Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

V. Krishnakumar,[a] F. Nawaz Khan,[a]* Venkatesha R. Hathwar,[b] P. Nithya[a] and S. Suresh[c]

[a]School of Advanced Sciences, VIT University, Vellore 632 014, Tamil Nadu, India, [b]Solid State and Structural Chemistry Unit, Indian Institute of Science, Bangalore 560012, Karnataka, India, and [c]Department of Chemistry, Aarupadai Veedu Institute of Technology, Vinayaka Missions University, Paiyanoor, Chennai 603 104, Tamil Nadu, India
Correspondence e-mail: nawaz_f@yahoo.co.in

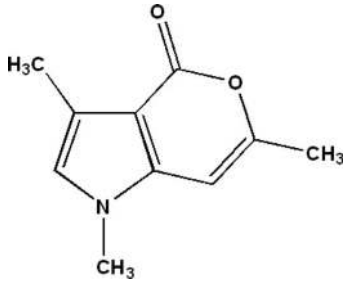
Received 18 December 2009; accepted 28 December 2009

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–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, $C_{10}H_{11}NO_2$, are almost coplanar [maximum deviation = 0.040 (3) Å]. The crystal structure is stabilized by C—H···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

$C_{10}H_{11}NO_2$
M_r = 177.20
Monoclinic, $P2_1/n$
a = 7.5556 (7) Å
b = 8.4819 (8) Å
c = 14.3081 (14) Å
β = 93.870 (6)°
V = 914.86 (15) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 K
0.33 × 0.28 × 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_{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
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10B···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).

References

Abid, O.-U.-R., Qadeer, G., Rama, N. H., Ruzicka, A. & Padelkova, Z. (2008). *Acta Cryst.* E**64**, o2018.
Abid, O., Rama, N. H., Qadeer, G., Khan, G. S. & Lu, X.-M. (2006). *Acta Cryst.* E**62**, o2895–o2896.
Barry, R. D. (1964). *Chem. Rev.* **64**, 229–260.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Hathwar, V. R., Manivel, P., Nawaz Khan, F. & Guru Row, T. N. (2007). *Acta Cryst.* E**63**, o3707.
Oxford Diffraction (2009). *CrysAlis PRO CCD* and *CrysAlis PRO RED*. Oxford Diffraction Ltd, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Watkin, D. J., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.

supporting information

Acta Cryst. (2010). E**66**, o381 [https://doi.org/10.1107/S1600536809055627]

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 CH_3 aromatic CH, respectively. The displacement parameters of the H atoms were constrained as U_{iso}(H) = 1.2U_{eq} (1.5U_{eq} for methyl) of the carrier atom.

