ORGANOMETALLICS
Organometallics, 1996, 15(5), 1511-1513, DOI:10.1021/om950679t

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L1513-1

Supplement to "*A Novel Synthesis of Butatriene Hexacarbonyldiiron Complexes. Stereochemical Aspects and an X-Ray Crystal Structure Determination*"

Structure Determination Summary

Crystal Data

Empirical Formula	$C_{14}H_{12}Fe_2O_6$
Color; Habit	red blocks
Crystal size (mm)	0.3 0.4 0.4
Crystal System	Triclinic
Space Group	P_1
Unit Cell Dimensions	a = 7.558 (14) Å
	b = 8.416 (7) Å
	c = 12.92 (2) Å
	α = 89.20 (7) °
	β = 98.11 (12) °
	γ = 104.25 (12) °
Volume	788 (2) $Å^3$
Z	2
Formula weight	387.9
Density (calc.)	1.634 Mg / m^3
Absorption Coefficient	1.863 mm^{-1}
F (000)	392

Data Collection

Diffractometer Used	Siemens R3m/V
Radiation	MoKa (k = 0.71073 Å)
Temperature (K)	295

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Monochromator	Highly orientated graphite crystal
2h Range	4.0 to 45.0 °
Scan Type	x
Scan Speed	Variable; 3.00 to 14.65 °/min. in x
Scan Range (x)	1.40 °
Background measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 10.0 % of total scan time
Standard Reflections	3 measured every 100 reflections
Index Ranges	-8 \ h \ 8, -9 \ k \ 9, -13 \ l \ 13
Reflections Collected	2728
Independent Reflections	2022 (R_{int} = 8.98 %)
Observed Reflections	1039 (F > 4.0 r (F))
Absorption Correction	N/A

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$Sw\ (F_o - F_c)^2$
Absolute Structure	N/A
Extinction Correction	N/A
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = r^2\ (F) + 0.0236\ F^2$
Number of Parameters Refined	199
Final R Indices (obs. data)	R = 10.70 %, wR = 14.83 %
R Indices (all data)	R = 28.43 %, wR = 49.11 %
Goodness-of-Fit	0.93
Largest and Mean D/r	0.129, 0.016

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Data-to-Parameter Ratio	5.2 : 1
Largest Difference Peak	1.63 $eÅ^{-3}$
Largest Difference Hole	-1.09 $eÅ^{-3}$

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Table 1. Atomic coordinates [$\times 10^4$] and equivalent isotropic displacement coefficients [$Å^2 \times 10^3$]

	x	y	z	U_{eq}
Fe (1)	1934 (5)	351 (3)	2794 (2)	41 (1)
Fe (2)	2391 (5)	-1972 (3)	1641 (2)	43 (1)
C (1)	2592 (28)	-3183 (21)	3311 (14)	34 (8)
C (2)	3034 (42)	-1480 (22)	3215 (13)	60 (11)
C (3)	4314 (42)	-416 (24)	2605 (14)	57 (10)
C (4)	5237 (36)	1165 (24)	2756 (15)	50 (9)
C (11)	823 (43)	-4092 (27)	3623 (18)	74 (14)
C (12)	4168 (43)	-4057 (24)	3454 (15)	60 (12)
C (41)	6132 (52)	1772 (23)	3866 (16)	84 (14)
C (42)	5822 (43)	2227 (23)	1900 (15)	67 (12)
C (5)	1449 (29)	1989 (24)	1903 (15)	38 (8)
O (1)	1264 (31)	3002 (17)	1354 (12)	74 (8)
C (6)	-529 (58)	-764 (31)	2617 (18)	71 (15)
O (2)	-2064 (30)	-1409 (24)	2553 (15)	74 (8)
C (7)	1897 (30)	1311 (25)	4013 (18)	47 (8)
O (3)	1865 (27)	1908 (24)	4796 (13)	79 (8)
C (8)	18 (52)	-3544 (31)	1213 (17)	61 (13)
O (4)	-1216 (38)	-4383 (24)	999 (16)	92 (10)
C (9)	2263 (36)	-681 (23)	522 (15)	49 (10)
O (5)	2196 (33)	120 (22)	-172 (11)	89 (10)
C (10)	3958 (38)	-3064 (22)	1155 (15)	44 (9)
O (6)	4719 (28)	-3757 (19)	817 (12)	66 (8)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

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Table 2. Bond lengths (Å)

