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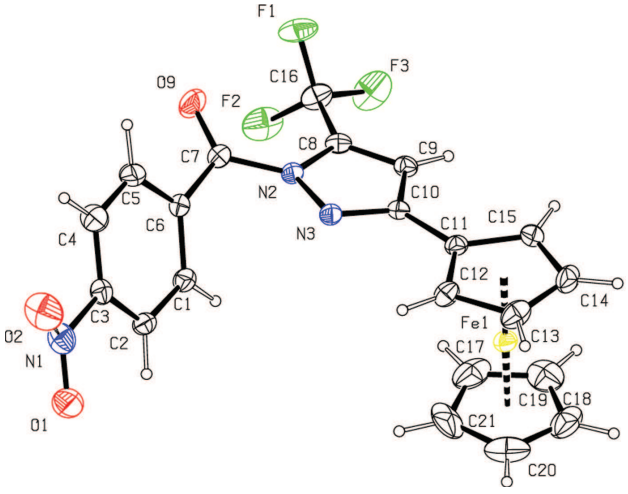
The crystal structure of (4-nitrophenyl) (5-ferrocenyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl) methanone, $C_{21}H_{12}F_3FeN_3O_3$



https://doi.org/10.1515/ncrs-2018-0102
Received March 10, 2018; accepted July 16, 2018; available online July 31, 2018

Abstract

$C_{21}H_{14}F_3FeN_3O_3$, triclinic, $P\bar{1}$ (no. 2), $a = 7.961(3)$ Å, $b = 11.136(4)$ Å, $c = 11.588(4)$ Å, $\alpha = 76.357(10)°$, $\beta = 84.998(10)°$, $\gamma = 76.989(10)°$ $V = 972.1(6)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0509$, $wR_{ref}(F^2) = 0.1304$, $T = 273(2)$ K.

CCDC no.: 1856034

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

A mixture of 5-ferrocenyl-3-(trifluoromethyl)-1*H*-pyrazole (0.32 g, 1 mmol, prepared by a literature method [4]), triethylamine (0.17 mL, 1.2 mmol), and 4-nitrobenzoyl chloride (0.22 g, 0.012 mol) in 5 mL of CH_2Cl_2 were refluxed overnight. After completion, the solvent was evaporated under reduced pressure. Water was added and the mixture was extracted with ethyl acetate twice. The combined organic layer was dried over Na_2SO_4, filtered and evaporated. The pure title compound was obtained by column chromatography on silica gel using petroleum ether/ethylacetate (2:1, *v/v*, Rf = 0.2) as eluent. Crystals of the title compound were obtained by slow evaporation of dichloromethane/methanol within 2 days.

Table 1: Data collection and handling.

Crystal:	Clear dark red block
Size:	0.25 × 0.23 × 0.21 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.83 mm^{-1}
Diffractometer, scan mode:	SMART, φ and ω-scans
θ_{max}, completeness:	26°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9817, 3821, 0.039
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2643
$N(param)_{refined}$:	289
Programs:	Bruker programs [1], SHELX [2, 3]

Experimental details

Single crystal X-ray diffraction measurements for title compound were carried out on a Siemens Smart 1000 CCD diffractometer equipped with a graphite crystal monochromator situated in the incident beam for data collection at room temperature. The hydrogen atoms were added theoretically and riding on the concerned atoms.

Comment

Organometallic compounds, which are defined as metal complexes containing at least one direct metal-carbon bond, have been proven to be a promising metal-based chemotherapy alternative option to platinum-based anticancer drugs [5, 6]. Among them, the use of ferrocene-containing compounds for medicinal applications has long been considered as an attractive way to develop anticancer drugs [7, 8], due to its low toxicity, significant stability and lipophilicity, facile functionalization, and unique electrochemical behavior. In the past few years, we have reported several ferrocenes and

