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3-(5-Oxo-3-phenyl-4,5-dihydro-1H-pyrazol-1-yl)benzonitrile

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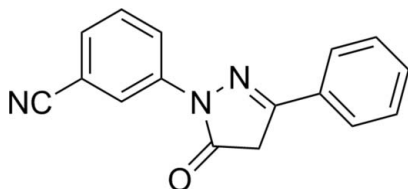
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.119; data-to-parameter ratio = 14.3.

In the title compound, $\text{C}_{16}\text{H}_{11}\text{N}_3\text{O}$, the dihedral angles between the 3-cyanobenzene and benzene planes and the 1H-pyrazol-5(4H)-one plane are 4.97 (9) and 9.91 (9)°, respectively.

Related literature

For a similar structure, see: Paulis *et al.* (2006).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{11}\text{N}_3\text{O}$
 $M_r = 261.28$

Monoclinic, $P2_1/c$
 $a = 7.6683$ (3) Å

$b = 17.8013$ (7) Å
 $c = 9.7574$ (4) Å
 $\beta = 106.506$ (4)°
 $V = 1277.05$ (9) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293$ K
 $0.34 \times 0.30 \times 0.28$ mm

Data collection

Agilent Xcalibur diffractometer
with an Eos CCD detector
Absorption correction: multi-scan
(*CrysAlis PRO*; Agilent, 2011)
 $T_{\min} = 0.967$, $T_{\max} = 1.000$

5145 measured reflections
2608 independent reflections
1686 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.119$
 $S = 1.02$
2608 reflections

182 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.14$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.13$ e Å⁻³

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *OLEX2.solve* (Bourhis *et al.*, 2012); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2212).

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supporting information

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3-(5-Oxo-3-phenyl-4,5-dihydro-1H-pyrazol-1-yl)benzotrile

Ling Li, Rong-sheng Tong, Jin-qi Li and Jian-you Shi

S1. Comment

In our research, 1,3-Diphenyl-1H-pyrazol-5(4H)-one is a member of a series of compounds which are being investigated for their potential as anticancer agents. In the analogous title compound, C₁₆H₁₁N₃O, (Fig. 1), the dihedral angles between the 3-cyanobenzene and benzene planes and the 1H-pyrazol-5(4)-one plane are 4.97 (9)° and 9.91 (9)°, respectively. Present also in the structure are intramolecular aromatic C—H···N and C—H···O interactions (Table 1). A similar structure has been previously been reported (Paulis *et al.*, 2006).

S2. Experimental

A mixture of 3-hydrazinylbenzotrile hydrochloride (1.96 g, 0.01 mol) and ethyl 3-oxo-3-phenylpropanoate (1.92 g, 0.01 mol) in acetic acid (50 mL) was heated under reflux for 1.5 h, then poured into ice water. The precipitated product was filtered, giving the title compound as a powder. Single crystals were by obtained by room temperature evaporation of a solution in CH₂Cl₂–MeOH after 5 days.

S3. Refinement

H atoms were positioned geometrically (C—H = 0.95–0.98 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

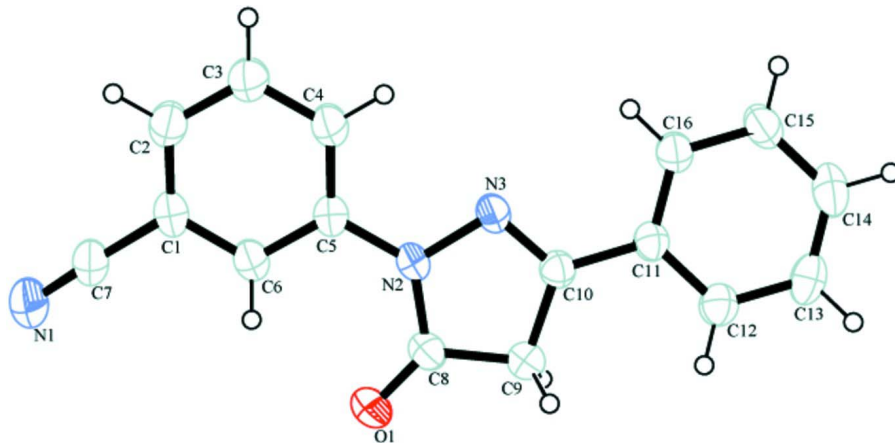


Figure 1

The molecular structure of title compound showing atom numbering, with displacement ellipsoids drawn at the 40% probability level.

