organic compounds

1-Phenyl-1*H*-pyrazole-4-carbaldehyde

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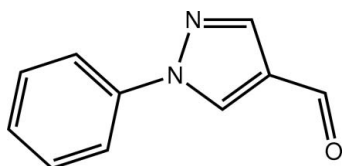Correspondence e-mail: edward.tiekink@gmail.com

In the title molecule, $C_{10}H_8N_2O$, the five- and six-membered rings form a dihedral angle of 10.14 (9)°. The aldehyde group is almost coplanar with the pyrazole ring to which it is connected [O—C—C—C torsion angle = −179.35 (17)°]. In the crystal, inversion dimers are linked by four C—H···O interactions as the carbonyl O atom accepts two such bonds. The dimeric aggregates are linked into supramolecular layers in the *ac* plane by C—H···π and π–π [ring centroid(pyrrole)···ring centroid(phenyl) = 3.8058 (10) Å] interactions.

Related literature

For the anti-bacterial properties of pyrazole derivatives, see: Kane *et al.* (2003). For related structures, see: Asiri, Al-Youbi, *et al.* (2012); Asiri, Faidallah *et al.* (2012). For the synthesis, see: Vera-DiVaio *et al.* (2009).

Experimental

Crystal data

$C_{10}H_8N_2O$	$V = 810.60$ (11) Å³
$M_r = 172.18$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 11.1657$ (10) Å	$\mu = 0.10$ mm⁻¹
$b = 5.0858$ (4) Å	$T = 100$ K
$c = 15.3034$ (11) Å	$0.30 \times 0.30 \times 0.15$ mm
$\beta = 111.130$ (9)°	

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	3485 measured reflections
	1814 independent reflections
	1359 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011) $T_{min} = 0.972$, $T_{max} = 0.986$	$R_{int} = 0.050$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$	151 parameters
$wR(F^2) = 0.136$	All H-atom parameters refined
$S = 0.99$	$\Delta\rho_{max} = 0.28$ e Å⁻³
1814 reflections	$\Delta\rho_{min} = -0.26$ e Å⁻³

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the C5–C10 ring.

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1ⁱ	0.960 (19)	2.432 (19)	3.379 (2)	168.6 (13)
C10—H10···O1ⁱ	0.978 (19)	2.335 (19)	3.303 (2)	170.8 (16)
C8—H8···*Cg*1ⁱⁱ	0.978 (18)	2.947 (18)	3.761 (2)	141.4 (14)

Symmetry codes: (i) $-x, -y + 1, -z + 1$; (ii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$.

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5843).

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supporting information

1-Phenyl-1*H*-pyrazole-4-carbaldehyde

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S1. Comment

In continuation of structural studies of pyrazole derivatives (Asiri, Al-Youbi, *et al.*, 2012; Asiri, Faidallah *et al.*, 2012), of interest, for example, owing to their anti-bacterial activity (Kane *et al.*, 2003), the title compound, 1-phenyl-1*H*-pyrazole-4-carbaldehyde (I), was investigated crystallographically.

In (I), Fig. 1, the dihedral angle between the five- and six-membered rings is 10.14 (9) °, indicating a slight twist in the molecule. The aldehyde group is co-planar with the pyrazole ring to which it is connected as seen in the value of the O1—C1—C2—C3 torsion angle of -179.35 (17)°.

Inversion related molecules are connected into dimers *via* C—H⋯O interactions involving a bifurcated carbonyl-O atom, Table 1. Dimers are linked into supramolecular layers in the *ac* plane *via* C—H⋯π and π—π interactions occurring between the five- and six-membered rings [ring centroid⋯ring centroid distance = 3.8058 (10) Å, angle of inclination = 10.14 (9)° for symmetry operation: *x*, -1 + *y*, *z*], Fig. 2 and Table 1. Layers stack with no specific intermolecular interactions between them, Fig. 3.

