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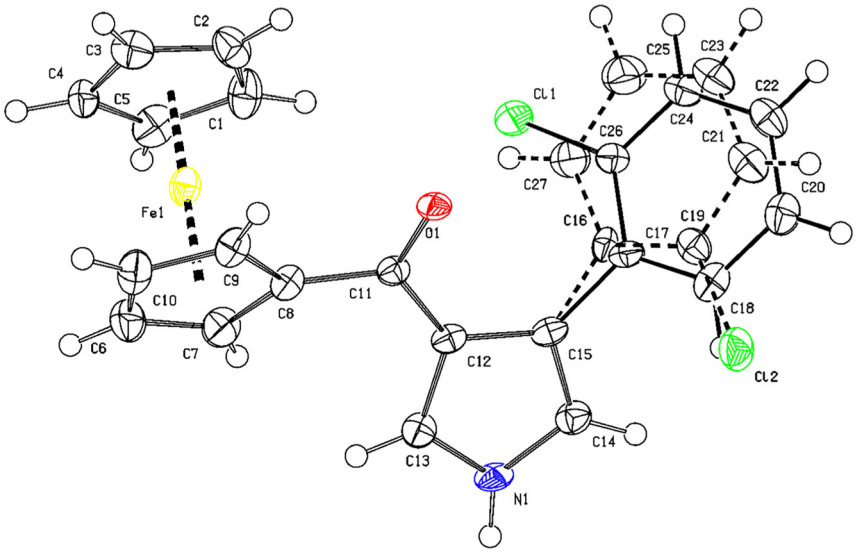
Crystal structure of (4-(2-chlorophenyl)-1*H*-pyrrol-3-yl)(ferrocenyl) methanone, $C_{21}H_{16}ClFeNO$



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Abstract

$C_{21}H_{16}ClFeNO$, monoclinic, $P2_1/c$ (no. 14), a = 10.3273(5) Å, b = 23.0536(11) Å, c = 7.2835(4) Å, β = 106.078(2)°, V = 1666.24(15) Å^3, Z = 4, $R_{gt}(F)$ = 0.0558, $wR_{ref}(F^2)$ = 0.1219, T = 170 K.

The molecular 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.

1 Source of materials

To a solution of 1-ferrocenyl-3-(2-chloropheny)-2-propen-1-one (3.50 g, 10 mmol) and tosylmethyl isocyanide (2.15 g, 11 mmol) in *N*,*N*-dimethylformamide (25 mL) was added potassium tert-butoxide (2.24 g, 20 mmol). The mixture was stirred at room temperature for 12 h, until the TLC indicated the reaction was completed. The mixture was diluted with brine, and then extracted with ethyl acetate (3 × 30 mL). The organic phase was washed with brine (30 mL), dried with anhydrous sodium sulfate, and then concentrated under pressure. The title compound was

