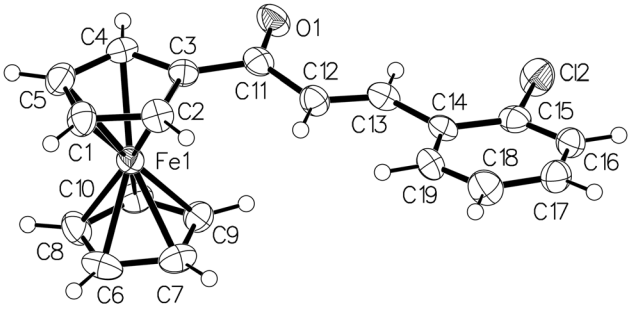
Lilei Zhang* and Mengru Xie

The crystal structure of (*E*)-3-(2-chlorophenyl)-1-ferrocenylprop-2-en-1-one, $C_{19}H_{15}ClFeO$



https://doi.org/10.1515/ncrs-2022-0042
Received January 22, 2022; accepted March 31, 2022;
published online April 12, 2022

Abstract

$C_{19}H_{15}ClFeO$, triclinic, $P\bar{1}$ (no. 2), a = 10.2693(5) Å, b = 11.2568(6) Å, c = 20.8930(10) Å, α = 80.350(2)°, β = 76.9640(10)°, γ = 86.921(2)°, V = 2319.4(2) Å^3, Z = 6, $R_{gt}(F)$ = 0.0534, $wR_{ref}(F^2)$ = 0.0968, T = 170 K.

CCDC no.: 2065551

One of the three crystallographically independent molecules of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixed solvent system comprising acetylferrocene (10 mmol) and 2-(4-chlorophenyl)acetaldehyde (10 mmol) were added to a flask. Then ethanol (25 mL) was added and stirred at room temperature for 20 min until all the solids are dissolved. To this solution 10 mL KOH (20%) was added and continue stirring until all raw materials disappear (monitored by thin layer chromatography). The reaction products were translated into 50 mL water with some solids separated out. The solids were obtained by suction filtration and washed with 30% ethanol aqueous solution. The single crystal of the title compound was obtained within seven days at room temperature by recrystallization.

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.14 × 0.11 × 0.06 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.15 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	26,628, 9450, 0.075
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5797
$N(param)_{refined}$:	596
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Experimental details

All hydrogen atoms were placed in theoretical positions and refined as riding atoms. Their U_{iso} values were set to 1.2U_{eq} of the parent atoms.

Comment

Ferrocene is known as a widely applicable organometallic framework structure for the development of various functional derivatives that are applicated in drug design, catalysis, material science, agriculture, and electrochemistry [5–7]. The wide application of ferrocene derivatives is attributable to the unique properties of ferrocene scaffold, such as native excellent air stability, solubility, thermal, and photochemical stability [8, 9]. Substituting the benzene ring of a bioactive molecule by ferrocene group will dramatically change hydrophobicity, lipophilicity, solubility, and stability [10]. Various ferrocene derivatives were designed to exhibit a wide range of biological properties, like anti-cancer [11], anti-oxidant [12], anti-HIV, analgesic, anti-infective [13], anti-viral [14], anti-microbial [15], anti-convulsant effects [5, 16]. Therefore, in this work, one ferrocene derivative,

***Corresponding author: Lilei Zhang**, College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, China, E-mail: zhanglilei@outlook.com. https://orcid.org/0000-0003-0798-1991