3-(5-Oxo-3-phenyl-4,5-dihydro-1H-pyrazol-1-yl)benzotrile

Crystal data

C₁₆H₁₁N₃O $M_r = 261.28$ Monoclinic, $P2_1/c$ $a = 7.6683$ (3) Å $b = 17.8013$ (7) Å $c = 9.7574$ (4) Å $\beta = 106.506$ (4)° $V = 1277.05$ (9) Å³ $Z = 4$ $F(000) = 544$ $D_x = 1.359$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å

Cell parameters from 1663 reflections

 $\theta = 3.0$ – 29.1 ° $\mu = 0.09$ mm⁻¹ $T = 293$ K

Block, orange

 $0.34 \times 0.30 \times 0.28$ mm

Data collection

Agilent Xcalibur

diffractometer with an Eos CCD detector

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.0874 pixels mm⁻¹ ω scans

Absorption correction: multi-scan

(CrysAlis PRO; Agilent, 2011)

 $T_{\min} = 0.967$, $T_{\max} = 1.000$

5145 measured reflections

2608 independent reflections

1686 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.023$ $\theta_{\max} = 26.4$ °, $\theta_{\min} = 3.0$ ° $h = -9 \rightarrow 9$ $k = -10 \rightarrow 22$ $l = -12 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.048$ $wR(F^2) = 0.119$ $S = 1.02$

2608 reflections

182 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier

map

Hydrogen site location: inferred from

neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0461P)^2 + 0.0009P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.14$ e Å⁻³ $\Delta\rho_{\min} = -0.13$ e Å⁻³

Extinction correction: SHELXL97 (Sheldrick,

2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0083 (15)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.10361 (18)	0.26129 (9)	0.88140 (16)	0.0787 (5)
N1	-0.4410 (2)	0.40574 (12)	1.0212 (2)	0.0843 (6)
N2	0.14347 (17)	0.36652 (9)	0.75427 (15)	0.0460 (4)

