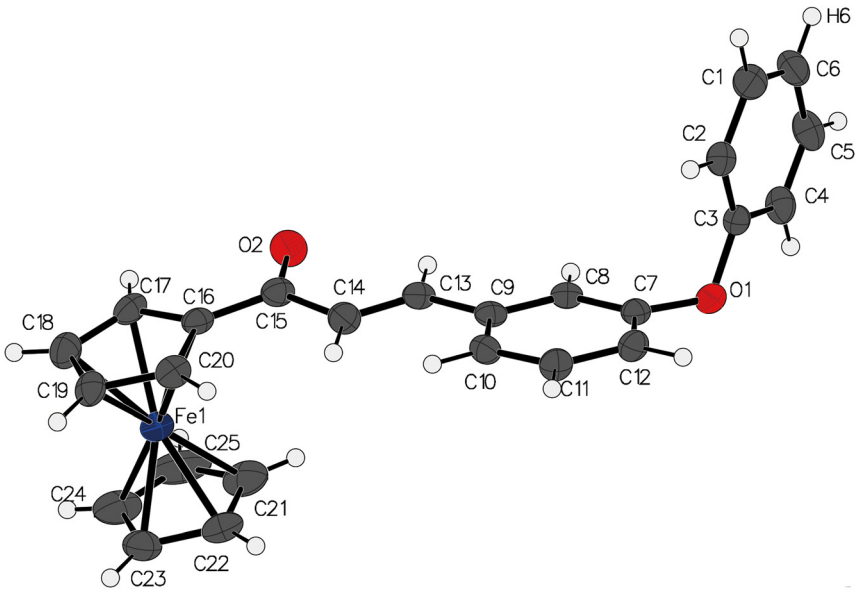
Jie Gao, Shuang Zhang, Yanan Wang, Yucong Guo and Bin Liu*

Crystal structure of (E)-(3-(4-phenoxyphenyl) acryloyl)ferrocene, $C_{25}H_{20}FeO_2$



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Abstract

$C_{25}H_{20}FeO_2$, monoclinic, $P2_1/c$ (no. 14), a = 15.9672(10) Å, b = 5.7830(4) Å, c = 20.9889(15) Å, β = 104.017(3)°, V = 1880.4(2) Å^3, Z = 4, $R_{gt}(F)$ = 0.0565 $wR_{ref}(F^2)$ = 0.1227, T = 173 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Red block
Size	0.15 × 0.08 × 0.06 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.16 mm^{-1}
Diffractometer, scan mode:	Bruker D8 Venture, φ and ω scans
θ_{max}, completeness:	26.4°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5300, 2520, 0.050
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,384
$N(param)_{refined}$:	191
Programs:	Bruker,[1] SHELX,[2,3] Olex2[4]

1 Source of materials

The 4-phenoxybenzaldehyde (4.36 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 ground 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 to afford a single crystal of high quality. For crystal growth, the crude product was dissolved in a minimal amount of hot ethanol and slowly cooled to room temperature.

***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
Jie Gao, **Shuang Zhang**, **Yanan Wang and Yucong Guo**, Xianyang Key Laboratory of Molecular Imaging and Drug Synthesis, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xianyang, Shaanxi, China

