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1,3,6-Trimethylpyrano[4,3-*b*]pyrrol-4(1*H*)-one

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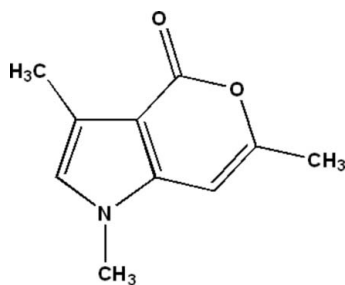
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 Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.051; wR factor = 0.179; data-to-parameter ratio = 13.9.

 All the non-H atoms of the title compound, $\text{C}_{10}\text{H}_{11}\text{NO}_2$, are almost coplanar [maximum deviation = 0.040 (3) Å]. The crystal structure is stabilized by C—H \cdots O hydrogen bonds.

Related literature

 For general background to isocoumarins, see: Barry (1964). For related structures, see: Abid *et al.* (2006, 2008); Hathwar *et al.* (2007).


Experimental

Crystal data

 $\text{C}_{10}\text{H}_{11}\text{NO}_2$
 $M_r = 177.20$
 Monoclinic, $P2_1/n$
 $a = 7.5556$ (7) Å
 $b = 8.4819$ (8) Å

 $c = 14.3081$ (14) Å
 $\beta = 93.870$ (6)°
 $V = 914.86$ (15) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

 $\mu = 0.09$ mm⁻¹
 $T = 295$ K

 $0.33 \times 0.28 \times 0.15$ mm

Data collection

 Oxford Xcalibur Eos (Nova) CCD detector diffractometer
 Absorption correction: multi-scan (*CrysAlis PRO RED*; Oxford Diffraction, 2009)
 $T_{\min} = 0.916$, $T_{\max} = 0.987$

 7291 measured reflections
 1692 independent reflections
 1176 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.179$
 $S = 1.18$
 1692 reflections

 122 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.27$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.18$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C10—H10B \cdots O1 ⁱ	0.96	2.46	3.404 (3)	170

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

 Data collection: *CrysAlis PRO CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO CCD*; data reduction: *CrysAlis PRO RED* (Oxford Diffraction, 2009); 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 *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1997).

We thank the Department of Science and Technology, India, for use of the CCD facility set up under the IRHPA–DST program at IISc. We thank Professor T. N. Guru Row, IISc, Bangalore, for useful crystallographic discussions. FNK thanks the DST for Fast Track Proposal funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5154).

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supporting information

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1,3,6-Trimethylpyrano[4,3-*b*]pyrrol-4(1*H*)-one

V. Krishnakumar, F. Nawaz Khan, Venkatesha R. Hathwar, P. Nithya and S. Suresh

S1. Comment

Isocoumarins (Barry, 1964) are also useful intermediates in the synthesis of a variety of important compounds including some carbocyclic and heterocyclic compounds. In view of their natural occurrence, biological activities and utility as synthetic intermediates, we have synthesized the title compound, and reported herein its crystal structure.

S2. Experimental

A mixture of 2-(carboxymethyl)-1, 4-dimethyl-1*H*-pyrrole-3-carboxylic acid (2 mmol) and acetic anhydride (8 mmol) in the presence of pyridine was refluxed for 4 h. Completion of the reaction was monitored by Thin Layer Chromatography. After completing of the reaction, the mixture was poured into crushed ice. The solids were separated and purified by silica gel column chromatography. The product was obtained with 90% yield.

S3. Refinement

All the H atoms were positioned geometrically and refined using a riding model, fixing the bond lengths at 0.96 and 0.93 Å for CH₃ aromatic CH, respectively. The displacement parameters of the H atoms were constrained as $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ ($1.5U_{\text{eq}}$ for methyl) of the carrier atom.