S2. Experimental

N,N-Dimethylformamide (25.6 ml, 0.33 mmol) was stirred in around flask within an ice-bath and POCl$_3$ (21.6 ml, 0.23 mmol) was added drop-wise. Then *N*-phenylpyrazole was added (4.4 ml, 0.033 mmol) to this cold mixture. The reaction was allowed to warm to room temperature and then heated at reflux for 6 h. The temperature was kept at 368–373 K. When the reaction was completed, the contents were poured onto crushed ice and made weakly alkaline with a saturated solution of sodium carbonate. The solid was filtered off, washed with water and recrystallized from ethanol. Yield: 65%. *M*.pt. 358–359 K. (lit. 358 K; Vera-DiVaio *et al.*, 2009).

S3. Refinement

H-atoms were freely refined; the range of C—H bond lengths is 0.960 (17)–1.023 (18) Å. Owing to poor agreement, the ($\bar{3}$ 2 6) reflection was omitted from the final cycles of refinement.
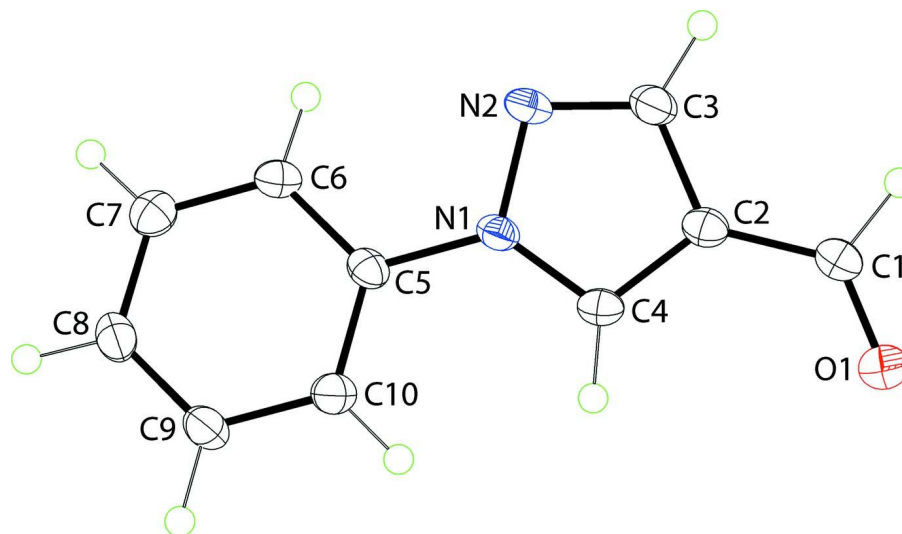
Figure 1

The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level.
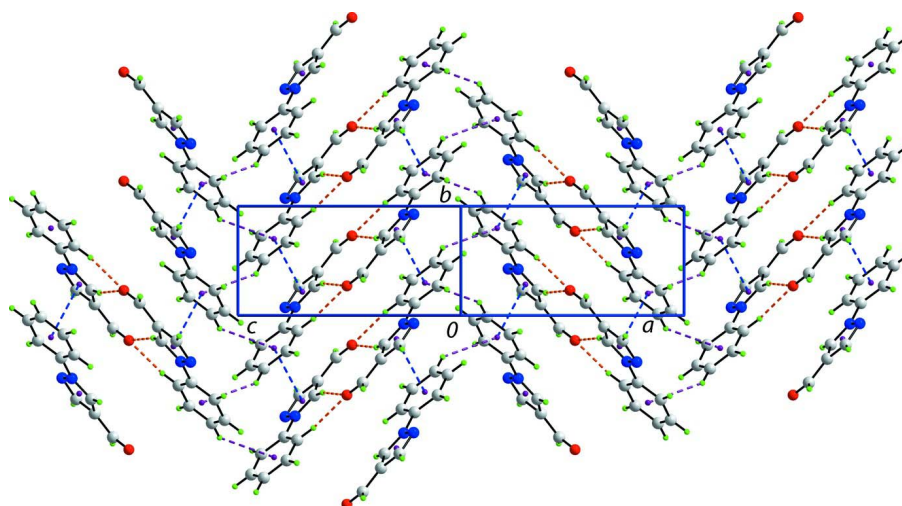
Figure 2