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.36030 (3)	0.28816 (8)	0.35293 (2)	0.02664 (17)
O1	0.95524 (16)	0.2543 (4)	0.32997 (12)	0.0347 (6)
O2	0.54593 (17)	0.7211 (4)	0.38580 (13)	0.0395 (7)
C1	1.0967 (2)	0.6339 (7)	0.44974 (19)	0.0344 (9)
H1	1.116426	0.654418	0.495862	0.041*
C2	1.0398 (2)	0.4556 (6)	0.42536 (17)	0.0292 (8)
H2	1.020816	0.353238	0.454353	0.035*
C3	1.0112 (2)	0.4297 (6)	0.35795 (17)	0.0259 (8)
C4	1.0398 (2)	0.5742 (7)	0.31548 (18)	0.0323 (9)
H4	1.020376	0.553564	0.269352	0.039*
C5	1.0969 (2)	0.7489 (7)	0.34063 (19)	0.0374 (10)
H5	1.117237	0.847979	0.311523	0.045*
C6	1.1252 (2)	0.7819 (7)	0.40770 (19)	0.0368 (9)
H6	1.163566	0.904875	0.424660	0.044*
C7	0.8918 (2)	0.1854 (6)	0.36169 (16)	0.0256 (8)
C8	0.8212 (2)	0.3252 (6)	0.35908 (16)	0.0250 (8)
H8	0.818098	0.472821	0.338763	0.030*
C9	0.7543 (2)	0.2508 (6)	0.38615 (16)	0.0240 (8)
C10	0.7611 (2)	0.0330 (6)	0.41594 (17)	0.0277 (8)
H10	0.715741	−0.021874	0.434038	0.033*
C11	0.8329 (2)	−0.1022 (6)	0.41921 (18)	0.0296 (9)
H11	0.837450	−0.248076	0.440609	0.035*
C12	0.8989 (2)	−0.0268 (6)	0.39135 (17)	0.0303 (9)
H12	0.948097	−0.121088	0.392944	0.036*
C13	0.6797 (2)	0.4035 (6)	0.38369 (16)	0.0270 (8)
H13	0.684067	0.557484	0.369071	0.032*
C14	0.6075 (2)	0.3470 (6)	0.39981 (18)	0.0314 (9)
H14	0.599676	0.191611	0.411813	0.038*
C15	0.5385 (2)	0.5168 (6)	0.39987 (17)	0.0286 (8)
C16	0.4612 (2)	0.4328 (6)	0.41859 (16)	0.0261 (8)
C17	0.3846 (2)	0.5666 (6)	0.41464 (17)	0.0300 (9)
H17	0.376794	0.724802	0.402420	0.036*
C18	0.3227 (3)	0.4215 (7)	0.43215 (18)	0.0356 (9)
H18	0.265901	0.465525	0.433711	0.043*
C19	0.3593 (2)	0.2000 (7)	0.44697 (17)	0.0314 (9)
H19	0.331429	0.070195	0.460422	0.038*
C20	0.4445 (2)	0.2034 (6)	0.43844 (16)	0.0291 (8)
H20	0.483579	0.076784	0.444722	0.035*
C21	0.3915 (3)	0.2719 (8)	0.26438 (19)	0.0482 (12)
H21	0.443507	0.325890	0.255220	0.058*
C22	0.3766 (3)	0.0469 (7)	0.28622 (17)	0.0366 (10)
H22	0.416884	−0.076925	0.294244	0.044*
C23	0.2915 (3)	0.0399 (7)	0.29388 (19)	0.0384 (10)
H23	0.264255	−0.089681	0.308095	0.046*
C24	0.2536 (3)	0.2584 (8)	0.2768 (2)	0.0523 (13)
H24	0.196404	0.301611	0.277480	0.063*
C25	0.3151 (4)	0.4010 (8)	0.25870 (19)	0.0578 (14)
H25	0.306726	0.557582	0.244960	0.069*

2 Experimental details

Single-crystal X-ray diffraction data were collected using a Bruker D8 Venture diffractometer with Mo Kα radiation.[1] The structure was solved using the SHELX-2014 software and refined through full-matrix least-squares on F^2,[2,3] with anisotropic displacement parameters for non-hydrogen atoms. Hydrogen atoms were positioned in idealized geometries and refined using a riding model. Data analysis and validation of the structural model were performed in the Olex2 software suite.[4]

3 Comment

Ferrocene derivatives have shown to have applications in materials science, catalysis, and medicinal chemistry.[5,6] The unique sandwich structure of ferrocene imparts remarkable stability and electronic properties, making it an excellent framework for functionalization.[7–11] (3-(4–Phenoxyphenyl)acryloyl) ferrocene represents an intriguing example of such a hybrid molecule, combining the ferrocene core with conjugated system, which is crucial for potential applications.

The ferrocene moiety adopts a typical sandwich structure, with the iron atom (Fe1) positioned symmetrically between the two cyclopentadienyl (Cp) rings. The Fe–C bond distances range from approximately 2.02 to 2.05 Å, consistent with standard values for substituted ferrocene derivatives. The Cp rings exhibit nearly ideal parallel alignment with a centroid-to-centroid distance of approximately 3.33 Å, confirming the stability of the ferrocene framework.[12–18] The acrylate unit is conjugated with the phenyl ring, forming an extended π-system that adopts an (E)-configuration. The C=C bond length (C13–C14) is 1.319(5) Å, indicative of a double bond. The carbonyl group (C=O) exhibits a bond length of 1.230(5) Å, typical for conjugated ketones.

The phenoxyphenyl group connected to the acrylate chain forms a nearly coplanar geometry with the conjugated system, minimizing steric hindrance and promoting delocalization of π-electrons. The bond angle between the phenoxy and phenyl rings is approximately 118.3°.

No significant hydrogen bonding interactions are observed in the crystal.

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