(3,5-Dimethyl-1*H*-pyrrol-2-yl)(phenyl)methanone

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[a]School of Chemistry and Chemical Engineering, Qufu Normal University, Shandong, People's Republic of China, and [b]Department of Chemistry, Taiyuan Normal University, Taiyuan, 030031, People's Republic of China. *Correspondence e-mail: ruitaozhu@126.com

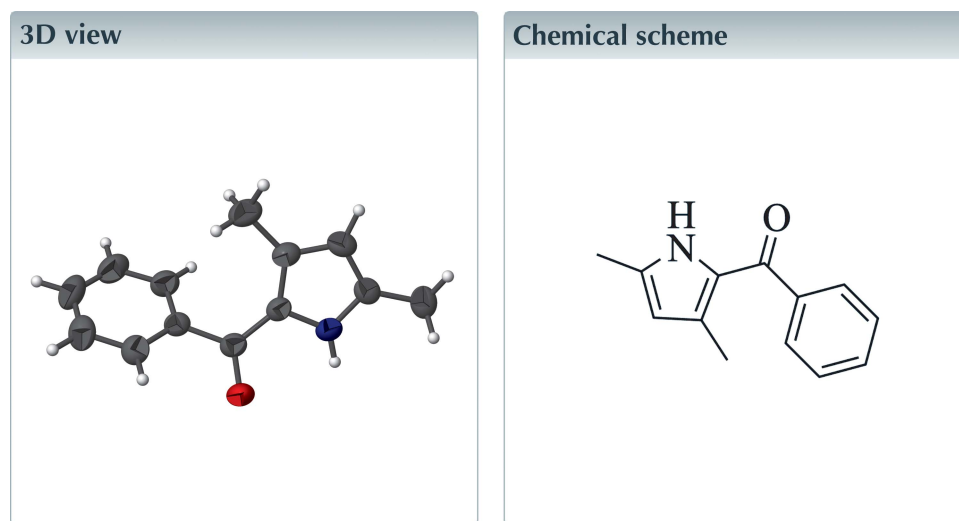In the title molecule, $C_{13}H_{13}NO$, the dihedral angle between phenyl and pyrrole rings is 57.2 (1)°. In the crystal, N—H⋯O hydrogen bonds link the molecules, forming chains propagating along the *b* axis.

3D view


Chemical scheme

Structure description

Pyrrole compounds are important units of many biologically active natural products and pharmaceutical compounds. Their transition metal-mediated synthesis (Gulevich *et al.*, 2013) and complexation behaviour with ruthenium has been reported (Lundrigan *et al.*, 2012).

The title molecule is shown in Fig. 1. The dihedral angle between the phenyl ring (C8–C13) and the pyrrole ring (N1/C1–C4) is 57.2 (1)°. In the crystal, molecules are linked by N—H⋯O hydrogen bonds (Table 1), forming chains propagating along the *b* axis (Fig. 2).

Synthesis and crystallization

The title compound was synthesized according to a literature method (Guo *et al.*, 2015). All of the reactions were carried out under a purified nitrogen atmosphere using the standard Schlenk techniques. Diethyl ether was distilled from sodium benzophenone under nitrogen. Hexane was dried using sodium potassium alloy and distilled under nitrogen prior to use. All commercial reagents were sublimed, recrystallized or distilled before use. To a solution of 3,5-dimethylpyrrole (0.31 ml, 3.0 mmol) in dry diethyl ether (20 ml), *n*-butyllithium (2.5 *M* in hexane, 1.2 mL, 3.0 mmol) was added at 273 K; the reaction mixture was then allowed to warm to room temperature and then stirred for 2 h under a nitrogen atmosphere. To this suspension, 2,6-dimethylaniline (0.18 ml, 1.5 mmol) was added dropwise and stirred for 30 min followed by the addition of benzaldehyde (0.61 ml, 6 mmol). Stirring was continued for 5 h at 303 K and the progress of the reaction
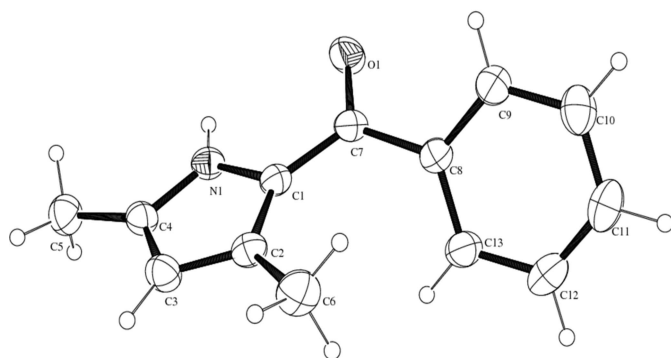
Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.
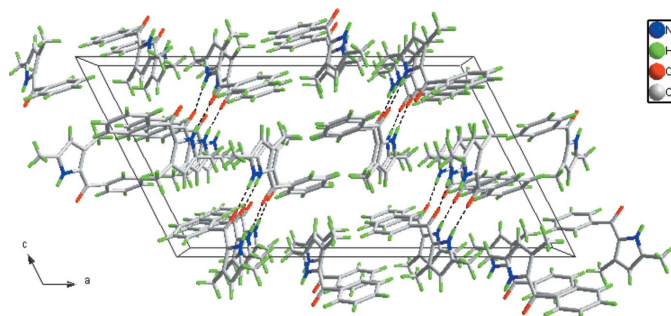
Figure 2
Part of the crystal structure, viewed along the b axis, with hydrogen bonds drawn as dashed lines.

