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3,4-Dimethyl-1-phenylpyrano[2,3-*c*]-pyrazol-6(1*H*)-one

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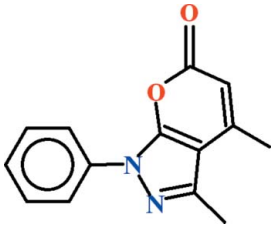
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.140; data-to-parameter ratio = 13.2.

In the title compound, $C_{14}H_{12}N_2O_2$, the dihedral angle between the phenyl ring and the 3,4-dimethylpyrano[2,3-*c*]-pyrazol-6(1*H*)-one system is 7.28 (6)°. An intramolecular C—H···O interaction generates an *S*(6) ring. In the crystal, the molecules are linked by C—H···O hydrogen bonds, forming *C*(8) chains. C–H···π and π–π interactions [centroid–centroid separation = 3.6374 (12) Å] further consolidate the packing.

Related literature

For a related structure, see: Ramsay & Steel (1985). For background to the pyrano[2,3-*c*]pyrazol-6-one ring system, see: Abdallah & Zaki (1999); Huang *et al.* (1992); Khan *et al.* (1982); Kuo *et al.* (1984); Ramsay & Steel (1985); Samaritoni *et al.* (2007). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{14}H_{12}N_2O_2$
M_r = 240.26
Monoclinic, *C*2/*c*
a = 15.1231 (9) Å
b = 13.3558 (8) Å
c = 13.8684 (8) Å
β = 120.965 (2)°
V = 2401.9 (3) Å^3
Z = 8
Mo *K*α radiation
μ = 0.09 mm^{-1}
T = 296 K
0.35 × 0.25 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.975, T_{max} = 0.982
9214 measured reflections
2171 independent reflections
1382 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.140
S = 1.02
2171 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

*Cg*3 is the centroid of the C1–C6 phenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{i}	0.93	2.51	3.407 (3)	163
C6—H6···O1	0.93	2.29	2.938 (3)	126
C14—H14*C*···*Cg*3^{ii}	0.96	2.75	3.506 (2)	136

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5827).

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supplementary materials

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3,4-Dimethyl-1-phenylpyrano[2,3-*c*]pyrazol-6(1*H*)-one

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Comment

The parent ring system pyrano[2,3-*c*]pyrazol-6-one is an isostere of coumarin. A number of its derivatives have been prepared from corresponding hydrazines and beta ketoesters (Khan *et al.*, 1982, Samaritoni *et al.*, 2007). It has been shown that these contain analgesic and anti-inflammatory activities (Kuo *et al.*, 1984, Abdallah & Zaki, 1999) and while others are tested for their antiplatelet activity (Huang *et al.*, 1992). The title compound (I, Fig. 1) has been synthesized and its crystal structure is being reported here.

The crystal structure of (II) *i.e.*, 3,4-dimethyl-1-(2-pyridyl)pyrano(2,3 - c)pyrazol-6(1*H*)-one (Ramsay & Steel, 1983) has been published which differs from (I) due to pyridal attachment instead of phenyl and hence is closely related.

In (I) the phyenyl ring A (C1–C6) and 3,4-dimethylpyrano[2,3-*c*]pyrazol -6(1*H*)-one moiety are almost planar with r. m. s. deviations of 0.003 and 0.023 Å, respectively. The dihedral angle between A/B is 7.28 (6)°. The molecules are linked by C(8) polymeric chains (Bernstein *et al.*, 1995) due to H-boning of C—H···O type (Table 1, Fig. 2). An intramolecular H-bonding and a C—H···π interaction (Table 1) also play an important role in stabilizing the molecules. There also exist π···π interactions between the pyrazole rings at a distance of 3.6374 (12) Å.

