B	1.369 (16)
Cl1—Ga1—Fe1	128.582 (16)	C2A—Fe1—C5A	64.8 (4)
Cl1—Ga1—Cl2	99.687 (18)	C1—Fe1—C1A	128.9 (3)
Fe1—Ga1—Cl2	115.906 (14)	C2—Fe1—C1A	137.9 (3)
Cl1—Ga1—Cl2^{i}	99.924 (18)	C3A—Fe1—C1A	65.1 (3)
Fe1—Ga1—Cl2^{i}	116.724 (14)	C4A—Fe1—C1A	65.3 (4)
Cl2—Ga1—Cl2^{i}	87.338 (14)	C2A—Fe1—C1A	38.3 (3)
C1—Fe1—C2	92.53 (9)	C5A—Fe1—C1A	38.8 (4)
C1—Fe1—C1B	117.0 (9)	Ga1—Cl2—Ga1^{i}	92.662 (14)
C2—Fe1—C1B	150.4 (9)	O1—C1—Fe1	178.04 (15)
C1—Fe1—C3A	98.1 (4)	O2—C2—Fe1	177.2 (2)
C2—Fe1—C3A	122.6 (5)	C2A—C1A—C5A	107.3 (8)
C1—Fe1—C4A	130.6 (5)	C2A—C1A—Fe1	69.6 (5)
C2—Fe1—C4A	95.0 (3)	C5A—C1A—Fe1	69.5 (5)
C3A—Fe1—C4A	39.2 (5)	C3A—C2A—C1A	109.6 (10)
C1—Fe1—C5B	155.8 (6)	C3A—C2A—Fe1	69.6 (5)
C2—Fe1—C5B	111.5 (6)	C1A—C2A—Fe1	72.1 (6)
C1B—Fe1—C5B	39.0 (6)	C2A—C3A—C4A	107.7 (9)
C1—Fe1—C2B	92.8 (4)	C2A—C3A—Fe1	71.7 (5)
C2—Fe1—C2B	148.1 (8)	C4A—C3A—Fe1	70.6 (5)
C1B—Fe1—C2B	39.2 (6)	C3A—C4A—C5A	108.1 (7)
C5B—Fe1—C2B	65.7 (5)	C3A—C4A—Fe1	70.2 (5)
C1—Fe1—C4B	142.8 (5)	C5A—C4A—Fe1	71.3 (4)
C2—Fe1—C4B	92.5 (4)	C1A—C5A—C4A	107.3 (6)
C1B—Fe1—C4B	64.9 (6)	C1A—C5A—Fe1	71.7 (5)
C5B—Fe1—C4B	38.7 (5)	C4A—C5A—Fe1	69.3 (5)

C2B—Fe1—C4B	65.3 (5)	C5B—C1B—C2B	109.0 (10)
C1—Fe1—C2A	98.2 (3)	C5B—C1B—Fe1	71.3 (7)
C2—Fe1—C2A	159.6 (3)	C2B—C1B—Fe1	71.3 (7)
C3A—Fe1—C2A	38.7 (4)	C3B—C2B—C1B	108.4 (12)
C4A—Fe1—C2A	64.9 (4)	C3B—C2B—Fe1	71.6 (7)
C1—Fe1—C3B	104.5 (4)	C1B—C2B—Fe1	69.5 (7)
C2—Fe1—C3B	110.3 (6)	C2B—C3B—C4B	107.0 (11)
C1B—Fe1—C3B	65.1 (5)	C2B—C3B—Fe1	70.2 (7)
C5B—Fe1—C3B	65.9 (4)	C4B—C3B—Fe1	69.4 (7)
C2B—Fe1—C3B	38.2 (6)	C5B—C4B—C3B	108.2 (10)
C4B—Fe1—C3B	39.9 (5)	C5B—C4B—Fe1	70.5 (7)
C1—Fe1—C5A	162.3 (2)	C3B—C4B—Fe1	70.7 (6)
C2—Fe1—C5A	102.4 (3)	C4B—C5B—C1B	107.4 (9)
C3A—Fe1—C5A	65.8 (4)	C4B—C5B—Fe1	70.8 (6)
C4A—Fe1—C5A	39.5 (4)	C1B—C5B—Fe1	69.7 (7)

Symmetry code: (i) $-x+2, -y+1, -z+1$.