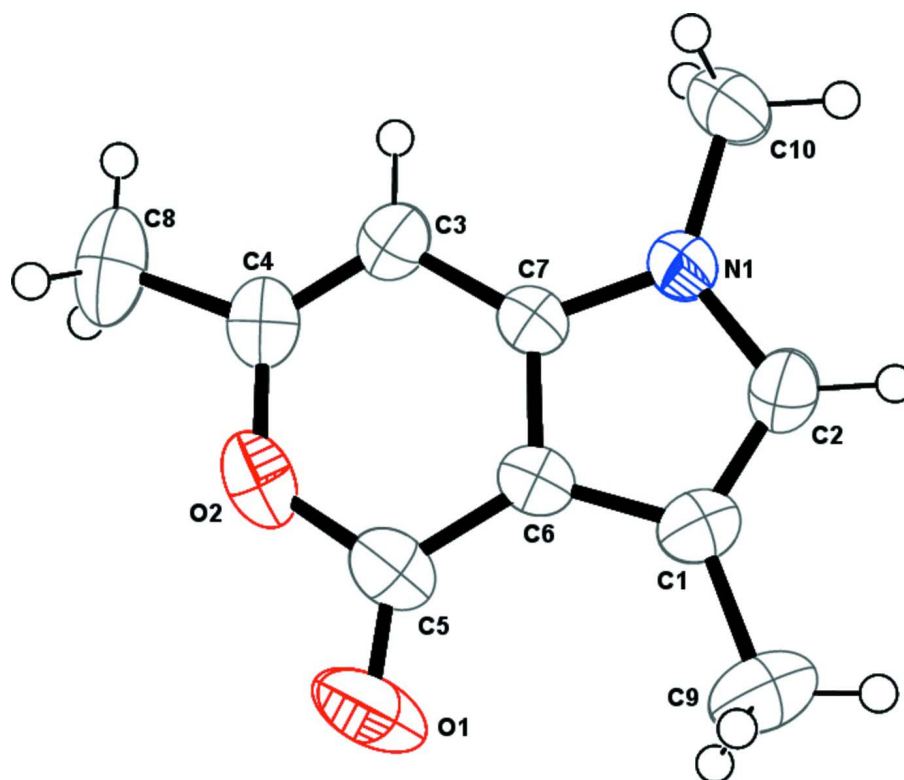


Figure 1

A view of the title complex, showing 50% probability displacement ellipsoids and the atom-numbering scheme.

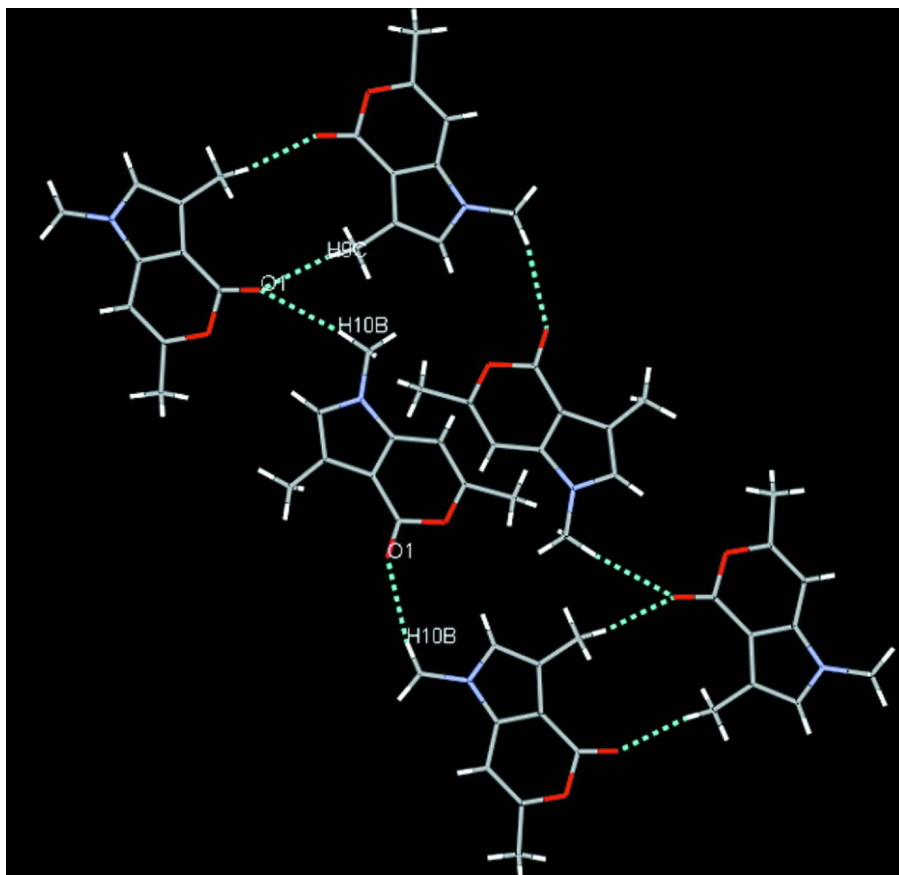


Figure 2

The packing diagram depicting C—H...O intermolecular interactions.

