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Crystal structure of 2,2-dimethyl-*N*-(pyridin-3-yl)propanamide

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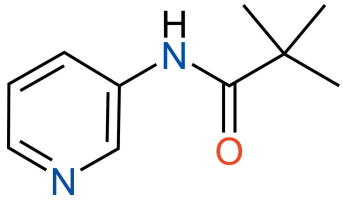
In the title compound, $C_{10}H_{14}N_2O$, the pyridine ring is inclined to the mean plane of the amide moiety [N—C(=O)C] by 17.60 (8)°. There is an intramolecular C—H···O hydrogen bond present involving the carbonyl O atom. In the crystal, molecules are linked *via* N—H···N hydrogen bonds, forming chains propagating along [100]. The *tert*-butyl group is disordered over two sets of sites with a refined occupancy ratio of 0.758 (12):0.242 (12).

Keywords: crystal structure; pyridine; propanamide; N—H···N hydrogen bonds.

CCDC reference: 1054113

1. Related literature

For related biologically active pyridine derivatives, see: de Candia *et al.* (2013); Thorat *et al.* (2013); Abdel-Megeed *et al.* (2012). For pyridine ring-system modifications, see: El-Hiti *et al.* (2015); Smith *et al.* (2012, 2013); Londregan *et al.* (2009); Joule & Mills (2000); Turner (1983). For the crystal structures of related compounds, see: El-Hiti *et al.* (2014); Seidler *et al.* (2011); Koch *et al.* (2008); Mazik *et al.* (2004).



2. Experimental

2.1. Crystal data

$C_{10}H_{14}N_2O$
M_r = 178.23
Orthorhombic, *Pbca*
a = 11.2453 (3) Å
b = 10.5272 (3) Å
c = 17.5339 (6) Å
V = 2075.69 (11) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 0.60 mm^{-1}
T = 293 K
0.23 × 0.19 × 0.06 mm

2.2. Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.840, T_{max} = 1.000
7164 measured reflections
2065 independent reflections
1722 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.127
S = 1.05
2065 reflections
153 parameters
114 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1	0.93	2.25	2.8263 (18)	119
N1—H1···N2^{i}	0.86	2.17	3.0012 (15)	164

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

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5094).

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supporting information

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Crystal structure of 2,2-dimethyl-*N*-(pyridin-3-yl)propanamide

Gamal A. El-Hiti, Keith Smith, Amany S. Hegazy, Saud A. Alanazi and Benson M. Kariuki

S1. Introduction

Pyridine derivatives are interesting compounds (Joule & Mills, 2000) since they show a range of biological activities (Thorat *et al.*, 2013) such as anticoagulant (de Candia *et al.*, 2013) and antimicrobial (Abdel-Megeed *et al.*, 2012) properties. Various simple and efficient processes have been developed for modification of the pyridine ring system (El-Hiti *et al.*, 2015; Smith *et al.*, 2013, Smith *et al.*, 2012, Londregan *et al.*, 2009; Turner, 1983). The X-ray crystal structures of related compounds have been reported (El-Hiti *et al.*, 2014; Seidler *et al.*, 2011; Koch *et al.*, 2008; Mazik *et al.*, 2004).

S2. Experimental

The title compound was obtained in 73% yield from the reaction of 3-aminopyridine with pivaloyl chloride in the presence of triethylamine in dichloromethane at 273 K for 15 min and then at room temperature for 2 h (Turner, 1983). Crystallization from a mixture of ethyl acetate and hexane gave colourless crystals of the title compound. The spectroscopic and analytical data for the title compound were identical with those reported previously (Turner, 1983)

S2.1. Refinement

The N- and C-bound H atoms were included in calculated positions and refined as riding: N—H = 0.86 Å, C—H = 0.93 - 0.98 Å with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and = 1.2U_{eq}(C) for other H atoms. The *t*-butyl group is disordered over two sites and was refined with bond length constraints to give a refined occupancy ratio of 0.758 (12):0.242 (12).

S3. Results and discussion

The molecular structure of the title compound is illustrated in Fig. 1. The pyridine ring is inclined to the mean plane of the amide moiety [N1—C6(═O1)—C7] by 17.60 (8) °. There is an intramolecular C—H···O hydrogen bond present involving the carbonyl O atom (Table 1).