A view of the supramolecular layer in the *ac* plane in (I). The C—H···O, C—H···π and π—π interactions are shown as orange, purple and blue dashed lines, respectively.
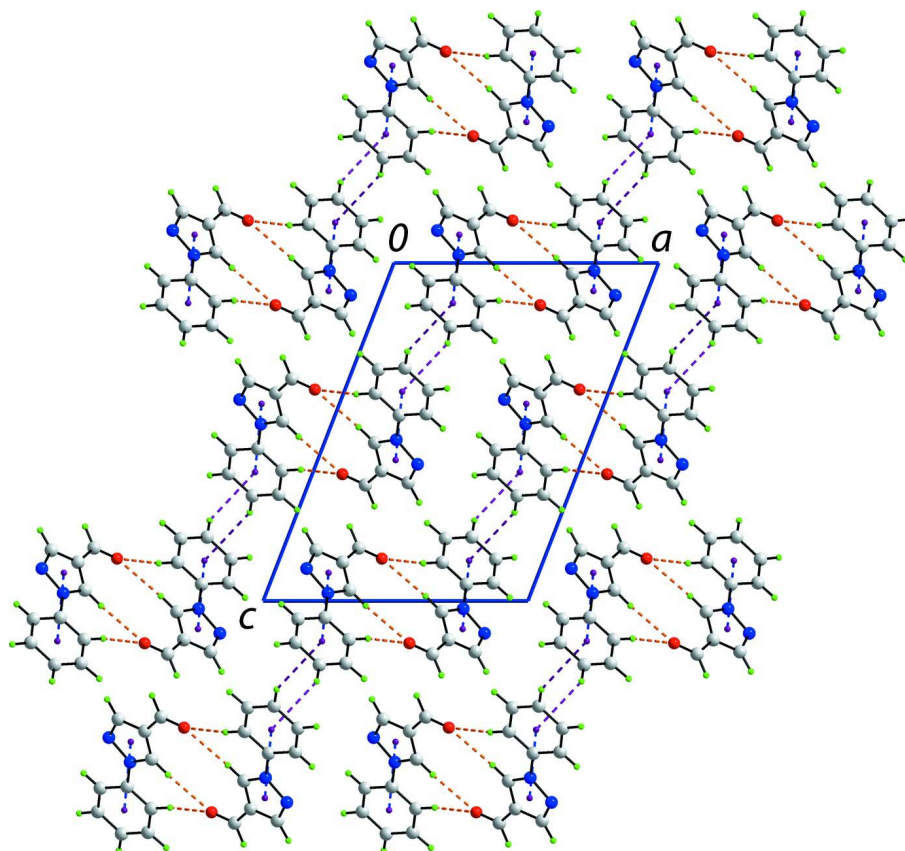
Figure 3

A view in projection down the *b* axis of the unit-cell contents of (I). The C—H···O, C—H···π and π—π interactions are shown as orange, purple and blue dashed lines, respectively.

1-Phenyl-1*H*-pyrazole-4-carbaldehyde

Crystal data

$C_{10}H_8N_2O$	$F(000) = 360$
$M_r = 172.18$	$D_x = 1.411$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1251 reflections
$a = 11.1657$ (10) Å	$\theta = 2.8–27.5°$
$b = 5.0858$ (4) Å	$\mu = 0.10$ mm^{-1}
$c = 15.3034$ (11) Å	$T = 100$ K
$\beta = 111.130$ (9)°	Prism, light-brown
$V = 810.60$ (11) Å^3	$0.30 \times 0.30 \times 0.15$ mm
$Z = 4$	

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
Radiation source: SuperNova (Mo) X-ray Source	$T_{min} = 0.972$, $T_{max} = 0.986$
Mirror monochromator	3485 measured reflections
Detector resolution: 10.4041 pixels mm^{-1}	1814 independent reflections
ω scan	1359 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.050$

