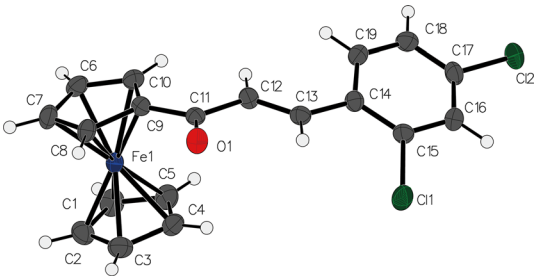
Shuang Zhang, Jie Gao, Juanjuan Wen, Yanrong Gao and Bin Liu*

Crystal structure of (E)-(3-(2,4-dichlorophenyl) acryloyl)ferrocene, $C_{19}H_{14}Cl_2FeO$



https://doi.org/10.1515/ncrs-2025-0121
Received March 7, 2025; accepted April 2, 2025;
published online April 15, 2025

Abstract

$C_{19}H_{14}Cl_2FeO$, monoclinic, $P2_{1/n}$ (no. 14), a = 7.3383(5) Å, b = 10.1441(6) Å, c = 21.4807(12) Å, α = 90 Å, β = 91.649(2) Å, γ = 90 Å, V = 1,598.37(17) Å^3, Z = 4, $R_{gt}(F)$ = 0.0443 $wR_{ref}(F^2)$ = 0.1132, T = 183 K.

The molecular structure is shown in the figure. Table 1 contains the crystallographic data. The list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

CCDC no.: 2425389

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.15 × 0.11 × 0.07 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.28 mm^{-1}
Diffractometer, scan mode:	Bruker APEX2, φ and ω scans
θ_{max}, completeness:	26.4°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9,517, 3,258, 0.067
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,630
$N(param)_{refined}$:	208
Programs:	Bruker,[1] SHELX,[2,3] Olex2[4]

1 Source of materials

The furan-2-carbaldehyde (3.83 g, 22.0 mmol), acetylferrocene (2.28 g, 10.0 mmol) and KOH (0.67 g, 12.0 mmol) were added to the mortar. After stirring well, the reaction mixture was grind for 20 min, until the TLC indicated the reaction was completed, then diluted with water and filtered. The solid was collected and washed with water, and dried overnight under vacuum. The crude product was further purified by flash silica chromatography. For crystal growth, the crude product was dissolved in a minimal amount of hot ethanol and slowly cooled to room temperature (Table 1).

2 Experimental details

The crystal structure was determined with the SHELXT program.[2] The initial solution was further refined through the SHELXL program,[3] which was implemented within the Olex2 software package.[4] Non-hydrogen atoms were refined anisotropically, while the hydrogen atoms were positioned at their idealized locations based on standard geometrical constraints.

3 Comment

Ferrocene, a typical organometallic compound, has long been a subject of extensive study due to its structural features and applications in several fields.[5] Crystallographic investigations of ferrocene and its derivatives provide detailed insights into packing, intermolecular interactions, and the influence of substituent groups on their physical properties.[6–11] The study of (E)- (3-(2,4-dichlorophenyl)acryloyl)ferrocene, a derivative of ferrocene, aims to explore the impact of the acrylate and dichlorophenyl substituents on the molecular arrangement and crystallization behavior, and contributes to the growing knowledge regarding the crystallography of ferrocene derivatives and their structure-property relationships.

The core of the molecule consists of a ferrocene unit, where an iron (Fe) atom is sandwiched between two cyclopentadienyl anions (C_5H_5). The Fe–C bond lengths range

***Corresponding author: Bin Liu**, Xianyang Key Laboratory of Molecular Imaging and Drug Synthesis, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xianyang, Shaanxi, China, E-mail: lb125lb@163.com. https://orcid.org/0000-0003-2852-7739
Shuang Zhang, Jie Gao, Juanjuan Wen and Yanrong Gao, Xianyang Key Laboratory of Molecular Imaging and Drug Synthesis, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xianyang, Shaanxi, China

from 3.04 Å to 3.06 Å, and the C–C bond lengths fall within the same range of 3.04 Å to 3.06 Å. The dihedral angle between the two C_5H_5 rings is 1.9 Å, consistent with previously reported similar structures.[12–16] The acrylate group is attached to the ferrocene core at position 3, with the (E)-configuration of the double bond. The C9–C11 bond length is 1.472(5) Å. The 2,4-dichlorophenyl ring is positioned adjacent to the acrylate group, with chlorine (Cl) atoms at the 2 and 4 positions. The C17–Cl2 bond length is 1.741(4) C_5H_5, and the C15–Cl1 bond length is 1.742(4) Å. The phenyl ring is not coplanar with the C_5H_5 ring, resulting in a torsion with a dihedral angle of 34.1°.

In the solid state, the molecules pack together via π-π stacking interactions between the aromatic phenyl rings, particularly between the 2,4-dichlorophenyl ring and neighboring phenyl groups from adjacent molecules. These π-π interactions stabilize the crystal structure and contribute to its high packing efficiency.