In the crystal, molecules are linked via N—H···N hydrogen bonds forming chains propagating along [100]; see Table 1 and Fig. 2.

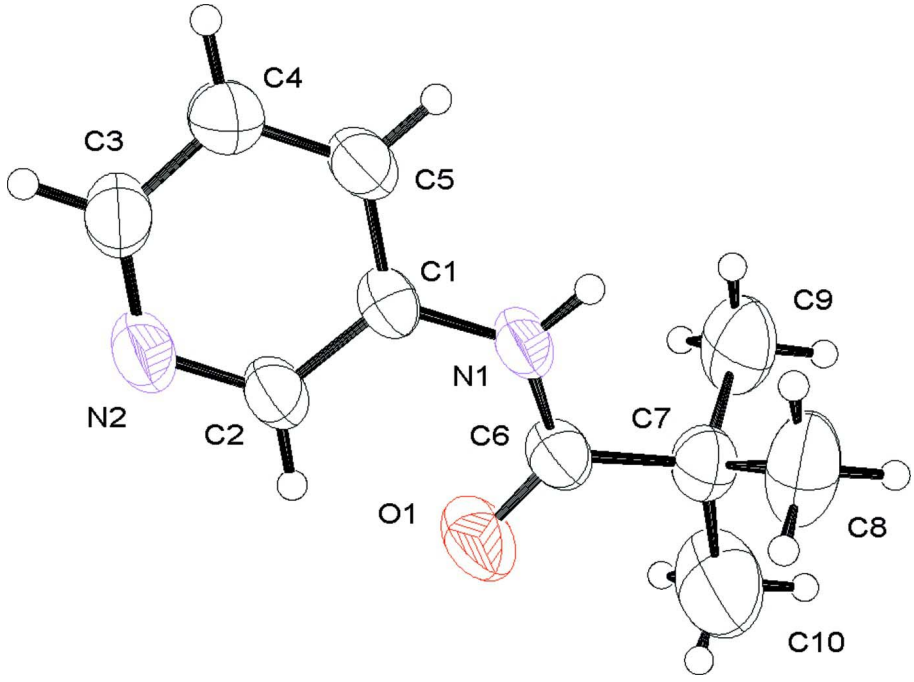

Figure 1

The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Only the major component of the disordered *t*-butyl group is shown.