$\theta_{max} = 27.6°$, $\theta_{min} = 2.8°$

$h = -14 \rightarrow 14$

$k = -5 \rightarrow 6$

$l = -19 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.050$

$wR(F^2) = 0.136$

$S = 0.99$

1814 reflections

151 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

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0732P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.28$ e Å^{-3}

$\Delta\rho_{min} = -0.26$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$

Extinction coefficient: 0.013 (4)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.11384 (11)	0.2706 (2)	0.62516 (8)	0.0286 (4)
N1	0.26714 (12)	0.9228 (3)	0.52304 (9)	0.0196 (3)
N2	0.38346 (13)	0.9268 (3)	0.59584 (9)	0.0247 (4)
C1	0.21644 (15)	0.3842 (3)	0.65682 (11)	0.0230 (4)
C2	0.25665 (15)	0.6026 (3)	0.61478 (11)	0.0209 (4)
C3	0.37465 (16)	0.7329 (3)	0.65005 (12)	0.0245 (4)
C4	0.18990 (15)	0.7336 (3)	0.53228 (11)	0.0204 (4)
C5	0.24267 (15)	1.1131 (3)	0.45059 (10)	0.0196 (4)
C6	0.34207 (16)	1.2698 (3)	0.44624 (11)	0.0226 (4)
C7	0.31714 (17)	1.4569 (4)	0.37669 (12)	0.0248 (4)
C8	0.19463 (17)	1.4863 (4)	0.31105 (11)	0.0247 (4)
C9	0.09706 (17)	1.3272 (4)	0.31553 (12)	0.0263 (4)
C10	0.11990 (16)	1.1412 (3)	0.38573 (12)	0.0242 (4)
H1	0.2818 (16)	0.327 (4)	0.7201 (13)	0.022 (4)*
H3	0.4456 (17)	0.689 (4)	0.7089 (13)	0.027 (5)*
H4	0.1060 (17)	0.707 (3)	0.4858 (12)	0.022 (5)*
H6	0.4267 (19)	1.239 (4)	0.4903 (14)	0.033 (5)*
H7	0.3846 (16)	1.567 (4)	0.3715 (12)	0.023 (5)*
H8	0.1801 (15)	1.619 (4)	0.2621 (12)	0.019 (4)*
H9	0.0057 (18)	1.351 (4)	0.2696 (14)	0.037 (5)*
H10	0.0500 (15)	1.030 (4)	0.3886 (12)	0.023 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0250 (7)	0.0306 (8)	0.0274 (6)	−0.0045 (5)	0.0063 (5)	−0.0006 (6)
N1	0.0183 (7)	0.0214 (8)	0.0154 (6)	0.0002 (5)	0.0015 (5)	−0.0005 (6)
N2	0.0193 (7)	0.0286 (8)	0.0194 (7)	−0.0011 (6)	−0.0013 (6)	0.0005 (6)
C1	0.0229 (9)	0.0254 (9)	0.0184 (8)	0.0017 (7)	0.0046 (7)	−0.0011 (7)
C2	0.0205 (8)	0.0216 (9)	0.0186 (7)	0.0007 (6)	0.0047 (6)	−0.0032 (7)

C3	0.0225 (9)	0.0270 (10)	0.0192 (8)	0.0005 (7)	0.0018 (7)	0.0001 (7)
C4	0.0182 (8)	0.0220 (9)	0.0185 (7)	−0.0007 (6)	0.0035 (7)	−0.0038 (7)
C5	0.0230 (8)	0.0197 (9)	0.0142 (7)	0.0011 (6)	0.0042 (6)	−0.0017 (6)
C6	0.0196 (9)	0.0263 (10)	0.0191 (8)	−0.0015 (7)	0.0038 (7)	−0.0026 (7)
C7	0.0271 (9)	0.0243 (9)	0.0238 (8)	−0.0024 (7)	0.0103 (7)	−0.0034 (8)
C8	0.0313 (9)	0.0205 (9)	0.0209 (8)	0.0021 (7)	0.0076 (7)	0.0027 (8)
C9	0.0242 (9)	0.0253 (10)	0.0236 (8)	0.0031 (7)	0.0017 (7)	0.0037 (8)
C10	0.0212 (9)	0.0222 (9)	0.0251 (8)	−0.0010 (7)	0.0034 (7)	0.0002 (8)