Experimental

A mixture of 3-methyl-1-phenylpyrazol-5-one (17.4 g, 0.1 mol) and ethyl acetoacetate (13 g, 0.1 mol) was heated at 413 K (oil bath) for an hour, cooled and triturated with 200 ml of pet. ether (bp. 313–333 K) and filtered to give the title compound. Light brown rods of (I) were obtained on recrystallization from ethanol. Yield 10.8 g, 45%: m.p. 415 K

Refinement

The H-atoms were positioned geometrically (C–H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = xU_{eq}(C), where x = 1.5 for methyl and x = 1.2 for aryl H-atoms.

Figures

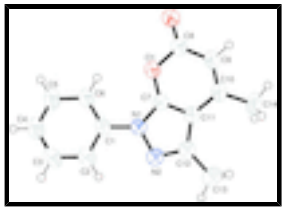

Fig. 1. View of (I) with displacement ellipsoids drawn at the 50% probability level.

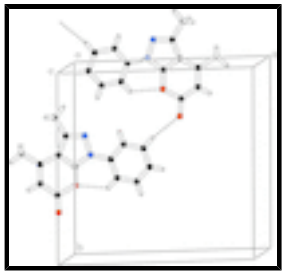

Fig. 2. Packing diagram of the title compound showing that polymeric chains are formed.

3,4-Dimethyl-1-phenylpyrano[2,3-*c*]pyrazol-6(1*H*)-one

Crystal data

$C_{14}H_{12}N_2O_2$	$F(000) = 1008$
$M_r = 240.26$	$D_x = 1.329$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 1382 reflections
$a = 15.1231$ (9) Å	$\theta = 2.2–25.3°$
$b = 13.3558$ (8) Å	$\mu = 0.09$ mm^{-1}
$c = 13.8684$ (8) Å	$T = 296$ K
$\beta = 120.965$ (2)°	Rod, light brown
$V = 2401.9$ (3) Å^3	0.35 × 0.25 × 0.25 mm
$Z = 8$	

Data collection

Bruker Kappa APEXII CCD diffractometer	2171 independent reflections
Radiation source: fine-focus sealed tube	1382 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.032$
Detector resolution: 8.10 pixels mm^{-1}	$\theta_{max} = 25.3°$, $\theta_{min} = 2.2°$
ω scans	$h = -18\rightarrow14$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$k = -15\rightarrow16$
$T_{min} = 0.975$, $T_{max} = 0.982$	$l = -9\rightarrow16$
9214 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.046$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.140$	H-atom parameters constrained
$S = 1.02$	$w = 1/[\sigma^2(F_o^2) + (0.0723P)^2 + 0.5405P]$ where $P = (F_o^2 + 2F_c^2)/3$
2171 reflections	$(\Delta/\sigma)_{max} < 0.001$