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Crystal data

$C_{10}H_{11}NO_2$

$M_r = 177.20$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 7.5556$ (7) Å

$b = 8.4819$ (8) Å

$c = 14.3081$ (14) Å

$\beta = 93.870$ (6)°

$V = 914.86$ (15) Å³

$Z = 4$

$F(000) = 376$

$D_x = 1.287$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1235 reflections

$\theta = 2.9$ – 20.4 °

$\mu = 0.09$ mm⁻¹

$T = 295$ K

Block, colorless

$0.33 \times 0.28 \times 0.15$ mm

Data collection

Oxford Xcalibur Eos (Nova) CCD detector
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

ω scans

Absorption correction: multi-scan

(*CrysAlis PRO RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.916$, $T_{\max} = 0.987$

7291 measured reflections

1692 independent reflections

1176 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 25.5$ °, $\theta_{\min} = 2.8$ °

$h = -9 \rightarrow 9$

$k = -9 \rightarrow 10$

$l = -17 \rightarrow 17$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.051$ $wR(F^2) = 0.179$ $S = 1.18$

1692 reflections

122 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0888P)^2 + 0.144P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$ Extinction correction: *SHELXL97* (Sheldrick,
2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.004 (1)

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.1293 (2)	0.4172 (2)	0.87406 (12)	0.0412 (5)
O1	0.3720 (3)	0.1280 (3)	1.12304 (13)	0.0818 (7)
O2	0.3792 (2)	0.3881 (2)	1.13262 (10)	0.0586 (6)
C1	0.1778 (3)	0.1674 (3)	0.92158 (16)	0.0466 (6)
C2	0.1079 (3)	0.2591 (3)	0.85100 (16)	0.0466 (6)
H2	0.0534	0.2216	0.7951	0.056*
C3	0.2612 (3)	0.5620 (3)	1.01528 (16)	0.0460 (6)
H3	0.2372	0.6633	0.9929	0.055*
C4	0.3421 (3)	0.5382 (3)	1.09968 (17)	0.0507 (7)
C5	0.3331 (3)	0.2497 (3)	1.08310 (16)	0.0526 (7)
C6	0.2454 (3)	0.2744 (3)	0.99253 (14)	0.0420 (6)
C7	0.2128 (3)	0.4269 (2)	0.96087 (14)	0.0389 (6)
C8	0.4000 (4)	0.6605 (4)	1.1693 (2)	0.0749 (9)
H8A	0.3742	0.7631	1.1435	0.112*
H8B	0.5253	0.6511	1.1842	0.112*
H8C	0.3380	0.6465	1.2251	0.112*
C9	0.1882 (4)	-0.0092 (3)	0.9237 (2)	0.0702 (9)
H9A	0.1145	-0.0518	0.8725	0.105*
H9B	0.1480	-0.0472	0.9818	0.105*
H9C	0.3087	-0.0416	0.9181	0.105*
C10	0.0743 (4)	0.5500 (3)	0.81496 (17)	0.0551 (7)
H10A	0.0008	0.6192	0.8487	0.083*
H10B	0.0085	0.5123	0.7597	0.083*
H10C	0.1771	0.6063	0.7974	0.083*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0466 (11)	0.0379 (11)	0.0382 (10)	0.0025 (8)	-0.0049 (8)	0.0018 (8)
O1	0.1038 (17)	0.0781 (16)	0.0623 (12)	0.0317 (12)	-0.0034 (11)	0.0266 (11)
O2	0.0562 (11)	0.0777 (14)	0.0403 (9)	0.0065 (9)	-0.0082 (7)	-0.0015 (9)
C1	0.0506 (13)	0.0375 (13)	0.0521 (14)	-0.0005 (11)	0.0074 (11)	-0.0007 (11)

C2	0.0488 (13)	0.0463 (14)	0.0442 (12)	-0.0021 (11)	-0.0008 (10)	-0.0089 (12)
C3	0.0474 (13)	0.0415 (14)	0.0489 (13)	-0.0035 (10)	0.0016 (10)	-0.0050 (10)
C4	0.0443 (13)	0.0604 (17)	0.0471 (13)	-0.0021 (12)	0.0015 (10)	-0.0097 (11)
C5	0.0541 (15)	0.0589 (17)	0.0448 (13)	0.0102 (13)	0.0022 (11)	0.0066 (13)
C6	0.0432 (12)	0.0416 (13)	0.0409 (12)	0.0030 (10)	0.0017 (9)	0.0050 (10)
C7	0.0392 (12)	0.0400 (13)	0.0372 (11)	0.0023 (10)	0.0005 (9)	0.0033 (9)
C8	0.0654 (18)	0.096 (2)	0.0630 (17)	-0.0162 (17)	-0.0004 (13)	-0.0329 (16)
C9	0.085 (2)	0.0395 (16)	0.087 (2)	0.0000 (15)	0.0159 (16)	-0.0025 (15)
C10	0.0634 (16)	0.0556 (17)	0.0450 (13)	0.0061 (13)	-0.0055 (11)	0.0116 (11)

