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The crystal structure of 1-(5-ferrocenyl-3-(trifluoromethyl)-1*H*-pyrazol-1-yl)pentan-1-on, $C_{19}H_{19}F_3FeN_2O$

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.30 × 0.24 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.87 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25.7°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	17705, 3439, 0.035
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2753
$N(\text{param})_{\text{refined}}$:	235
Programs:	Bruker programs [1], SHELX [2]

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Abstract

$C_{19}H_{19}F_3FeN_2O$, monoclinic, $P2_1/c$ (no. 14), $a = 10.9948(6)$ Å, $b = 21.9829(10)$ Å, $c = 7.8962(4)$ Å, $\beta = 108.643(2)^\circ$, $V = 1808.35(16)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0380$, $wR_{\text{ref}}(F^2) = 0.0934$, $T = 273(2)$ K.

CCDC no.: 1824876

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 material

A mixture of 5-ferrocenyl-3-(trifluoromethyl)-1*H*-pyrazole (3.20 g, 0.01 mol, prepared by a literature method [3]), triethylamine (1.21 g, 0.012 mol), and valeryl chloride (1.45 g, 0.012 mol) in 15 mL of CH_2Cl_2 were refluxed overnight. After the completion of the reaction, the mixture was evaporated under reduced pressure. The residue was dissolved in ethyl acetate and extracted with water three times to remove the triethylamine. The organic layer was dried over Na_2SO_4 , filtered and evaporated to yield the crude mixture. The

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.88725(3)	0.12843(2)	0.98636(4)	0.04861(13)
F1	0.53190(18)	−0.00312(7)	0.3031(2)	0.0895(6)
F2	0.36621(15)	0.04874(8)	0.2900(2)	0.0769(5)
F3	0.45909(16)	0.06054(7)	0.09188(18)	0.0759(5)
N1	0.64979(17)	0.18722(9)	0.5076(2)	0.0469(5)
N2	0.54901(17)	0.15912(8)	0.3813(2)	0.0447(4)
O1	0.35650(18)	0.16783(9)	0.1691(3)	0.0832(6)
C1	0.8847(3)	0.12795(15)	1.2430(4)	0.0761(9)
H1A	0.933210	0.155376	1.338608	0.091*
C2	0.9270(3)	0.07202(14)	1.2009(4)	0.0822(10)
H2A	1.010156	0.053094	1.262042	0.099*
C3	0.8280(4)	0.04698(15)	1.0548(4)	0.0875(10)
H3A	0.830885	0.007791	0.997084	0.105*
C4	0.7264(3)	0.08821(17)	1.0074(4)	0.0810(10)
H4A	0.645102	0.083047	0.910530	0.097*
C5	0.7605(3)	0.13835(17)	1.1245(4)	0.0808(9)
H5A	0.707188	0.174200	1.123120	0.097*
C6	1.0009(3)	0.19920(17)	0.9659(4)	0.0849(10)
H6A	1.044787	0.227446	1.062063	0.102*
C7	1.0510(3)	0.1444(2)	0.9266(4)	0.0893(11)
H7A	1.135956	0.127816	0.991126	0.107*
C8	0.9577(2)	0.11627(16)	0.7802(4)	0.0729(8)
H8A	0.966815	0.077451	0.724396	0.088*
C9	0.8484(2)	0.15522(12)	0.7292(3)	0.0542(6)
C10	0.8753(3)	0.20631(12)	0.8448(4)	0.0693(8)
H10A	0.817260	0.240464	0.841406	0.083*
C11	0.7285(2)	0.14287(11)	0.5841(3)	0.0464(5)
C12	0.6814(2)	0.08579(11)	0.5085(3)	0.0502(6)
H12A	0.720481	0.048079	0.539615	0.060*
C13	0.5684(2)	0.09732(10)	0.3823(3)	0.0467(5)

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C14	0.4797(3)	0.05169(12)	0.2648(3)	0.0583(6)
C15	0.4452(2)	0.19398(11)	0.2716(3)	0.0517(6)
C16	0.4569(2)	0.26102(11)	0.2917(3)	0.0528(6)
H16A	0.527692	0.274839	0.253250	0.063*
H16B	0.476232	0.271554	0.416808	0.063*
C17	0.3345(2)	0.29349(12)	0.1828(3)	0.0607(7)
H17A	0.311562	0.280138	0.059500	0.073*
H17B	0.265407	0.281847	0.227750	0.073*
C18	0.3465(3)	0.36106(12)	0.1885(4)	0.0689(7)
H18A	0.417538	0.372902	0.147865	0.083*
H18B	0.365335	0.374854	0.310932	0.083*
C19	0.2237(3)	0.39176(16)	0.0715(5)	0.0892(10)
H19A	0.234738	0.435111	0.078247	0.134*
H19B	0.205706	0.378751	−0.050132	0.134*
H19C	0.153546	0.380702	0.112799	0.134*

title compound was obtained by column chromatography on silica gel using petroleum ether/ethylacetate (4:1, v/v, R_f = 0.3) as eluent. Single crystals were obtained by the slow evaporation of dichloromethane/methanol solutions.

Experimental details

All the non-hydrogen atoms were located in successive difference Fourier syntheses. The final refinement was performed by full-matrix least-squares methods with anisotropic thermal parameters for non-hydrogen atoms. The hydrogen atoms were added geometrically and riding on the concerned atoms.

Discussion

The use of ferrocene-containing compounds for medicinal applications has long been considered as an attractive way to develop anticancer drugs [4, 5], due to its low toxicity, significant stability, lipophilicity, and facile functionalization [6]. For example, the substitution of one of the phenyl rings of tamoxifen with a ferrocenyl group obtains the so-called ‘ferrocifen’ derivative with improved antiproliferative activity [7]. Therefore, there is an increasing interest focused on the discovery of new compounds with a ferrocene core, suitable to develop active drugs, for example, as potential chemotherapeutic agents. The ferrocenyl moiety has been incorporated with some natural products [8], resulting in an increase of their biological activities or create new properties.

We have an on-going interest in studying the anticancer activity of ferrocene-based compounds. Previous studies

from our groups have shown that ferrocene analogs containing a pyrazolyl group could display impressive anticancer activity and act as effective metallodrugs [9].

The title compound crystallizes in the space group *P*2₁/*c* with one molecule in the asymmetric unit. The principal geometric parameters are in the expected ranges [10]. The average bond length of Fe1–C (Cp1–5) is 2.036 Å, which is comparable to that (2.029 Å) of the other five-member ring (Cp6–10). The longest bond length is Fe1···C4 (2.04 Å), and the shortest Fe–C (Cp) separation is 2.02 Å (Fe1···C9).

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