Geometric parameters (Å, º)

O1—C1	1.2168 (19)	C5—C10	1.380 (2)
N1—C4	1.333 (2)	C5—C6	1.387 (2)
N1—N2	1.3731 (17)	C6—C7	1.379 (2)
N1—C5	1.422 (2)	C6—H6	0.96 (2)
N2—C3	1.315 (2)	C7—C8	1.383 (2)
C1—C2	1.435 (2)	C7—H7	0.963 (18)
C1—H1	1.023 (18)	C8—C9	1.379 (3)
C2—C4	1.384 (2)	C8—H8	0.978 (18)
C2—C3	1.398 (2)	C9—C10	1.384 (2)
C3—H3	0.986 (18)	C9—H9	1.016 (18)
C4—H4	0.960 (17)	C10—H10	0.977 (17)
C4—N1—N2	112.57 (12)	C10—C5—N1	119.55 (14)
C4—N1—C5	128.61 (13)	C6—C5—N1	119.75 (14)
N2—N1—C5	118.81 (13)	C7—C6—C5	119.40 (15)
C3—N2—N1	103.68 (13)	C7—C6—H6	122.1 (12)
O1—C1—C2	126.22 (15)	C5—C6—H6	118.4 (12)
O1—C1—H1	119.3 (10)	C6—C7—C8	120.47 (16)
C2—C1—H1	114.5 (10)	C6—C7—H7	121.1 (11)
C4—C2—C3	104.28 (15)	C8—C7—H7	118.4 (11)
C4—C2—C1	129.01 (15)	C9—C8—C7	119.52 (17)
C3—C2—C1	126.71 (15)	C9—C8—H8	121.9 (10)
N2—C3—C2	112.73 (14)	C7—C8—H8	118.6 (10)
N2—C3—H3	121.7 (11)	C8—C9—C10	120.80 (16)
C2—C3—H3	125.6 (11)	C8—C9—H9	120.6 (11)
N1—C4—C2	106.74 (14)	C10—C9—H9	118.5 (11)
N1—C4—H4	121.3 (10)	C5—C10—C9	119.10 (16)
C2—C4—H4	131.9 (11)	C5—C10—H10	120.7 (10)
C10—C5—C6	120.70 (15)	C9—C10—H10	120.2 (10)
C4—N1—N2—C3	0.04 (18)	N2—N1—C5—C10	−169.04 (14)
C5—N1—N2—C3	179.12 (14)	C4—N1—C5—C6	−170.62 (15)
O1—C1—C2—C4	1.2 (3)	N2—N1—C5—C6	10.5 (2)
O1—C1—C2—C3	−179.35 (17)	C10—C5—C6—C7	0.6 (2)
N1—N2—C3—C2	0.16 (19)	N1—C5—C6—C7	−178.94 (14)
C4—C2—C3—N2	−0.3 (2)	C5—C6—C7—C8	−0.8 (3)
C1—C2—C3—N2	−179.83 (16)	C6—C7—C8—C9	0.0 (3)

N2—N1—C4—C2	−0.21 (18)	C7—C8—C9—C10	1.0 (3)
C5—N1—C4—C2	−179.18 (15)	C6—C5—C10—C9	0.4 (3)
C3—C2—C4—N1	0.29 (17)	N1—C5—C10—C9	179.95 (15)
C1—C2—C4—N1	179.81 (16)	C8—C9—C10—C5	−1.2 (3)
C4—N1—C5—C10	9.9 (3)		

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C5–C10 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1[i]	0.960 (19)	2.432 (19)	3.379 (2)	168.6 (13)
C10—H10···O1[i]	0.978 (19)	2.335 (19)	3.303 (2)	170.8 (16)
C8—H8···*Cg*1[ii]	0.978 (18)	2.947 (18)	3.761 (2)	141.4 (14)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2.