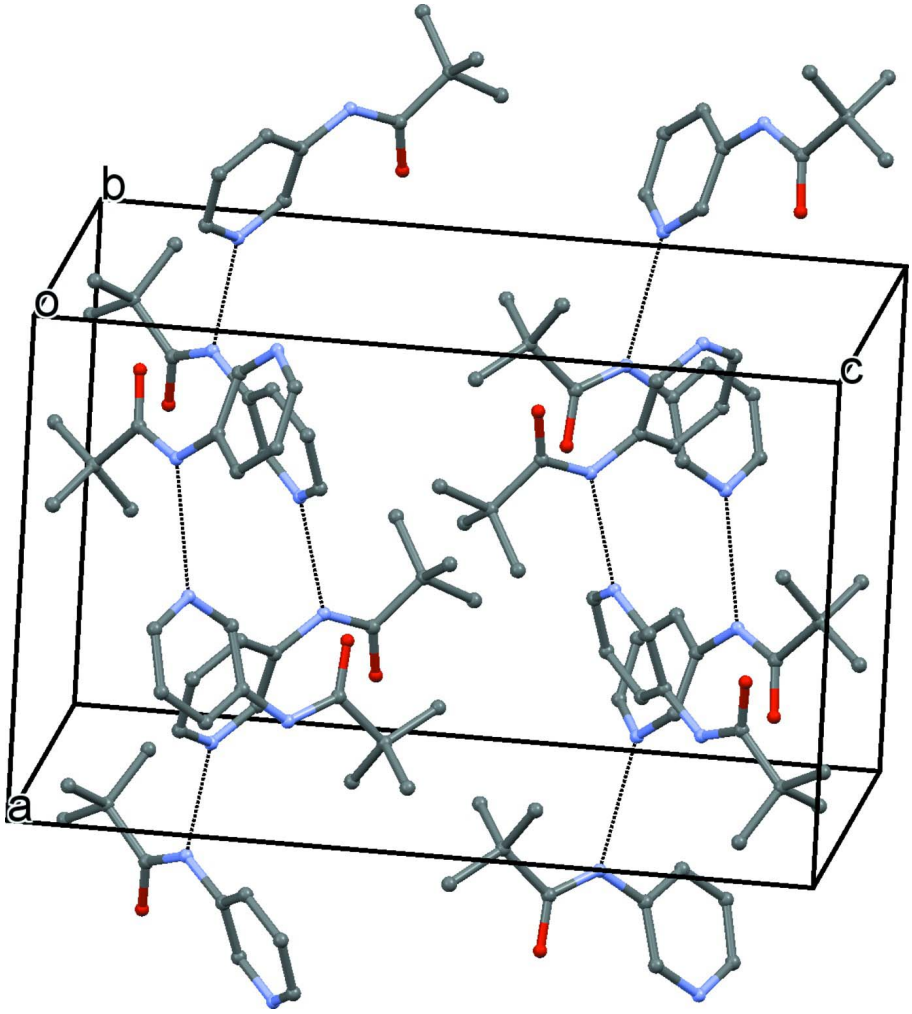

Figure 2

Crystal packing of the title compound, viewed along the *b* axis, with the N—H···N interactions shown as dashed lines (see Table 1 for details). The minor component of the disordered *t*-butyl group has been omitted for clarity.

2,2-Dimethyl-*N*-(pyridin-3-yl)propanamide

Crystal data

$C_{10}H_{14}N_2O$
$M_r = 178.23$
Orthorhombic, *Pbca*
$a = 11.2453\ (3)$ Å
$b = 10.5272\ (3)$ Å
$c = 17.5339\ (6)$ Å
$V = 2075.69\ (11)$ Å^3
$Z = 8$
$F(000) = 768$
$D_x = 1.141$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 3061 reflections
$\theta = 5.0–73.4°$
$\mu = 0.60$ mm^{-1}
$T = 293$ K
Plate, colourless
0.23 × 0.19 × 0.06 mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas) diffractometer
Radiation source: sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.840, T_{max} = 1.000
7164 measured reflections
2065 independent reflections
1722 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 73.8°, θ_{min} = 5.1°
h = −14→8
k = −13→12
l = −20→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.127
S = 1.05
2065 reflections
153 parameters
114 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0688P)^2 + 0.2387P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL2013* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0016 (4)

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.72804 (10)	0.10403 (12)	0.27358 (7)	0.0490 (3)	
C2	0.61380 (11)	0.14128 (14)	0.25328 (8)	0.0585 (4)	
H2	0.5822	0.2140	0.2756	0.070*	
C3	0.59204 (13)	−0.02662 (15)	0.17123 (8)	0.0663 (4)	
H3	0.5452	−0.0722	0.1371	0.080*	
C4	0.70473 (13)	−0.06915 (16)	0.18680 (9)	0.0702 (4)	
H4	0.7344	−0.1414	0.1630	0.084*	
C5	0.77323 (12)	−0.00301 (14)	0.23832 (8)	0.0619 (4)	
H5	0.8499	−0.0303	0.2494	0.074*	
C6	0.76123 (12)	0.24951 (14)	0.38234 (8)	0.0585 (3)	
C7	0.85629 (13)	0.29549 (15)	0.43884 (9)	0.0671 (4)	
C8	0.9594 (3)	0.3605 (4)	0.3935 (2)	0.0798 (10)	0.758 (12)
H8A	0.9277	0.4278	0.3627	0.120*	0.758 (12)
H8B	0.9974	0.2988	0.3613	0.120*	0.758 (12)
H8C	1.0165	0.3947	0.4287	0.120*	0.758 (12)
C9	0.9077 (5)	0.1835 (4)	0.4813 (4)	0.0838 (11)	0.758 (12)
H9A	0.9680	0.2125	0.5159	0.126*	0.758 (12)
H9B	0.9421	0.1250	0.4455	0.126*	0.758 (12)
H9C	0.8458	0.1417	0.5094	0.126*	0.758 (12)