Mengru Xie, College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	1.0155 (4)	0.4610 (4)	0.6198 (2)	0.0373 (11)
H1	0.960078	0.462717	0.588888	0.045*
C2	1.0073 (4)	0.5422 (4)	0.66520 (19)	0.0328 (10)
H2	0.945593	0.607692	0.670343	0.039*
C3	1.1095 (4)	0.5077 (4)	0.70243 (19)	0.0281 (9)
C4	1.1787 (4)	0.4048 (4)	0.67834 (19)	0.0311 (10)
H4	1.251413	0.362770	0.693579	0.037*
C5	1.1198 (4)	0.3768 (4)	0.6279 (2)	0.0377 (11)
H5	1.146038	0.312180	0.603505	0.045*
C6	0.7975 (4)	0.2952 (4)	0.7341 (2)	0.0452 (12)
H6	0.739908	0.302203	0.703906	0.054*
C7	0.7985 (4)	0.3716 (4)	0.7808 (2)	0.0405 (11)
H7	0.740970	0.439368	0.787733	0.049*
C8	0.8982 (4)	0.2058 (4)	0.7405 (2)	0.0408 (12)
H8	0.919446	0.141657	0.715442	0.049*
C9	0.8997 (4)	0.3306 (4)	0.81551 (19)	0.0347 (11)
H9	0.922170	0.365818	0.849766	0.042*
C10	0.9613 (4)	0.2281 (4)	0.7903 (2)	0.0359 (11)
H10	1.032992	0.182115	0.804459	0.043*
C11	1.1288 (4)	0.5564 (4)	0.7604 (2)	0.0313 (10)
C12	1.0310 (4)	0.6468 (4)	0.7865 (2)	0.0326 (10)
H12	0.961178	0.672929	0.764049	0.039*
C13	1.0368 (4)	0.6930 (4)	0.8400 (2)	0.0333 (10)
H13	1.102249	0.660014	0.863935	0.040*
C14	0.9513 (4)	0.7905 (4)	0.8655 (2)	0.0304 (10)
C15	0.9658 (4)	0.8380 (4)	0.92081 (19)	0.0326 (10)
C16	0.8944 (4)	0.9384 (4)	0.9400 (2)	0.0357 (11)
H16	0.907633	0.969031	0.977684	0.043*
C17	0.8042 (4)	0.9941 (4)	0.9046 (2)	0.0379 (11)
H17	0.755690	1.063612	0.917485	0.045*
C18	0.7845 (4)	0.9485 (4)	0.8502 (2)	0.0438 (12)
H18	0.721330	0.986000	0.825976	0.053*
C19	0.8561 (4)	0.8492 (4)	0.8313 (2)	0.0355 (11)
H19	0.841250	0.818792	0.793790	0.043*
Cl2	1.07745 (11)	0.77116 (11)	0.96854 (5)	0.0466 (3)
Fe1	0.97948 (6)	0.37285 (5)	0.71595 (3)	0.02744 (16)
O1	1.2249 (3)	0.5230 (3)	0.78557 (14)	0.0403 (8)
C20	0.9865 (4)	1.1423 (4)	0.54650 (19)	0.0285 (10)
C21	1.1182 (4)	1.1056 (4)	0.5347 (2)	0.0370 (11)
H21	1.183095	1.153643	0.502557	0.044*
C22	1.1568 (4)	0.9992 (4)	0.5695 (2)	0.0430 (12)
H22	1.247855	0.973656	0.561463	0.052*
C23	1.0610 (4)	0.9299 (4)	0.6164 (2)	0.0412 (12)
H23	1.086541	0.857123	0.641055	0.049*
C24	0.9295 (4)	0.9668 (4)	0.6270 (2)	0.0348 (10)
H24	0.864572	0.916814	0.657899	0.042*
C25	0.8876 (4)	1.0757 (4)	0.59371 (19)	0.0282 (10)
C26	0.7478 (4)	1.1173 (4)	0.60747 (19)	0.0302 (10)
H26	0.723246	1.182520	0.577036	0.036*
C27	0.6534 (4)	1.0723 (4)	0.65834 (19)	0.0312 (10)
H27	0.675838	1.008159	0.689980	0.037*
C28	0.5135 (4)	1.1179 (4)	0.66806 (19)	0.0277 (9)
C29	0.4128 (4)	1.0457 (3)	0.71899 (19)	0.0266 (9)
C30	0.2730 (4)	1.0696 (4)	0.73013 (19)	0.0302 (10)
H30	0.229766	1.132473	0.705908	0.036*