was monitored by TLC. The reaction mixture was then cooled to room temperature, quenched with saturated aqueous ammonium chloride solution, filtered over Celite^R and extracted into ethyl acetate. The organic layer was then washed with brine, dried over anhydrous sodium sulfate and concentrated under reduced pressure to get the crude mixture. The product was isolated from the crude mixture by column chromatography on silica gel using an ethyl acetate hexane mixture (1:7) as an eluent and characterized by spectroscopic methods. Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the title compound in an ethyl acetate–hexane mixture (1:7) at room temperature for one week.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
N1—H1⋯O1^i	0.86	2.08	2.898 (2)	160

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{13}NO$
M_r	199.24
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	296
a, b, c (Å)	25.755 (7), 6.5962 (16), 14.503 (4)
β (°)	116.935 (5)
V (Å^3)	2196.5 (10)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	$0.30 \times 0.23 \times 0.20$
Data collection	
Diffractometer	Bruker *SMART APEX* CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2007)
T_{min}, T_{max}	0.977, 0.985
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5885, 1941, 1254
R_{int}	0.039
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.596
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.117, 1.01
No. of reflections	1941
No. of parameters	138
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	0.16, −0.20

Computer programs: *APEX2* and *SAINT* (Bruker, 2007), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *DIAMOND* (Brandenburg, 2006) and *publCIF* (Westrip, 2010).

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full crystallographic data

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Crystal data

$C_{13}H_{13}NO$	$F(000) = 848$
$M_r = 199.24$	$D_x = 1.205$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 898 reflections
$a = 25.755$ (7) Å	$\theta = 2.8–21.5°$
$b = 6.5962$ (16) Å	$\mu = 0.08$ mm^{-1}
$c = 14.503$ (4) Å	$T = 296$ K
$\beta = 116.935$ (5)°	Block, colourless
$V = 2196.5$ (10) Å^3	$0.30 \times 0.23 \times 0.20$ mm
$Z = 8$	