Fe (l)-Fe (2)	2.592 (6)	Fe (l)-C (2)	1.960 (25)
Fe (l)-C (3)	2.100 (32)	Fe (l)-C (4)	2.429 (27)
Fe (l)-C (5)	1.856 (21)	Fe (l)-C (6)	1.850 (37)
Fe (l)-C (7)	1.786 (24)	Fe (2)-C (1)	2.375 (19)
Fe (2)-C (2)	2.046 (17)	Fe (2)-C (3)	1.994 (21)
Fe (2)-C (8)	1.961 (31)	Fe (2)-C (9)	1.802 (20)
Fe (2)-C (10)	1.845 (28)	C (1)-C (2)	1.397 (25)
C (1)-C (11)	1.475 (35)	C (1)-C (12)	1.535 (38)
C (2)-C (3)	1.459 (33)	C (3)-C (3)	1.344 (27)
C (4)-C (41)	1.533 (28)	C (4)-C (42)	1.462 (28)
C (5)-0 (1)	1.124 (25)	C (6)-0 (2)	1.145 (45)
C (7)-0 (3)	1.142 (30)	C (8)-0 (4)	1.027 (37)
C (9)-0 (5)	1.118 (25)	C (10)-0 (6)	1.050 (34)

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Table 3. Bond angles (°)

Fe (2)-Fe (l)-C (2)	51.1 (5)	Fe (2)-Fe (l)-C (3)	48.9 (5)
C (2)-Fe (l)-C (3)	41.9 (10)	Fe (2)-Fe (l)-C (4)	78.5 (5)
C (2)-Fe (l)-C (4)	70.1 (10)	C (3)-Fe (l)-C (4)	33.5 (7)
Fe (2)-Fe (l)-C (5)	107.1 (7)	C (2)-Fe (l)-C (5)	154.3 (10)
C (3)-Fe (l)-C (5)	115.0 (9)	C (4)-Fe (l)-C (5)	93.9 (8)
Fe (2)-Fe (l)-C (6)	85.7 (10)	C (2)-Fe (1)-C (6)	99.1 (13)
C (3)-Fe (l)-C (6)	131.4 (12)	C (4)-Fe (l)-C (6)	164.1 (11)
C (5)-Fe (l)-C (6)	91.8 (11)	Fe (2)-Fe (l)-C (7)	153.9 (8)
C (2)-Fe (l)-C (7)	103.2 (9)	C (3)-Fe (l)-C (7)	117.6 (9)
C (4)-Fe (l)-C (7)	98.3 (8)	C (5)-Fe (l)-C (7)	98.9 (10
C (6)-Fe (l)-C (7)	95.5 (11)	Fe (l)-Fe (2)-C (1)	79.5 (5)
Fe (l)-Fe (2)-C (2)	48.2 (7)	C (l)-Fe (2)-C (2)	35.9 (7)
Fe (l)-Fe (2)-C (3)	52.6 (9)	C (l)-Fe (2)-C (3)	72.3 (7)
C (2)-Fe (2)-C (3)	42.3 (9)	Fe (l)-Fe (2)-C (8)	109.9 (10)
C (l)-Fe (2)-C (8)	89.9 (8)	C (2)-Fe (2)-C (8)	114.9 (10)
C (3)-Fe (2)-C (8)	156.3 (11)	Fe (l)-Fe (2)-C (9)	89.1 (7)
C (l)-Fe (2)-C (9)	168.4 (8)	C (2)-Fe (2)-C (9)	132.8 (8)
C (3)-Fe (2)-C (9)	99.1 (8)	C (8)-Fe (2)-C (9)	96.1 (10
Fe (l)-Fe (2)-C (10)	149.1 (7)	C (l)-Fe (2)-C (10)	93.6 (8)
C (2)-Fe (2)-C (10)	112.1 (11)	C (3)-Fe (2)-C (10)	96.6 (11)
C (8)-Fe (2)-C (10)	100.0 (12)	C (9)-Fe (2)-C (10)	95.2 (10)
Fe (2)-C (l)-C (2)	59.1 (9)	Fe (2)-C (l)-C (11)	115.5 (14)
C (2)-C (l)-C (11)	122.0 (23)	Fe (2)-C (l)-C (12)	110.3 (13)
C (2)-C (l)-C (12)	118.4 (20)	C (ll)-C (l)-C (12)	116.6 (18)
Fe (l)-C (2)-Fe (2)	80.6 (8)	Fe (l)-C (2)-C (1)	142.2 (21)
Fe (2)-C (2)-C (1)	85.0 (11)	Fe (l)-C (2)-C (3)	74.2 (14)
Fe (2)-C (2)-C (3)	66.9 (10)	C (l)-C (2)-C (3)	130.6 (24)