165 parameters	$\Delta\rho_{max} = 0.15$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.19$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.01582 (9)	0.63464 (9)	0.13521 (10)	0.0584 (5)
O2	−0.07464 (12)	0.77525 (12)	0.09062 (16)	0.0994 (7)
N1	0.09619 (12)	0.47598 (11)	0.16813 (13)	0.0526 (6)
N2	0.06513 (14)	0.37672 (12)	0.14972 (16)	0.0689 (7)
C1	0.20381 (14)	0.49922 (15)	0.22105 (15)	0.0526 (7)
C2	0.27219 (16)	0.42241 (17)	0.24254 (18)	0.0661 (8)
C3	0.37688 (16)	0.44318 (19)	0.2968 (2)	0.0794 (9)
C4	0.41168 (17)	0.5391 (2)	0.3279 (2)	0.0825 (9)
C5	0.34341 (16)	0.61512 (19)	0.30539 (19)	0.0770 (9)
C6	0.23861 (15)	0.59574 (16)	0.25142 (17)	0.0613 (8)
C7	0.01255 (14)	0.53448 (14)	0.13093 (15)	0.0484 (7)
C8	−0.08080 (15)	0.68528 (17)	0.08827 (18)	0.0659 (8)
C9	−0.17132 (15)	0.62522 (17)	0.04570 (17)	0.0638 (8)
C10	−0.17218 (14)	0.52456 (16)	0.04351 (15)	0.0537 (7)
C11	−0.07438 (14)	0.47628 (14)	0.08801 (16)	0.0519 (7)
C12	−0.03607 (17)	0.37748 (16)	0.10228 (19)	0.0657 (8)
C13	−0.0944 (2)	0.28099 (17)	0.0706 (3)	0.1025 (13)
C14	−0.27056 (15)	0.46714 (18)	−0.00325 (19)	0.0709 (8)
H2	0.24847	0.35727	0.22090	0.0793*
H3	0.42364	0.39162	0.31212	0.0953*
H4	0.48198	0.55248	0.36445	0.0990*
H5	0.36739	0.68028	0.32639	0.0924*
H6	0.19215	0.64766	0.23586	0.0736*
H9	−0.23416	0.65797	0.01749	0.0766*
H13A	−0.04834	0.22680	0.08183	0.1536*
H13B	−0.14832	0.28336	−0.00699	0.1536*
H13C	−0.12397	0.27081	0.11674	0.1536*
H14A	−0.32644	0.51265	−0.02276	0.1064*
H14B	−0.26542	0.42052	0.05210	0.1064*
H14C	−0.28305	0.43135	−0.06914	0.1064*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0380 (8)	0.0474 (9)	0.0769 (9)	0.0008 (6)	0.0203 (7)	−0.0016 (6)
O2	0.0515 (10)	0.0514 (11)	0.1565 (16)	0.0033 (8)	0.0259 (10)	−0.0053 (10)
N1	0.0409 (9)	0.0431 (10)	0.0687 (10)	0.0025 (8)	0.0245 (8)	0.0023 (7)
N2	0.0509 (12)	0.0454 (11)	0.1012 (13)	−0.0024 (8)	0.0325 (10)	0.0020 (9)
C1	0.0375 (11)	0.0555 (13)	0.0584 (11)	0.0036 (9)	0.0201 (9)	0.0015 (9)
C2	0.0503 (13)	0.0581 (14)	0.0807 (14)	0.0098 (11)	0.0272 (11)	0.0040 (10)
C3	0.0478 (14)	0.0753 (17)	0.1009 (17)	0.0200 (13)	0.0282 (13)	0.0069 (13)
C4	0.0374 (12)	0.0844 (18)	0.1021 (18)	0.0021 (13)	0.0191 (12)	−0.0092 (14)
C5	0.0428 (13)	0.0728 (16)	0.0992 (17)	−0.0039 (12)	0.0249 (12)	−0.0147 (13)
C6	0.0401 (12)	0.0550 (14)	0.0797 (13)	0.0014 (10)	0.0243 (10)	−0.0064 (10)
C7	0.0422 (11)	0.0434 (12)	0.0572 (11)	0.0013 (9)	0.0238 (9)	0.0009 (9)
C8	0.0431 (12)	0.0519 (14)	0.0856 (14)	0.0050 (11)	0.0208 (11)	−0.0010 (11)
C9	0.0357 (11)	0.0631 (15)	0.0793 (14)	0.0022 (10)	0.0201 (10)	−0.0039 (11)
C10	0.0411 (11)	0.0572 (13)	0.0587 (11)	−0.0032 (9)	0.0228 (9)	−0.0021 (9)
C11	0.0410 (11)	0.0496 (12)	0.0620 (12)	−0.0029 (9)	0.0244 (9)	−0.0002 (9)
C12	0.0525 (13)	0.0489 (14)	0.0905 (15)	−0.0057 (10)	0.0331 (12)	0.0013 (10)
C13	0.0688 (17)	0.0521 (16)	0.166 (3)	−0.0132 (13)	0.0457 (17)	−0.0009 (15)
C14	0.0444 (12)	0.0789 (16)	0.0838 (14)	−0.0161 (11)	0.0289 (11)	−0.0101 (12)