C10	0.8023 (4)	0.3941 (7)	0.4916 (4)	0.1205 (18)	0.758 (12)
H10A	0.7365	0.3574	0.5186	0.181*	0.758 (12)
H10B	0.7751	0.4653	0.4621	0.181*	0.758 (12)
H10C	0.8613	0.4222	0.5274	0.181*	0.758 (12)
C8A	0.9208 (16)	0.4067 (14)	0.4095 (8)	0.108 (4)	0.242 (12)
H8D	0.9534	0.3873	0.3602	0.162*	0.242 (12)
H8E	0.9841	0.4281	0.4439	0.162*	0.242 (12)
H8F	0.8672	0.4772	0.4052	0.162*	0.242 (12)
C9A	0.9385 (16)	0.1880 (15)	0.4658 (11)	0.089 (4)	0.242 (12)
H9D	0.8920	0.1137	0.4766	0.133*	0.242 (12)
H9E	0.9796	0.2142	0.5111	0.133*	0.242 (12)
H9F	0.9953	0.1690	0.4265	0.133*	0.242 (12)
C10A	0.7832 (11)	0.3342 (17)	0.5131 (6)	0.098 (4)	0.242 (12)
H10D	0.7211	0.3924	0.4993	0.147*	0.242 (12)
H10E	0.8355	0.3739	0.5491	0.147*	0.242 (12)
H10F	0.7488	0.2595	0.5355	0.147*	0.242 (12)
N1	0.79882 (8)	0.16796 (11)	0.32735 (6)	0.0543 (3)	
H1	0.8741	0.1539	0.3252	0.065*	
N2	0.54740 (9)	0.07752 (13)	0.20320 (7)	0.0650 (3)	
O1	0.65818 (9)	0.28385 (14)	0.38687 (7)	0.0885 (4)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0369 (6)	0.0624 (7)	0.0478 (6)	0.0007 (5)	0.0006 (5)	0.0031 (5)
C2	0.0400 (6)	0.0715 (8)	0.0640 (7)	0.0064 (6)	−0.0046 (5)	−0.0063 (6)
C3	0.0529 (7)	0.0841 (9)	0.0617 (8)	−0.0037 (7)	−0.0065 (6)	−0.0111 (7)
C4	0.0630 (9)	0.0778 (9)	0.0697 (9)	0.0116 (7)	−0.0067 (7)	−0.0172 (7)
C5	0.0450 (7)	0.0766 (8)	0.0641 (8)	0.0130 (6)	−0.0051 (6)	−0.0078 (6)
C6	0.0461 (7)	0.0715 (8)	0.0579 (7)	0.0057 (6)	−0.0020 (6)	−0.0039 (6)
C7	0.0620 (8)	0.0739 (8)	0.0653 (8)	−0.0024 (7)	−0.0092 (6)	−0.0103 (7)
C8	0.0716 (17)	0.0753 (17)	0.0926 (19)	−0.0182 (13)	−0.0135 (13)	0.0017 (14)
C9	0.089 (3)	0.101 (2)	0.062 (2)	−0.0202 (16)	−0.0235 (18)	0.0146 (16)
C10	0.098 (2)	0.134 (4)	0.129 (4)	−0.002 (3)	−0.004 (2)	−0.073 (3)
C8A	0.129 (8)	0.107 (7)	0.087 (6)	−0.026 (6)	−0.027 (6)	0.000 (6)
C9A	0.080 (7)	0.116 (7)	0.071 (7)	0.008 (6)	−0.023 (5)	−0.011 (5)
C10A	0.102 (6)	0.115 (8)	0.076 (5)	0.013 (6)	−0.026 (4)	−0.046 (5)
N1	0.0341 (5)	0.0708 (7)	0.0581 (6)	0.0041 (4)	−0.0035 (4)	−0.0056 (5)
N2	0.0408 (6)	0.0867 (8)	0.0676 (7)	0.0032 (5)	−0.0078 (5)	−0.0079 (6)
O1	0.0543 (6)	0.1273 (10)	0.0838 (8)	0.0235 (6)	−0.0048 (5)	−0.0356 (7)

Geometric parameters (Å, º)