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Fe (l)-C (3)-Fe (2)	78.5 (11)	Fe (l)-C (3)-C (2)	63.9 (16)
Fe (2)-C (3)-C (2)	70.8 (11)	Fe (l)-C (3)-C (4)	86.7 (18)
Fe (2)-C (3)-C (4)	143.9 (17)	C (2)-C (3)-C (4)	130.8 (21)
Fe (l)-C (4)-C (3)	59.7 (17)	Fe (l)-C (4)-C (41)	106.4 (19)
C (3)-C (4)-C (41)	118.8 (17)	Fe (l)-C (4)-C (42)	115.5 (18)
C (3)-C (4)-C (42)	122.7 (18)	C (41)-C (4)-C (42)	116.8 (17)
Fe (l)-C (5)-O (1)	175.8 (21)	Fe (1)-C (6)-0 (2)	176.4 (26)
Fe (l)-C (7)-0 (3)	179.2 (17)	Fe (2)-C (8)-O (4)	178.9 (27)
Fe (2)-C (9)-0 (5)	179.5 (25)	Fe (2)-C (10)-O (6)	173.4 (20)

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Table 4. Anisotropic displacement coefficients ($Å^2 \times 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe (l)	53 (3)	35 (2)	40 (2)	19 (1)	9 (1)	-7 (1)
Fe (2)	65 (3)	33 (2)	35 (2)	21 (2)	7 (2)	-6 (1)
C (l)	11 (14)	44 (11)	52 (11)	11 (9)	14 (10)	11 (8)
C (2)	134 (28)	34 (11)	19 (9)	25 (13)	29 (11)	-4 (7)
C (3)	83 (22)	51 (13)	37 (10)	32 (13)	-21 (11)	-8 (9)
C (4)	49 (19)	42 (12)	56 (12)	4 (11)	5 (11)	-15 (10)
C (ll)	103 (31)	57 (14)	68 (14)	26 (16)	18 (16)	-8 (11)
C (12)	111 (27)	37 (11)	41 (10)	36 (14)	9 (12)	15 (9)
C (41)	157 (33)	19 (10)	60 (14)	-15 (13)	30 (16)	-9 (9)
C (42)	112 (27)	40 (11)	46 (11)	24 (14)	-8 (13)	-18 (9)
C (5)	22 (14)	44 (11)	53 (10)	15 (9)	7 (10)	-18 (10)
O (1)	108 (19)	43 (9)	70 (10)	22 (10)	7 (10)	22 (8)
C (6)	120 (34)	62 (15)	49 (12)	61 (20)	-2 (17)	2 (11)
O (2)	34 (15)	94 (14)	85 (12)	-8 (11)	14 (11)	9 (10)
C (7)	2 (13)	59 (12)	78 (14)	3 (10)	1 (11)	-24 (11)
O (3)	48 (14)	122 (14)	78 (11)	38 (11)	4 (9)	-49 (10)
C (8)	101 (29)	65 (16)	36 (11)	40 (17)	37 (14)	6 (11)
O (4)	104 (22)	65 (12)	94 (14)	15 (12)	-26 (14)	-39 (11)
C (9)	73 (21)	37 (10)	47 (12)	26 (12)	22 (11)	-5 (9)
O (5)	147 (24)	88 (12)	37 (8)	50 (13)	-3 (10)	17 (9)
C (10)	61 (19)	18 (9)	57 (12)	7 (10)	27 (11)	1 (8)
O (6)	72 (17)	55 (10)	82 (11)	25 (10)	35 (10)	4 (8)

The anisotropic displacement exponent takes the form:

$-2p^2(h^2a^2U_{11} + ... + 2hka^*b^*U_{12})$

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Table 5. H-Atom coordinates ($\times 10^4$) and isotropic displacement coeficients ($Å^2 \times 10^3$)

	x	y	z	U
H(llA)	1052	-4535	4307	80
H(llB)	9	-3365	3639	80
H(llC)	265	-4965	3129	80
H(12A)	4243	-4549	4130	80
H(12B)	3976	-4886	2920	80
H(12C)	5295	-3270	3401	80
H(41A)	7443	2099	3874	80
H(41B)	5674	2690	4056	80
H(41C)	5856	913	4358	80
H(42A)	7145	2459	1977	80
H(42B)	5319	1678	1241	80
H(42C)	5421	3231	1924	80