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

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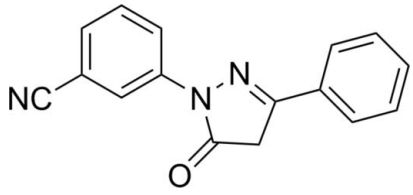
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.048; $wR$ factor = 0.119; data-to-parameter ratio = 14.3.

In the title compound, $C_{16}H_{11}N_3O$, the dihedral angles between the 3-cyanobenzene and benzene planes and the 1*H*-pyrazol-5(4*H*)-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*

$C_{16}H_{11}N_3O$
$M_r$ = 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) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 293 K
0.34 × 0.30 × 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 $I > 2\sigma(I)$
$R_{int}$ = 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
$\Delta\rho_{max}$ = 0.14 e Å$^{-3}$
$\Delta\rho_{min}$ = −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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# supplementary materials

*Acta Cryst.* (2012). E**68**, o2230 [doi:10.1107/S1600536812027444]

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

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

### Comment

In our research, 1,3-Diphenyl-1*H*-pyrazol-5(4*H*)-one is a member of a series of compounds which are being investigated for their potential as anticancer agents. In the analogous title compound, $C_{16}H_{11}N_3O$, (Fig. 1), the dihedral angles between the 3-cyanobenzene and benzene planes and the 1*H*-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 peviously been reported (Paulis *et al.*, 2006).

### Experimental

A mixture of 3-hydrazinylbenzonitrile 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 $CH_2Cl_2$–MeOH after 5 days.

### Refinement

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

### Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); 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 (Dolomanov *et al.*, 2009).

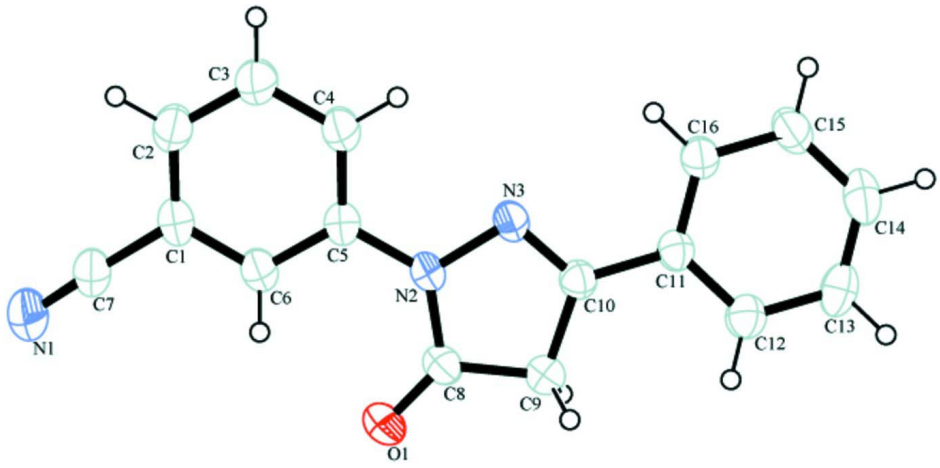


**Figure 1**

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-1*H*-pyrazol-1-yl)benzonitrile

*Crystal data*

$C_{16}H_{11}N_3O$
$M_r = 261.28$
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) Å$^3$
$Z = 4$
$F(000) = 544$
$D_x = 1.359$ Mg m$^{-3}$
Mo $K\alpha$ 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 × 0.30 × 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$^{-1}$
$\omega$ 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 $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.0°$
$h = -9\rightarrow9$
$k = -10\rightarrow22$
$l = -12\rightarrow11$

*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), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$

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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4···N3 | 0.93 | 2.44 | 2.785 (2) | 102 |
| C6—H6···O1 | 0.93 | 2.24 | 2.880 (3) | 125 |