Geometric parameters (Å, °)

O1—C7	1.339 (2)	C10—C11	1.430 (3)
O1—C8	1.427 (3)	C10—C14	1.493 (3)
O2—C8	1.204 (3)	C11—C12	1.414 (3)
N1—N2	1.386 (2)	C12—C13	1.494 (4)
N1—C1	1.433 (3)	C2—H2	0.9300
N1—C7	1.344 (3)	C3—H3	0.9300
N2—C12	1.319 (4)	C4—H4	0.9300
C1—C2	1.376 (3)	C5—H5	0.9300
C1—C6	1.374 (3)	C6—H6	0.9300
C2—C3	1.387 (4)	C9—H9	0.9300
C3—C4	1.368 (4)	C13—H13A	0.9600
C4—C5	1.365 (4)	C13—H13B	0.9600
C5—C6	1.385 (4)	C13—H13C	0.9600
C7—C11	1.372 (3)	C14—H14A	0.9600
C8—C9	1.426 (3)	C14—H14B	0.9600
C9—C10	1.345 (3)	C14—H14C	0.9600
C7—O1—C8	116.58 (17)	N2—C12—C13	119.9 (2)
N2—N1—C1	119.31 (17)	C11—C12—C13	128.8 (3)
N2—N1—C7	108.83 (18)	C1—C2—H2	120.00
C1—N1—C7	131.86 (16)	C3—C2—H2	120.00
N1—N2—C12	106.32 (18)	C2—C3—H3	120.00
N1—C1—C2	118.57 (18)	C4—C3—H3	120.00
N1—C1—C6	121.0 (2)	C3—C4—H4	120.00

C2—C1—C6	120.4 (2)	C5—C4—H4	120.00
C1—C2—C3	119.3 (2)	C4—C5—H5	120.00
C2—C3—C4	120.3 (2)	C6—C5—H5	120.00
C3—C4—C5	120.1 (3)	C1—C6—H6	120.00
C4—C5—C6	120.4 (2)	C5—C6—H6	120.00
C1—C6—C5	119.5 (2)	C8—C9—H9	118.00
O1—C7—N1	123.92 (19)	C10—C9—H9	118.00
O1—C7—C11	126.2 (2)	C12—C13—H13A	109.00
N1—C7—C11	109.87 (17)	C12—C13—H13B	109.00
O1—C8—O2	114.4 (2)	C12—C13—H13C	109.00
O1—C8—C9	117.49 (19)	H13A—C13—H13B	109.00
O2—C8—C9	128.1 (2)	H13A—C13—H13C	109.00
C8—C9—C10	124.6 (2)	H13B—C13—H13C	109.00
C9—C10—C11	116.4 (2)	C10—C14—H14A	109.00
C9—C10—C14	121.3 (2)	C10—C14—H14B	109.00
C11—C10—C14	122.28 (19)	C10—C14—H14C	109.00
C7—C11—C10	118.64 (18)	H14A—C14—H14B	109.00
C7—C11—C12	103.7 (2)	H14A—C14—H14C	109.00
C10—C11—C12	137.7 (2)	H14B—C14—H14C	109.00
N2—C12—C11	111.3 (2)		
C8—O1—C7—N1	177.83 (18)	C1—C2—C3—C4	−0.5 (4)
C8—O1—C7—C11	−1.5 (3)	C2—C3—C4—C5	−0.2 (4)
C7—O1—C8—O2	−177.76 (19)	C3—C4—C5—C6	0.3 (4)
C7—O1—C8—C9	2.7 (3)	C4—C5—C6—C1	0.3 (3)
C1—N1—N2—C12	179.03 (18)	O1—C7—C11—C10	−0.4 (3)
C7—N1—N2—C12	−0.1 (2)	O1—C7—C11—C12	179.28 (19)
N2—N1—C1—C2	5.9 (3)	N1—C7—C11—C10	−179.82 (17)
N2—N1—C1—C6	−173.10 (19)	N1—C7—C11—C12	−0.1 (2)
C7—N1—C1—C2	−175.1 (2)	O1—C8—C9—C10	−2.2 (3)
C7—N1—C1—C6	5.8 (3)	O2—C8—C9—C10	178.3 (2)
N2—N1—C7—O1	−179.24 (17)	C8—C9—C10—C11	0.3 (3)
N2—N1—C7—C11	0.2 (2)	C8—C9—C10—C14	−179.6 (2)
C1—N1—C7—O1	1.7 (3)	C9—C10—C11—C7	1.0 (3)
C1—N1—C7—C11	−178.87 (19)	C9—C10—C11—C12	−178.6 (2)
N1—N2—C12—C11	0.1 (2)	C14—C10—C11—C7	−179.04 (19)
N1—N2—C12—C13	179.6 (2)	C14—C10—C11—C12	1.4 (4)
N1—C1—C2—C3	−178.0 (2)	C7—C11—C12—N2	0.0 (3)
C6—C1—C2—C3	1.0 (3)	C7—C11—C12—C13	−179.5 (3)
N1—C1—C6—C5	178.11 (19)	C10—C11—C12—N2	179.6 (2)
C2—C1—C6—C5	−0.9 (3)	C10—C11—C12—C13	0.1 (5)