N3	0.25680 (17)	0.38881 (9)	0.67037 (14)	0.0453 (4)
C1	-0.2203 (2)	0.45145 (12)	0.88230 (18)	0.0505 (5)
C2	-0.2216 (2)	0.52432 (12)	0.8356 (2)	0.0588 (5)
H2	-0.3015	0.5594	0.8547	0.071*
C3	-0.1018 (2)	0.54437 (13)	0.7599 (2)	0.0658 (6)
H3	-0.1013	0.5934	0.7272	0.079*
C4	0.0173 (2)	0.49230 (12)	0.7321 (2)	0.0574 (5)
H4	0.0964	0.5064	0.6800	0.069*
C5	0.0196 (2)	0.41964 (11)	0.78127 (17)	0.0446 (5)
C6	-0.1013 (2)	0.39869 (12)	0.85627 (17)	0.0502 (5)
H6	-0.1024	0.3496	0.8888	0.060*
C7	-0.3450 (2)	0.42714 (12)	0.9599 (2)	0.0597 (6)
C8	0.1780 (2)	0.29424 (12)	0.8051 (2)	0.0521 (5)
C9	0.3237 (2)	0.26591 (11)	0.7431 (2)	0.0535 (5)
H9A	0.4313	0.2508	0.8176	0.064*
H9B	0.2809	0.2239	0.6791	0.064*
C10	0.3602 (2)	0.33246 (10)	0.66476 (16)	0.0413 (4)
C11	0.4987 (2)	0.33671 (10)	0.58749 (17)	0.0415 (4)
C12	0.6261 (2)	0.27999 (12)	0.60224 (19)	0.0518 (5)
H12	0.6224	0.2387	0.6598	0.062*
C13	0.7591 (2)	0.28432 (12)	0.5318 (2)	0.0574 (6)
H13	0.8448	0.2462	0.5428	0.069*
C14	0.7649 (2)	0.34450 (13)	0.4461 (2)	0.0622 (6)
H14	0.8542	0.3472	0.3987	0.075*
C15	0.6379 (2)	0.40112 (12)	0.4301 (2)	0.0627 (6)
H15	0.6411	0.4420	0.3714	0.075*
C16	0.5064 (2)	0.39714 (11)	0.50101 (19)	0.0527 (5)
H16	0.4218	0.4357	0.4904	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0755 (9)	0.0780 (12)	0.1024 (11)	0.0167 (8)	0.0571 (9)	0.0415 (10)
N1	0.0917 (12)	0.0754 (15)	0.1115 (14)	0.0126 (11)	0.0702 (12)	0.0119 (13)
N2	0.0474 (8)	0.0476 (10)	0.0511 (9)	0.0017 (7)	0.0271 (7)	0.0071 (8)
N3	0.0486 (8)	0.0450 (10)	0.0495 (8)	-0.0003 (7)	0.0256 (7)	0.0014 (8)
C1	0.0449 (10)	0.0616 (14)	0.0504 (10)	-0.0010 (9)	0.0222 (8)	-0.0041 (11)
C2	0.0574 (11)	0.0548 (14)	0.0718 (13)	0.0049 (10)	0.0310 (10)	-0.0039 (12)
C3	0.0687 (13)	0.0503 (14)	0.0912 (14)	0.0033 (10)	0.0436 (12)	0.0046 (13)
C4	0.0591 (12)	0.0523 (14)	0.0727 (13)	-0.0005 (10)	0.0380 (10)	0.0022 (11)
C5	0.0407 (9)	0.0496 (12)	0.0464 (10)	0.0001 (8)	0.0171 (8)	-0.0023 (9)
C6	0.0515 (10)	0.0528 (13)	0.0532 (11)	-0.0004 (9)	0.0264 (8)	0.0018 (10)
C7	0.0591 (12)	0.0620 (15)	0.0681 (12)	0.0062 (10)	0.0345 (10)	-0.0010 (12)
C8	0.0482 (10)	0.0575 (14)	0.0555 (11)	0.0037 (9)	0.0227 (9)	0.0151 (11)
C9	0.0534 (11)	0.0523 (13)	0.0606 (12)	0.0079 (9)	0.0257 (9)	0.0132 (11)
C10	0.0420 (9)	0.0424 (11)	0.0408 (9)	0.0002 (8)	0.0141 (7)	-0.0007 (9)
C11	0.0414 (9)	0.0431 (11)	0.0420 (9)	-0.0018 (8)	0.0153 (7)	-0.0044 (9)
C12	0.0559 (11)	0.0524 (13)	0.0503 (10)	0.0055 (9)	0.0203 (9)	-0.0023 (10)

C13	0.0493 (11)	0.0630 (15)	0.0632 (12)	0.0073 (10)	0.0214 (10)	-0.0177 (12)
C14	0.0557 (12)	0.0697 (16)	0.0726 (14)	-0.0102 (10)	0.0366 (10)	-0.0184 (13)
C15	0.0710 (13)	0.0543 (14)	0.0783 (14)	-0.0056 (11)	0.0464 (11)	0.0027 (12)
C16	0.0563 (11)	0.0451 (12)	0.0658 (12)	0.0016 (9)	0.0319 (9)	0.0011 (10)

Geometric parameters (Å, °)