Acknowledgments: This work was financially supported by the projects of Social Development in Shaanxi Province Science and Technology Department (2023–YBSF-036), the 2023 research and development project of the Xianyang Science and Technology Bureau (L2023–ZDYF–SF-030), Key Laboratory of Molecular Imaging and Drug Synthesis of Xianyang city (2021QXNL–PT-0008), School-level Scientific and Technological Innovation Team for Design, Synthesis and Structural Modification of Drug Molecules (2024KCTD04).

References

1. Bruker. SAINT, APEX2 and SADABS; Bruker AXS Inc.: Madison, WI, USA, 2012.
2. Sheldrick, G. M. SHELXT – Integrated Space-Group and Crystal-Structure Determination. *Acta Crystallogr.* **2015**, *A71*, 3–8.
3. Sheldrick, G. M. Crystal Structure Refinement with *SHELXL*. *Acta Crystallogr.* **2015**, *C71*, 3–8.
4. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A.; Puschmann, H. OLEX2: a Complete Structure Solution, Refinement and Analysis Program. *J. appl. crystallogr.* **2009**, *42*, 339–341.
5. Štěpnička, P. Forever Young: the First Seventy Years of Ferrocene. *Dalton Trans.* **2022**, *51*, 8085–8102.
6. Artigas, V.; González, D.; Fuentealba, M. Syntheses, Characterisation and Crystal Structures of Ferrocenyl β-diketones and Their Schiff Base NNO Ligand Derivatives with 2-picolylamine. *J. Mol. Struct.* **2017**, *1129*, 325–332.
7. Shi, Y. -C.; Yang, H. -M.; Shen, W. -B.; Yan, C. -G.; Hu, X. -Y. Syntheses and Crystal Structures of Ferrocene-Containing Enaminones and Their Copper Complexes. *Polyhedron* **2004**, *23*, 15–21.
8. Tang, W.; Gao, Y.; Tong, H.; Xu, X.; Zhu, Z.; Liu, B. Green Synthesis of Ferrocenyl Chalcones against Triple Negative Breast Cancer. *J. Organomet. Chem.* **2023**, *989*, 122640.
9. Delavaux-Nicot, B.; Maynadié, J.; Lavabre, D.; Lepetit, C.; Donnadieu, B. The First X-Ray Characterized Monosubstituted Ferrocenyl Azacrown Chalcone: Focus on its Calcium Interaction/ Electrochemical Detection Studies. *Eur. J. Inorg. Chem.* **2005**, *2005*, 2493–2505.
10. Allison, M.; Caramés-Méndez, P.; Pask, C. M.; Phillips, R. M.; Lord, R. M.; McGowan, P. C. Bis(bipyridine)Ruthenium(II) Ferrocenyl β–Diketonate Complexes: Exhibiting Nanomolar Potency against Human Cancer Cell Lines. *Chem.-Eur. J.* **2021**, *27*, 3737–3744.
11. Novoa, N.; Roisnel, T.; Dorcet, V.; Hamon, J. -R.; Carrillo, D.; Manzur, C.; Robin-Le Guen, F.; Cabon, N. Anisyl and Ferrocenyl Adducts of Methylenepyran-Containing β-diketone: Synthesis, Spectral, Structural, and Redox Properties. *J. Organomet. Chem.* **2014**, *762*, 19–28.
12. Long, S. -J.; Liu, X. -L.; Liu, Y. -H. (E)-1–Ferrocenyl-3-(4-methoxyphenyl) prop-2-en-1-one. *Acta Crystallogr.Sec. E* **2008**, *64*, m1164.
13. Otano Vega, M. R.; Rivero, K. I.; Montes Gonzalez, I. (E)-1–Ferrocenyl-3-(2-Methoxyphenyl)prop-2-en-1-one. *Acta Crystallogr. Sec. E* **2014**, *70*, m108–m109.
14. Ding, F.; Xu, X.; Zhu, Z.; Tong, H.; Tang, W. Crystal Structure of (*E*)-(3-(thiophen-2-yl)acryloyl)ferrocene. *Z. Kristallogr.- N. Cryst. Struct.* **2025**, *240*, 295–297.
15. Zhang, J.; Hu, X.; Liu, X.; He, Y. The crystal structure of (E) -1-ferrocenyl-3-(4-isopropylphenyl)prop-2-en-1-one, $C–H_{22}FeO$. *Z. Kristallogr. - N. Cryst. Struct.* **2022**, *237*, 437–439.
16. Wang, Y.; Zheng, H.; Zhang, K.; Li, X.; Liu, B. Crystal Structure of (*E*)-(3-(3-methylthiophen-2-yl)acryloyl)ferrocene. *Z. Kristallogr. - N. Cryst. Struct.* **2025**, *240*, 337–339.