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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	0.75072(6)	−0.22990(4)	0.96441(4)	0.04639(19)
F1	1.3456(3)	0.1125(2)	0.5665(2)	0.0862(8)
F2	1.1666(4)	0.0785(3)	0.4588(2)	0.1040(9)
F3	1.3307(4)	−0.0758(3)	0.5675(3)	0.1087(10)
O1	0.1424(4)	0.5165(3)	0.6849(3)	0.0876(10)
O2[a]	0.264(2)	0.6429(13)	0.7359(11)	0.108(4)
O2'[b]	0.2614(19)	0.6768(11)	0.6659(9)	0.092(3)
O9	1.0422(3)	0.2998(3)	0.5523(3)	0.0827(9)
N1	0.2687(5)	0.5589(3)	0.6836(3)	0.0686(9)
N2	0.9667(3)	0.1290(2)	0.6769(2)	0.0399(6)
N3	0.8720(3)	0.0912(2)	0.7788(2)	0.0411(6)
C1	0.6087(4)	0.2869(3)	0.6338(3)	0.0483(8)
H1	0.617451	0.204733	0.625071	0.058*
C2	0.4488(4)	0.3621(3)	0.6465(3)	0.0477(8)
H2	0.348908	0.332750	0.643911	0.057*
C3	0.4399(4)	0.4808(3)	0.6630(3)	0.0477(8)
C4	0.5833(5)	0.5294(4)	0.6620(4)	0.0661(11)
H4	0.572944	0.611067	0.672386	0.079*
C5	0.7424(5)	0.4560(3)	0.6453(4)	0.0634(10)
H5	0.840915	0.488663	0.641744	0.076*
C6	0.7568(4)	0.3324(3)	0.6338(3)	0.0448(8)
C7	0.9316(4)	0.2570(3)	0.6154(3)	0.0507(9)
C8	1.1069(4)	0.0343(3)	0.6640(3)	0.0425(8)
C9	1.1003(4)	−0.0643(3)	0.7564(3)	0.0413(7)
H9	1.176687	−0.142365	0.771058	0.050*
C10	0.9535(4)	−0.0245(3)	0.8261(3)	0.0365(7)
C11	0.8928(4)	−0.0946(3)	0.9398(3)	0.0415(7)
C12	0.7364(5)	−0.0569(3)	1.0010(3)	0.0515(9)
H12	0.652661	0.015783	0.975804	0.062*
C13	0.7288(6)	−0.1493(4)	1.1079(3)	0.0656(11)
H13	0.639393	−0.147441	1.165149	0.079*
C14	0.8781(6)	−0.2431(4)	1.1124(3)	0.0658(11)
H14	0.904928	−0.315042	1.173094	0.079*
C15	0.9823(5)	−0.2109(3)	1.0094(3)	0.0544(9)
H15	1.089553	−0.257254	0.990584	0.065*
C16	1.2368(5)	0.0397(4)	0.5640(4)	0.0616(10)
C17	0.7348(9)	−0.2717(6)	0.8063(4)	0.1003(16)
H17	0.794942	−0.240812	0.737277	0.120*
C18	0.7882(7)	−0.3812(5)	0.8882(5)	0.0932(15)
H18	0.891723	−0.439025	0.884038	0.112*
C19	0.6655(8)	−0.3918(5)	0.9765(5)	0.0942(15)
H19	0.671269	−0.458675	1.042634	0.113*
C20	0.5345(7)	−0.2909(7)	0.9538(6)	0.1121(18)
H20	0.435555	−0.274557	1.001413	0.134*
C21	0.5745(9)	−0.2155(6)	0.8457(7)	0.124(2)
H21	0.505339	−0.140321	0.806701	0.149*

Occupancies: [a] = 0.48, [b] = 0.52

found that some of them displayed impressive anticancer activities [9, 10]. Herein we report the synthesis and crystal structure of a ferrocene-based amide bearing a pyrazolyl moiety.

The title compound crystallizes in the triclinic space group $P\bar{1}$, with two molecules in the unit cell. The average bond length of Fe1-C (Cp11-15) is 2.036 Å, which is comparable to that (2.02 Å) of the other Cp ring (Cp17-21). The longest bond length is Fe1$\cdots$C13 (2.045 Å), and the shortest Fe—C (Cp) separation is 2.02 Å (Fe1$\cdots$C21). The Cp-Fe-Cp angle (179.3°) deviates slightly from linearity. The pyrazole moiety is rotated out the plane of the adjacent Cp ring (C17—C18—C19—C20—C21) by approximately 8.9°.

Acknowledgements: This work was supported by the Key Scientific and Technological Project of Anhui Provincial Tobacoo Company (No. 20150551007).

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