Table 2: (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C31	0.2100 (4)	0.9835 (4)	0.7834 (2)	0.0414 (12)
H31	0.116440	0.978203	0.801212	0.050*
C32	0.4358 (4)	0.9435 (4)	0.76660 (19)	0.0334 (10)
H32	0.519906	0.907585	0.770895	0.040*
C33	0.3085 (4)	0.9064 (4)	0.8061 (2)	0.0449 (12)
H33	0.292546	0.840643	0.841696	0.054*
C34	0.3406 (7)	1.0847 (6)	0.9093 (3)	0.084 (2)
H34	0.330805	1.016347	0.943366	0.100*
C35	0.4615 (6)	1.1296 (8)	0.8686 (4)	0.099 (3)
H35	0.548392	1.097618	0.870448	0.118*
C36	0.4308 (6)	1.2304 (6)	0.8247 (3)	0.079 (2)
H36	0.493395	1.278366	0.791218	0.094*
C37	0.2935 (5)	1.2477 (4)	0.8386 (2)	0.0506 (13)
H37	0.245483	1.309811	0.816460	0.061*
C38	0.2378 (5)	1.1580 (4)	0.8908 (2)	0.0532 (14)
H38	0.145249	1.148599	0.910401	0.064*
Cl3	0.94509 (10)	1.27827 (10)	0.50079 (5)	0.0379 (3)
Fe3	0.34247 (6)	1.08104 (6)	0.81255 (3)	0.03316 (17)
O3	0.4824 (3)	1.2107 (3)	0.63423 (14)	0.0410 (8)
C39	0.5045 (5)	0.8046 (5)	0.5223 (3)	0.0551 (15)
H39	0.564827	0.822707	0.480342	0.066*
C40	0.5044 (5)	0.8554 (4)	0.5770 (3)	0.0540 (14)
H40	0.564738	0.914693	0.579522	0.065*
C41	0.4022 (5)	0.8065 (4)	0.6284 (2)	0.0503 (14)
H41	0.379970	0.826266	0.672050	0.060*
C42	0.3371 (4)	0.7224 (4)	0.6043 (3)	0.0509 (14)
H42	0.263011	0.674663	0.628543	0.061*
C43	0.4027 (5)	0.7225 (5)	0.5376 (3)	0.0522 (14)
H43	0.380916	0.674609	0.508220	0.063*
C44	0.6925 (4)	0.5675 (4)	0.5647 (2)	0.0432 (12)
H44	0.739090	0.568845	0.519796	0.052*
C45	0.5810 (5)	0.4954 (4)	0.5981 (2)	0.0422 (12)
H45	0.539773	0.440223	0.579333	0.051*
C46	0.7221 (4)	0.6369 (4)	0.60951 (19)	0.0351 (11)
H46	0.791959	0.693528	0.600000	0.042*
C47	0.6293 (4)	0.6075 (4)	0.67183 (19)	0.0268 (9)
C48	0.5417 (4)	0.5193 (4)	0.6639 (2)	0.0327 (10)
H48	0.469530	0.483114	0.697084	0.039*
C49	0.6258 (4)	0.6647 (4)	0.73057 (19)	0.0268 (9)
C50	0.5211 (4)	0.6247 (3)	0.79105 (19)	0.0269 (9)
H50	0.448838	0.579188	0.787123	0.032*
C51	0.5247 (4)	0.6498 (3)	0.85000 (19)	0.0299 (10)
H51	0.594528	0.700316	0.851886	0.036*
C52	0.4309 (4)	0.6072 (4)	0.91309 (19)	0.0288 (10)
C53	0.3495 (4)	0.5080 (4)	0.9195 (2)	0.0352 (11)
H53	0.352028	0.470118	0.881845	0.042*
C54	0.2655 (4)	0.4644 (4)	0.9796 (2)	0.0416 (12)
H54	0.210028	0.397868	0.982867	0.050*
C55	0.2622 (4)	0.5173 (4)	1.0349 (2)	0.0426 (12)
H55	0.205722	0.485907	1.076325	0.051*
C56	0.3398 (4)	0.6149 (4)	1.0305 (2)	0.0404 (11)
H56	0.336586	0.651961	1.068528	0.048*
C57	0.4229 (4)	0.6589 (4)	0.97017 (19)	0.0314 (10)
Cl1	0.51792 (13)	0.78416 (11)	0.96709 (6)	0.0520 (3)
Fe2	0.53173 (6)	0.67378 (5)	0.59881 (3)	0.02652 (15)
O2	0.7091 (3)	0.7394 (3)	0.73009 (13)	0.0400 (8)

i.e., (*E*)-3-(2-chlorophenyl)-1-ferrocenylprop-2-en-1-one, was synthesized and crystallized.