O1—C8	1.2105 (19)	C8—C9	1.501 (2)
N1—C7	1.138 (2)	C9—H9A	0.9700
N2—N3	1.4096 (17)	C9—H9B	0.9700
N2—C5	1.417 (2)	C9—C10	1.479 (2)
N2—C8	1.377 (2)	C10—C11	1.468 (2)
N3—C10	1.289 (2)	C11—C12	1.384 (2)
C1—C2	1.374 (3)	C11—C16	1.379 (2)
C1—C6	1.382 (2)	C12—H12	0.9300
C1—C7	1.445 (2)	C12—C13	1.384 (2)
C2—H2	0.9300	C13—H13	0.9300
C2—C3	1.379 (2)	C13—C14	1.368 (3)
C3—H3	0.9300	C14—H14	0.9300
C3—C4	1.381 (2)	C14—C15	1.379 (3)
C4—H4	0.9300	C15—H15	0.9300
C4—C5	1.378 (2)	C15—C16	1.377 (2)
C5—C6	1.386 (2)	C16—H16	0.9300
C6—H6	0.9300		
N3—N2—C5	118.46 (15)	C8—C9—H9B	111.3
C8—N2—N3	112.64 (13)	H9A—C9—H9B	109.2
C8—N2—C5	128.83 (14)	C10—C9—C8	102.19 (15)
C10—N3—N2	107.08 (14)	C10—C9—H9A	111.3
C2—C1—C6	121.59 (16)	C10—C9—H9B	111.3
C2—C1—C7	120.86 (18)	N3—C10—C9	113.07 (14)
C6—C1—C7	117.55 (19)	N3—C10—C11	121.66 (16)
C1—C2—H2	120.7	C11—C10—C9	125.27 (15)
C1—C2—C3	118.59 (18)	C12—C11—C10	120.09 (17)
C3—C2—H2	120.7	C16—C11—C10	121.17 (16)
C2—C3—H3	119.7	C16—C11—C12	118.73 (15)
C2—C3—C4	120.6 (2)	C11—C12—H12	119.8
C4—C3—H3	119.7	C13—C12—C11	120.39 (19)
C3—C4—H4	119.8	C13—C12—H12	119.8
C5—C4—C3	120.37 (17)	C12—C13—H13	119.9
C5—C4—H4	119.8	C14—C13—C12	120.24 (18)
C4—C5—N2	120.32 (15)	C14—C13—H13	119.9
C4—C5—C6	119.48 (17)	C13—C14—H14	120.1
C6—C5—N2	120.20 (17)	C13—C14—C15	119.82 (17)
C1—C6—C5	119.32 (19)	C15—C14—H14	120.1
C1—C6—H6	120.3	C14—C15—H15	120.0
C5—C6—H6	120.3	C16—C15—C14	119.97 (19)
N1—C7—C1	177.8 (2)	C16—C15—H15	120.0

O1—C8—N2	126.74 (17)	C11—C16—H16	119.6
O1—C8—C9	128.31 (18)	C15—C16—C11	120.84 (18)
N2—C8—C9	104.95 (14)	C15—C16—H16	119.6
C8—C9—H9A	111.3		
C5—N2—N3—C10	177.95 (14)	C3—C4—C5—C6	1.4 (3)
C8—N2—N3—C10	0.94 (18)	N2—C5—C6—C1	179.60 (15)
N3—N2—C5—C4	-3.1 (2)	C4—C5—C6—C1	-1.0 (2)
N3—N2—C5—C6	176.29 (14)	O1—C8—C9—C10	-178.40 (19)
C8—N2—C5—C4	173.33 (17)	N2—C8—C9—C10	2.61 (18)
C8—N2—C5—C6	-7.3 (3)	C8—C9—C10—N3	-2.28 (19)
N3—N2—C8—O1	178.67 (18)	C8—C9—C10—C11	177.51 (15)
N3—N2—C8—C9	-2.32 (19)	N3—C10—C11—C12	169.70 (16)
C5—N2—C8—O1	2.0 (3)	N3—C10—C11—C16	-9.4 (2)
C5—N2—C8—C9	-178.95 (16)	C9—C10—C11—C12	-10.1 (3)
N2—N3—C10—C9	0.97 (18)	C9—C10—C11—C16	170.82 (16)
N2—N3—C10—C11	-178.83 (14)	C10—C11—C12—C13	-178.80 (16)
C6—C1—C2—C3	0.6 (3)	C16—C11—C12—C13	0.3 (3)
C7—C1—C2—C3	-178.93 (17)	C10—C11—C16—C15	179.29 (16)
C2—C1—C6—C5	0.0 (3)	C12—C11—C16—C15	0.2 (3)
C7—C1—C6—C5	179.54 (16)	C11—C12—C13—C14	-0.5 (3)
C1—C2—C3—C4	-0.2 (3)	C12—C13—C14—C15	0.1 (3)
C2—C3—C4—C5	-0.8 (3)	C13—C14—C15—C16	0.4 (3)
C3—C4—C5—N2	-179.18 (16)	C14—C15—C16—C11	-0.5 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C4—H4...N3	0.93	2.44	2.785 (2)	102
C6—H6...O1	0.93	2.24	2.880 (3)	125