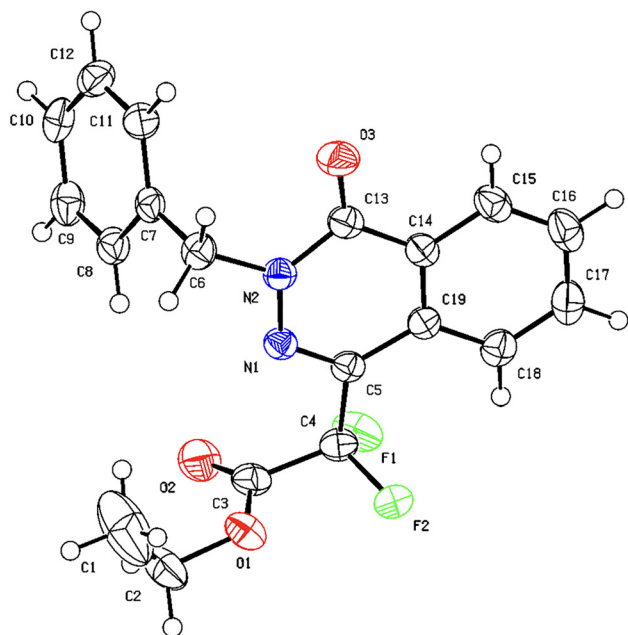


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Crystal structure of ethyl 2-(3-benzyl-4-oxo-3,4-dihydrophthalazin-1-yl)-2,2-difluoroacetate, $C_{19}H_{16}F_2N_2O_3$

**Table 1:** Data collection and handling.

Crystal:	Colourless block
Size:	0.43 × 0.29 × 0.26 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.5°, >99 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	15,133, 3852, 0.064
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 2734
$N(\text{param})_{\text{refined}}$:	236
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

1 Source of material

To an oven-dried Schlenk tube equipped with a magnetic stir bar were sequentially added phenylhydrazine (21.6 mg, 0.2 mmol), 2-formylbenzoic acid (30.0 mg, 0.2 mmol), ethyl 2-bromo-2,2-difluoroacetate (100.9 mg, 0.5 mmol), *fac*-[Ir(ppy)₃] (1.3 mg, 1.0 mol%), DBU (30.4 mg, 0.2 mmol) and THF (2.0 mL). After being degassed and refilled with nitrogen three times, the tube was sealed, and the reaction mixture was stirred under 10 W blue LED irradiation at room temperature for 30 h. After that, the solvent was removed by vacuum evaporation, and the crude residue was purified by column chromatography on silica gel using a mixture of ethyl acetate and hexanes (1:10 to 1:8) to give the desired product.

2 Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

3 Comment

Nitrogen-containing heterocyclic compounds are widely distributed in nature and play an important role in

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Abstract

$C_{19}H_{16}F_2N_2O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 17.5688(15)$ Å, $b = 13.8304(11)$ Å, $c = 7.0600(7)$ Å, $\beta = 100.254(3)^\circ$, $V = 1688.1(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0516$, $wR_{\text{ref}}(F^2) = 0.1424$, $T = 200(2)$ K.

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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.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
F1	0.58735 (6)	0.35329 (9)	0.49860 (17)	0.0502 (3)
F2	0.57371 (6)	0.33722 (8)	0.79582 (17)	0.0462 (3)
O1	0.57209 (7)	0.51988 (9)	0.8477 (2)	0.0441 (3)
O2	0.58564 (8)	0.54667 (11)	0.5401 (2)	0.0519 (4)
O3	0.92893 (7)	0.37812 (10)	0.8758 (2)	0.0461 (4)
N1	0.73490 (8)	0.45593 (10)	0.7701 (2)	0.0298 (3)
N2	0.81293 (8)	0.45416 (10)	0.8208 (2)	0.0302 (3)
C1	0.6157 (2)	0.6720 (2)	0.9853 (6)	0.1120 (14)
H1A	0.628744	0.644241	1.114749	0.168*
H1B	0.602074	0.740315	0.994792	0.168*
H1C	0.660329	0.666441	0.920152	0.168*
C2	0.55076 (13)	0.62034 (15)	0.8765 (4)	0.0567 (6)
H2A	0.534388	0.652012	0.750022	0.068*
H2B	0.506693	0.622137	0.946426	0.068*
C3	0.58809 (9)	0.49637 (13)	0.6777 (3)	0.0366 (4)
C4	0.61203 (10)	0.38930 (13)	0.6776 (3)	0.0341 (4)
C5	0.69799 (9)	0.37527 (11)	0.7346 (2)	0.0283 (4)
C6	0.84907 (10)	0.55012 (12)	0.8454 (3)	0.0346 (4)
H6A	0.899181	0.545302	0.934835	0.042*
H6B	0.815343	0.594512	0.903145	0.042*
C7	0.86224 (9)	0.59079 (11)	0.6563 (2)	0.0301 (4)
C8	0.80080 (10)	0.62926 (12)	0.5259 (3)	0.0366 (4)
H8	0.750159	0.628894	0.555674	0.044*
C9	0.81312 (11)	0.66782 (13)	0.3538 (3)	0.0419 (5)
H9	0.771043	0.694739	0.266512	0.050*
C10	0.88630 (12)	0.66755 (12)	0.3076 (3)	0.0402 (5)
H10	0.894634	0.694300	0.189000	0.048*
C11	0.93525 (10)	0.59060 (13)	0.6086 (3)	0.0367 (4)
H11	0.977635	0.564432	0.695953	0.044*
C12	0.94705 (11)	0.62831 (13)	0.4342 (3)	0.0408 (5)
H12	0.997287	0.627059	0.402047	0.049*
C13	0.85840 (10)	0.37194 (12)	0.8352 (3)	0.0326 (4)
C14	0.81552 (10)	0.28099 (12)	0.7995 (2)	0.0306 (4)
C15	0.85613 (11)	0.19378 (13)	0.8177 (3)	0.0393 (4)
H15	0.910988	0.193888	0.849918	0.047*
C16	0.81664 (13)	0.10777 (14)	0.7890 (3)	0.0447 (5)
H16	0.844111	0.048290	0.803533	0.054*
C17	0.73623 (12)	0.10781 (13)	0.7385 (3)	0.0427 (5)
H17	0.709307	0.048047	0.719319	0.051*
C18	0.69529 (11)	0.19299 (13)	0.7160 (3)	0.0362 (4)
H18	0.640608	0.192021	0.678676	0.043*
C19	0.73472 (9)	0.28143 (12)	0.7483 (2)	0.0292 (4)

agricultural chemicals, chemistry, and medicine. Dihydrophthalazine is an azo heterocyclic compound. Modern

pharmacological studies have shown that dihydrophthalazine derivatives have biological activities [5–7]. Therefore, researchers are increasingly interested in efficient methods for synthesizing dihydrophthalazine compounds.

The title compound has one carbonyl group, one ester group and difluoromethyl. The bond lengths and angles which were derived from the title structure are within normal ranges [8, 9]. The carbonyl group was determined at the distance of 1.224(2) Å (C₁₃–O₃). The ester group was 1.321(2) Å (C₃–O₁), 1.190(2) Å (C₃–O₂). The methylene dioxygen groups were 1.356(2) Å (C₄–F₁), 1.367(2) Å (C₄–F₂).

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