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.15 × 0.08 × 0.05 mm
Wavelength:	Mo*Kα* radiation (0.71073 Å)
μ:	1.07 mm^{-1}
Diffractometer, scan mode:	D8 VENTURE, φ and ω
θ_{max}, completeness:	27.1°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,222, 3657, 0.089
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2468
$N(param)_{refined}$:	269
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.25336 (5)	0.35105 (2)	0.82963 (8)	0.02294 (17)
Cl1[a]	0.05697 (16)	0.54491 (7)	0.6815 (3)	0.0296 (4)
O1	0.3585 (2)	0.50292 (10)	0.8515 (3)	0.0208 (6)
Cl2[b]	0.4185 (3)	0.67316 (10)	0.5448 (4)	0.0336 (6)
N1	0.3308 (3)	0.51250 (14)	0.2226 (5)	0.0234 (7)
C12	0.3212 (3)	0.49782 (15)	0.5200 (5)	0.0188 (8)
C15	0.2811 (3)	0.55675 (16)	0.4674 (5)	0.0196 (8)
C8	0.3697 (4)	0.40858 (15)	0.7316 (5)	0.0223 (8)
C11	0.3477 (3)	0.47212 (15)	0.7092 (5)	0.0191 (8)
C9	0.4467 (4)	0.38270 (16)	0.9075 (6)	0.0253 (9)
H9	0.488558	0.402693	1.022686	0.030*
C13	0.3521 (4)	0.47309 (16)	0.3639 (5)	0.0228 (8)
H13	0.383181	0.434551	0.357755	0.027*
C14	0.2881 (4)	0.56354 (16)	0.2841 (5)	0.0237 (8)
H14	0.266825	0.598075	0.210790	0.028*
C17[a]	0.2521 (5)	0.60872 (17)	0.5843 (8)	0.0196 (16)
C18[a]	0.3223 (5)	0.6602 (2)	0.5853 (7)	0.0276 (17)
H18[a]	0.387529	0.662960	0.515894	0.033*
C20[a]	0.2970 (5)	0.70770 (16)	0.6878 (7)	0.0306 (15)
H20[a]	0.344970	0.742891	0.688515	0.037*
C22[a]	0.2015 (5)	0.70368 (17)	0.7894 (7)	0.0289 (16)
H22[a]	0.184217	0.736136	0.859464	0.035*
C24[a]	0.1313 (4)	0.6522 (2)	0.7884 (7)	0.0257 (16)
H24[a]	0.066022	0.649450	0.857795	0.031*
C26[a]	0.1566 (4)	0.60471 (16)	0.6859 (7)	0.0216 (14)
C4	0.1220 (4)	0.29255 (16)	0.8919 (6)	0.0273 (9)
H4	0.128840	0.251492	0.888335	0.033*
C2	0.1458 (4)	0.38677 (17)	0.9970 (6)	0.0308 (9)
H2	0.171445	0.419968	1.075962	0.037*
C3	0.1795 (4)	0.32886 (17)	1.0519 (6)	0.0296 (9)
H3	0.231615	0.316251	1.174234	0.036*
C10	0.4488 (4)	0.32197 (17)	0.8781 (6)	0.0315 (10)
H10	0.493608	0.294103	0.969879	0.038*
C5	0.0524 (4)	0.32887 (17)	0.7385 (6)	0.0310 (9)
H5	0.004414	0.316462	0.613795	0.037*
C7	0.3237 (4)	0.36291 (16)	0.5980 (6)	0.0304 (9)
H7	0.269559	0.367342	0.470073	0.037*
C1	0.0675 (4)	0.38699 (17)	0.8051 (6)	0.0325 (10)
H1A	0.030952	0.420357	0.732611	0.039*
C6	0.3723 (4)	0.31001 (17)	0.6877 (7)	0.0358 (10)
H6	0.356600	0.272691	0.630540	0.043*
C27[b]	0.1280 (8)	0.5781 (3)	0.6613 (12)	0.030 (2)
H27[b]	0.091465	0.540182	0.635835	0.036*
C16[b]	0.2318 (8)	0.5957 (3)	0.5868 (12)	0.019 (2)
C19[b]	0.2852 (7)	0.6512 (3)	0.6241 (12)	0.024 (2)
C21[b]	0.2348 (8)	0.6891 (2)	0.7359 (12)	0.032 (2)
H21[b]	0.271251	0.727051	0.761447	0.038*
C23[b]	0.1309 (8)	0.6715 (3)	0.8105 (11)	0.031 (3)
H23[b]	0.096427	0.697380	0.886866	0.037*
C25[b]	0.0775 (7)	0.6160 (3)	0.7731 (11)	0.038 (2)
H25[b]	0.006533	0.603946	0.824062	0.046*
H1	0.341 (4)	0.5053 (18)	0.107 (7)	0.040 (13)*

[a]Occupancy: 0.6, [b]Occupancy: 0.4.

separated by silica-gel column chromatography with ethyl acetate-petroleum ether (25 %). The target product was obtained as a white solid. Yield: 60.2 %. For crystal growth, the product was dissolved in a minimal amount of hot ethanol and slowly cooled to room temperature.

2 Experimental details

The C-bound H atoms were placed in idealized positions and refined by the riding model with U_{iso}(H) = 1.2 U_{eq}(C).

3 Comment

Ferrocene derivatives have garnered significant interest as candidate drugs for their potential anticancer, antibacterial, antifungal, and antiparasitic properties [5]. Over the past decades, numerous crystal structures of diverse ferrocene derivatives have been reported, leading to the development of novel drug molecules [6–8].

The asymmetric unit contains a single molecule of the title compound. The stability of the crystal lattice primarily relies on the N–H···O intermolecular interactions between the oxygen atom of the keto group and the hydrogen atoms located on pyrrole. The dihedral angles between the two cyclopentadienyl rings of the ferrocene scaffold and the pyrrole ring are measured to be 39.9° and 39.7°, respectively, while the dihedral angles between the ferrocene scaffold and the chloro-phenyl ring are 80.9° and 80.2°, respectively. All bond lengths fall within the expected range, ensuring the compound's structural integrity [9–12]. These findings contribute to the expanding knowledge of ferrocene derivatives and their potential in drug devel.

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

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