Hydrogen-bond geometry (Å, °)

Cg3 is the centroid of the C1–C6 phenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O2^{i}	0.93	2.51	3.407 (3)	163
C6—H6···O1	0.93	2.29	2.938 (3)	126
C14—H14C···Cg3^{ii}	0.96	2.75	3.506 (2)	136

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

Fig. 1

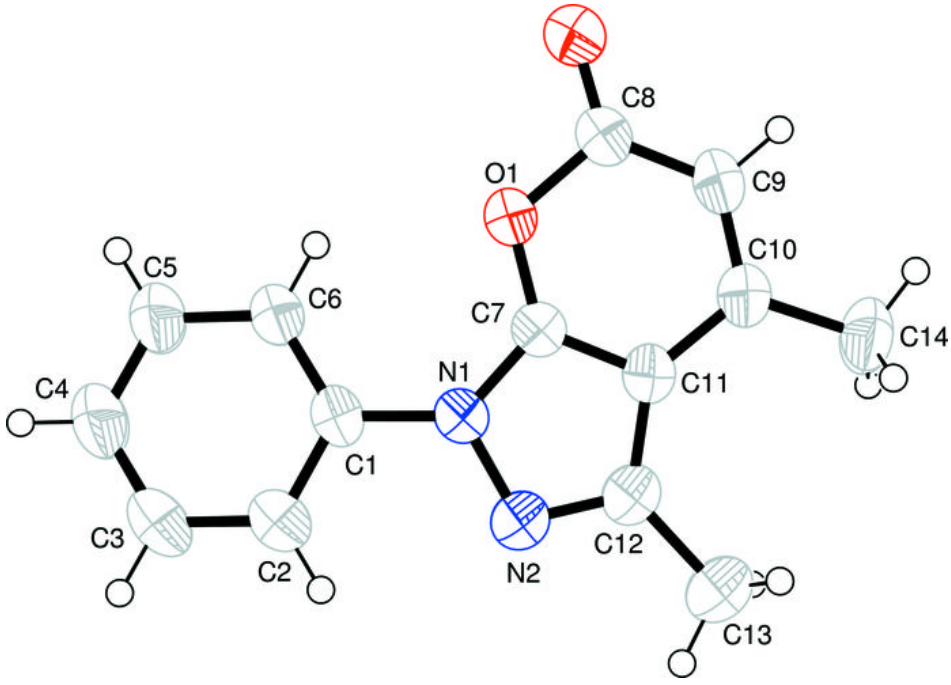

Fig. 2