As displayed in the figure, the title compound adopts the *s-trans* configuration in all three crystallographically independent molecules. The bulky substituent is incorporated at C3 position of ferrocene unit, which makes the bond distances of C2–C3 (1.442(7) Å) and C3–C4 (1.431(7) Å) more elongate. Other bond length in the ferrocenyl group ranges from 1.404(7) Å to 1.418(6) Å. Compared with 3-ferrocenylprop-2-enal, the introducing of 2-chlorophenyl leads to bond lengths of C–C and C=C closer, which are closer to the typical single and double bonds [17]. However, similar to 3-ferrocenylprop-2-enal, the crystal structure of the title compound is also stabilized mainly by intermolecular C–H···O interactions [17].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was financially supported by the Key Scientific Research Projects of Colleges and Universities in Henan Province (22A430032).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

1. Bruker. Smart Apex-II CCD; Bruker AXS Inc.: Madison, WI, USA, 2006.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
5. Larik F. A., Saeed A., Fattah T. A., Muqadar U., Channar P. A. Recent advances in the synthesis, biological activities and various applications of ferrocene derivatives. *Appl. Organomet. Chem.* 2017, *31*, e3664.
6. Gao D.-W., Yin Q., Gu Q., You S.-L. Enantioselective synthesis of planar chiral ferrocenes via Pd(0)-catalyzed intramolecular direct C–H bond arylation. *J. Am. Chem. Soc.* 2014, *136*, 4841–4844.
7. Tong R., Zhao Y., Wang L., Yu H., Ren F., Saleem M., Amer W. A. Recent research progress in the synthesis and properties of burning rate catalysts based on ferrocene-containing polymers and derivatives. *J. Organomet. Chem.* 2014, *755*, 16–32.
8. Lal B., Badshah A., Altaf A. A., Khan N., Ullah S. Miscellaneous applications of ferrocene-based peptides/amides. *Appl. Organomet. Chem.* 2011, *25*, 843–855.
9. Ebadi-Dehaghani H., Mehdipour-Ataei S. Novel ferrocene-based organometallic poly(ether sulfone amide imide)s: preparation, characterization, and properties. *J. Inorg. Organomet. Polym. Mater.* 2012, *22*, 223–234.
10. Ferreira C. L., Ewart C. B., Barta C. A., Little S., Yardley V., Martins C., Polishchuk E., Smith P. J., Moss J. R., Merkel M., Adam M. J., Orvig C. Synthesis, structure, and biological activity of ferrocenyl carbohydrate conjugates. *Inorg. Chem.* 2006, *45*, 8414–8422.
11. Ornelas C. Application of ferrocene and its derivatives in cancer research. *New J. Chem.* 2011, *35*, 1973–1985.
12. Khelef A., Lanez T. In vitro assays of the antioxidant activities of ferrocene derivatives bearing amine, amide or hydrazine groups. *Der Pharma Chem.* 2015, *7*, 318–323.
13. Ludwig B. S., Correia J. D. G., Kühn F. E. Ferrocene derivatives as anti-infective agents. *Coord. Chem. Rev.* 2019, *396*, 22–48.
14. Asghar F., Badshah A., Lal B., Butler I. S., Tabassum S., Tahir M. N. Bioactivity of new ferrocene incorporated *N*,*N*′- disubstituted ureas: synthesis, structural elucidation and DFT study. *Inorg. Chim. Acta.* 2016, *439*, 82–91.
15. Yagnam S., Trivedi R., Krishna S., Giribabu L., Praveena G., Prakasham R. S. Bioactive isatin (oxime)-triazole-thiazolidinedione ferrocene molecular conjugates: design, synthesis and antimicrobial activities. *J. Organomet. Chem.* 2021, *937*, 121716.
16. Fouda M. F. R., Abd-Elzaher M. M., Abdelsamaia R. A., Labib A. A. On the medicinal chemistry of ferrocene. *Appl. Organomet. Chem.* 2007, *21*, 613–625.
17. Imhof W. 3-Ferrocenyl prop-2-enal. *Acta Crystallogr.* 2004, *E60*, m1234–m1236.