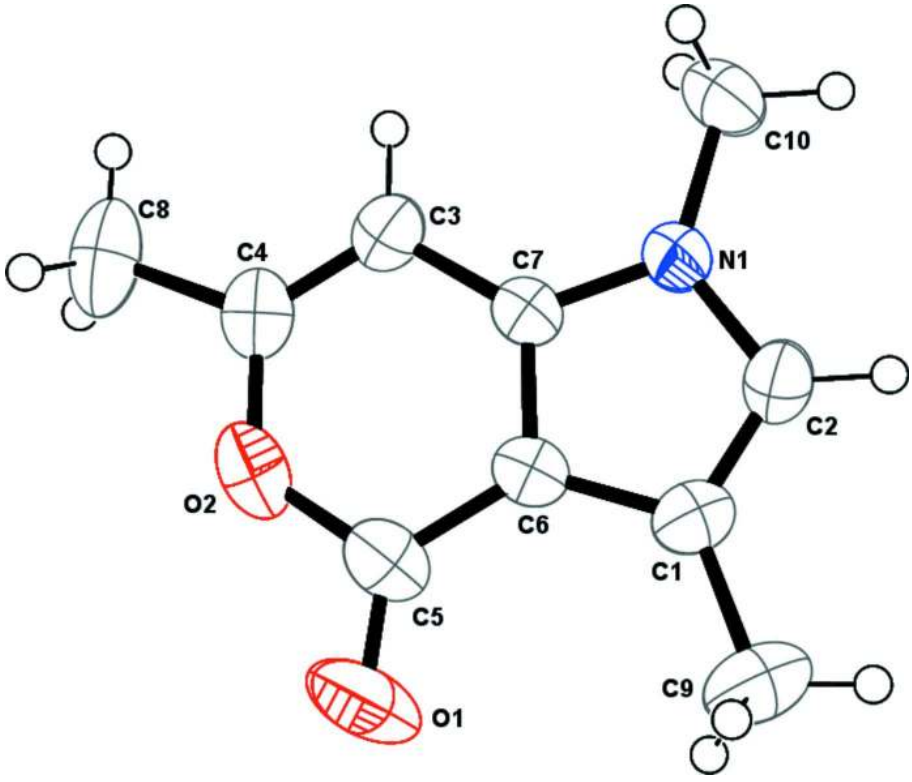


Figure 1

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

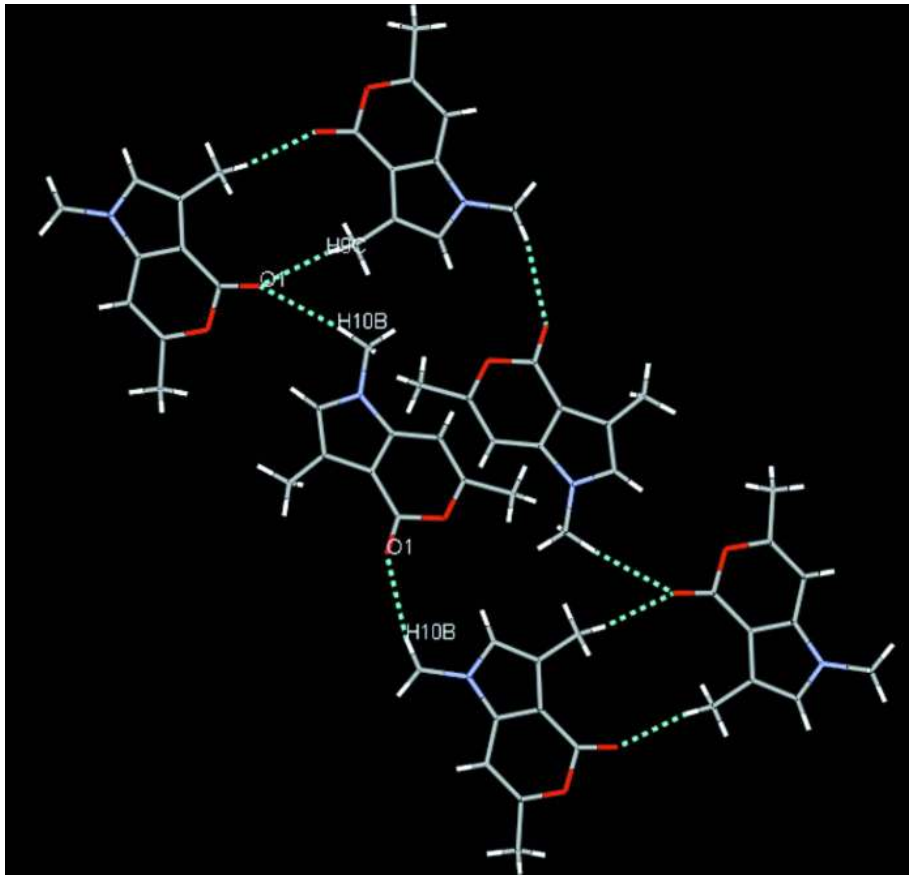


Figure 2

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

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

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
$\beta = 93.870\ (6)°$
$V = 914.86\ (15)$ Å^3
$Z = 4$
$F(000) = 376$
$D_x = 1.287$ 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 × 0.28 × 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 $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.8°$
$h = -9\rightarrow9$
$k = -9\rightarrow10$
$l = -17\rightarrow17$

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 methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0888P)^2 + 0.144P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.004 (1)

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

	x	*y*	*z*	U_{iso}*/U_{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 (Å^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, º)

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, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*B*···O1^{i}	0.96	2.46	3.404 (3)	170

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