C1—C5	1.3820 (19)	C8—H8A	0.9600
C1—C2	1.3895 (17)	C8—H8B	0.9600
C1—N1	1.4054 (16)	C8—H8C	0.9600
C2—N2	1.3339 (18)	C9—H9A	0.9600
C2—H2	0.9300	C9—H9B	0.9600

C3—N2	1.3297 (19)	C9—H9C	0.9600
C3—C4	1.372 (2)	C10—H10A	0.9600
C3—H3	0.9300	C10—H10B	0.9600
C4—C5	1.376 (2)	C10—H10C	0.9600
C4—H4	0.9300	C8A—H8D	0.9600
C5—H5	0.9300	C8A—H8E	0.9600
C6—O1	1.2165 (17)	C8A—H8F	0.9600
C6—N1	1.3585 (17)	C9A—H9D	0.9600
C6—C7	1.5356 (19)	C9A—H9E	0.9600
C7—C8A	1.470 (7)	C9A—H9F	0.9600
C7—C9	1.510 (4)	C10A—H10D	0.9600
C7—C10	1.517 (4)	C10A—H10E	0.9600
C7—C9A	1.536 (8)	C10A—H10F	0.9600
C7—C8	1.563 (3)	N1—H1	0.8600
C7—C10A	1.593 (7)		

C5—C1—C2	117.09 (12)	H8B—C8—H8C	109.5
C5—C1—N1	118.83 (10)	C7—C9—H9A	109.5
C2—C1—N1	124.07 (11)	C7—C9—H9B	109.5
N2—C2—C1	122.97 (12)	H9A—C9—H9B	109.5
N2—C2—H2	118.5	C7—C9—H9C	109.5
C1—C2—H2	118.5	H9A—C9—H9C	109.5
N2—C3—C4	122.28 (13)	H9B—C9—H9C	109.5
N2—C3—H3	118.9	C7—C10—H10A	109.5
C4—C3—H3	118.9	C7—C10—H10B	109.5
C3—C4—C5	118.86 (14)	H10A—C10—H10B	109.5
C3—C4—H4	120.6	C7—C10—H10C	109.5
C5—C4—H4	120.6	H10A—C10—H10C	109.5
C4—C5—C1	120.02 (12)	H10B—C10—H10C	109.5
C4—C5—H5	120.0	C7—C8A—H8D	109.5
C1—C5—H5	120.0	C7—C8A—H8E	109.5
O1—C6—N1	122.04 (13)	H8D—C8A—H8E	109.5
O1—C6—C7	121.83 (13)	C7—C8A—H8F	109.5
N1—C6—C7	116.13 (11)	H8D—C8A—H8F	109.5
C9—C7—C10	112.8 (3)	H8E—C8A—H8F	109.5
C8A—C7—C6	111.6 (5)	C7—C9A—H9D	109.5
C9—C7—C6	109.8 (3)	C7—C9A—H9E	109.5
C10—C7—C6	109.3 (2)	H9D—C9A—H9E	109.5
C8A—C7—C9A	113.4 (7)	C7—C9A—H9F	109.5
C6—C7—C9A	112.7 (9)	H9D—C9A—H9F	109.5
C9—C7—C8	107.9 (2)	H9E—C9A—H9F	109.5
C10—C7—C8	107.9 (2)	C7—C10A—H10D	109.5
C6—C7—C8	109.05 (17)	C7—C10A—H10E	109.5
C8A—C7—C10A	109.7 (5)	H10D—C10A—H10E	109.5
C6—C7—C10A	104.4 (5)	C7—C10A—H10F	109.5
C9A—C7—C10A	104.3 (6)	H10D—C10A—H10F	109.5
C7—C8—H8A	109.5	H10E—C10A—H10F	109.5
C7—C8—H8B	109.5	C6—N1—C1	127.05 (10)

H8A—C8—H8B	109.5	C6—N1—H1	116.5
C7—C8—H8C	109.5	C1—N1—H1	116.5
H8A—C8—H8C	109.5	C3—N2—C2	118.76 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1	0.93	2.25	2.8263 (18)	119
N1—H1···N2^{i}	0.86	2.17	3.0012 (15)	164

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