Geometric parameters (Å, °)

N1—C7	1.357 (3)	C4—C8	1.483 (4)
N1—C2	1.388 (3)	C5—C6	1.431 (3)
N1—C10	1.452 (3)	C6—C7	1.387 (3)
O1—C5	1.206 (3)	C8—H8A	0.9600
O2—C4	1.380 (3)	C8—H8B	0.9600
O2—C5	1.403 (3)	C8—H8C	0.9600
C1—C2	1.353 (3)	C9—H9A	0.9600
C1—C6	1.430 (3)	C9—H9B	0.9600
C1—C9	1.500 (4)	C9—H9C	0.9600
C2—H2	0.9300	C10—H10A	0.9600
C3—C4	1.332 (3)	C10—H10B	0.9600
C3—C7	1.419 (3)	C10—H10C	0.9600
C3—H3	0.9300		
C7—N1—C2	108.41 (19)	N1—C7—C6	107.66 (19)
C7—N1—C10	125.64 (19)	N1—C7—C3	129.6 (2)
C2—N1—C10	125.94 (19)	C6—C7—C3	122.7 (2)
C4—O2—C5	124.21 (19)	C4—C8—H8A	109.5
C2—C1—C6	105.5 (2)	C4—C8—H8B	109.5
C2—C1—C9	127.3 (2)	H8A—C8—H8B	109.5
C6—C1—C9	127.2 (2)	C4—C8—H8C	109.5
C1—C2—N1	110.2 (2)	H8A—C8—H8C	109.5
C1—C2—H2	124.9	H8B—C8—H8C	109.5
N1—C2—H2	124.9	C1—C9—H9A	109.5
C4—C3—C7	117.4 (2)	C1—C9—H9B	109.5
C4—C3—H3	121.3	H9A—C9—H9B	109.5
C7—C3—H3	121.3	C1—C9—H9C	109.5
C3—C4—O2	121.3 (2)	H9A—C9—H9C	109.5
C3—C4—C8	126.8 (3)	H9B—C9—H9C	109.5
O2—C4—C8	111.9 (2)	N1—C10—H10A	109.5
O1—C5—O2	115.6 (2)	N1—C10—H10B	109.5
O1—C5—C6	129.6 (3)	H10A—C10—H10B	109.5
O2—C5—C6	114.8 (2)	N1—C10—H10C	109.5
C7—C6—C1	108.3 (2)	H10A—C10—H10C	109.5
C7—C6—C5	119.6 (2)	H10B—C10—H10C	109.5
C1—C6—C5	132.2 (2)		

C6—C1—C2—N1	0.3 (2)	O1—C5—C6—C7	179.4 (2)
C9—C1—C2—N1	-177.7 (2)	O2—C5—C6—C7	0.0 (3)
C7—N1—C2—C1	-0.5 (2)	O1—C5—C6—C1	-0.4 (4)
C10—N1—C2—C1	178.7 (2)	O2—C5—C6—C1	-179.8 (2)
C7—C3—C4—O2	0.6 (3)	C2—N1—C7—C6	0.4 (2)
C7—C3—C4—C8	-178.2 (2)	C10—N1—C7—C6	-178.74 (19)
C5—O2—C4—C3	-1.5 (3)	C2—N1—C7—C3	-178.8 (2)
C5—O2—C4—C8	177.5 (2)	C10—N1—C7—C3	2.1 (4)
C4—O2—C5—O1	-178.4 (2)	C1—C6—C7—N1	-0.2 (2)
C4—O2—C5—C6	1.1 (3)	C5—C6—C7—N1	179.96 (18)
C2—C1—C6—C7	-0.1 (2)	C1—C6—C7—C3	179.06 (19)
C9—C1—C6—C7	177.9 (2)	C5—C6—C7—C3	-0.8 (3)
C2—C1—C6—C5	179.7 (2)	C4—C3—C7—N1	179.6 (2)
C9—C1—C6—C5	-2.2 (4)	C4—C3—C7—C6	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>
C10—H10B...O1 ⁱ	0.96	2.46	3.404 (3)	170

Symmetry code: (i) $x-1/2, -y+1/2, z-1/2$.