Data collection

Bruker SMART APEX CCD diffractometer	5885 measured reflections
Radiation source: fine-focus sealed tube	1941 independent reflections
Graphite monochromator	1254 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.039$
Absorption correction: multi-scan	$\theta_{max} = 25.0°$, $\theta_{min} = 2.8°$
(*SADABS*; Bruker, 2007)	$h = -30\rightarrow30$
$T_{min} = 0.977$, $T_{max} = 0.985$	$k = -7\rightarrow4$
	$l = -13\rightarrow17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.048$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.117$	
$S = 1.01$	H-atom parameters constrained
1941 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 1.514P]$
138 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.16$ e Å^{-3}
	$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
N1	0.19605 (7)	0.2779 (3)	0.08665 (13)	0.0425 (5)
H1	0.2241	0.2467	0.1458	0.051*
O1	0.21736 (6)	0.5700 (2)	0.23331 (12)	0.0638 (5)
C1	0.16027 (8)	0.4441 (3)	0.06853 (16)	0.0398 (5)
C2	0.12220 (9)	0.4446 (3)	−0.03716 (17)	0.0455 (6)
C3	0.13576 (9)	0.2726 (4)	−0.07836 (18)	0.0520 (6)
H3	0.1170	0.2332	−0.1474	0.062*
C4	0.18111 (9)	0.1708 (3)	−0.00092 (18)	0.0450 (5)
C5	0.21193 (10)	−0.0191 (3)	−0.0016 (2)	0.0593 (7)
H5A	0.2525	−0.0062	0.0457	0.089*
H5B	0.2075	−0.0432	−0.0701	0.089*
H5C	0.1957	−0.1306	0.0191	0.089*
C6	0.07803 (10)	0.6011 (4)	−0.10058 (19)	0.0641 (7)
H6A	0.0411	0.5693	−0.1027	0.096*
H6B	0.0741	0.6016	−0.1696	0.096*
H6C	0.0906	0.7324	−0.0700	0.096*
C7	0.17080 (9)	0.5785 (3)	0.15294 (17)	0.0431 (5)
C8	0.12592 (9)	0.7268 (3)	0.14765 (16)	0.0418 (5)
C9	0.14329 (10)	0.9203 (3)	0.18634 (18)	0.0503 (6)
H9	0.1822	0.9577	0.2120	0.060*
C10	0.10339 (12)	1.0572 (4)	0.1870 (2)	0.0623 (7)
H10	0.1151	1.1880	0.2114	0.075*
C11	0.04640 (12)	1.0020 (4)	0.1520 (2)	0.0692 (8)
H11	0.0196	1.0950	0.1533	0.083*
C12	0.02863 (11)	0.8103 (5)	0.1150 (2)	0.0677 (8)
H12	−0.0101	0.7730	0.0922	0.081*
C13	0.06793 (9)	0.6722 (4)	0.11150 (18)	0.0545 (6)
H13	0.0556	0.5430	0.0850	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0375 (10)	0.0438 (11)	0.0401 (10)	0.0007 (8)	0.0121 (8)	0.0039 (9)
O1	0.0453 (9)	0.0663 (11)	0.0542 (11)	0.0107 (8)	0.0001 (8)	−0.0147 (9)
C1	0.0338 (11)	0.0390 (12)	0.0437 (13)	0.0005 (10)	0.0150 (10)	0.0026 (10)
C2	0.0381 (12)	0.0516 (15)	0.0429 (13)	0.0001 (11)	0.0149 (10)	0.0048 (11)
C3	0.0523 (14)	0.0583 (16)	0.0410 (13)	−0.0033 (12)	0.0172 (12)	−0.0034 (11)
C4	0.0459 (13)	0.0429 (13)	0.0480 (14)	−0.0067 (11)	0.0228 (11)	−0.0037 (11)
C5	0.0643 (15)	0.0483 (15)	0.0712 (18)	−0.0002 (12)	0.0359 (14)	−0.0049 (12)
C6	0.0580 (15)	0.0708 (18)	0.0513 (16)	0.0122 (13)	0.0140 (13)	0.0138 (13)

C7	0.0371 (12)	0.0414 (13)	0.0454 (13)	−0.0029 (10)	0.0140 (11)	0.0013 (10)
C8	0.0399 (12)	0.0457 (13)	0.0382 (12)	0.0016 (10)	0.0162 (10)	0.0044 (10)
C9	0.0525 (14)	0.0441 (14)	0.0572 (15)	0.0000 (12)	0.0273 (12)	0.0049 (11)
C10	0.0782 (18)	0.0439 (15)	0.0730 (18)	0.0106 (14)	0.0413 (15)	0.0079 (13)
C11	0.0667 (18)	0.0670 (19)	0.080 (2)	0.0265 (15)	0.0386 (16)	0.0149 (15)
C12	0.0432 (14)	0.084 (2)	0.0740 (19)	0.0088 (14)	0.0246 (14)	0.0075 (16)
C13	0.0431 (13)	0.0582 (16)	0.0572 (16)	−0.0015 (12)	0.0184 (12)	−0.0003 (12)

Geometric parameters (Å, º)

N1—C4	1.348 (3)	C6—H6B	0.9600
N1—C1	1.379 (3)	C6—H6C	0.9600
N1—H1	0.8600	C7—C8	1.490 (3)
O1—C7	1.238 (2)	C8—C13	1.388 (3)
C1—C2	1.396 (3)	C8—C9	1.384 (3)
C1—C7	1.434 (3)	C9—C10	1.371 (3)
C2—C3	1.399 (3)	C9—H9	0.9300
C2—C6	1.502 (3)	C10—C11	1.367 (3)
C3—C4	1.374 (3)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.369 (4)
C4—C5	1.486 (3)	C11—H11	0.9300
C5—H5A	0.9600	C12—C13	1.380 (3)
C5—H5B	0.9600	C12—H12	0.9300
C5—H5C	0.9600	C13—H13	0.9300
C6—H6A	0.9600		
C4—N1—C1	110.87 (17)	C2—C6—H6C	109.5
C4—N1—H1	124.6	H6A—C6—H6C	109.5
C1—N1—H1	124.6	H6B—C6—H6C	109.5
N1—C1—C2	106.72 (19)	O1—C7—C1	120.2 (2)
N1—C1—C7	118.50 (18)	O1—C7—C8	118.4 (2)
C2—C1—C7	134.6 (2)	C1—C7—C8	121.32 (18)
C1—C2—C3	106.25 (19)	C13—C8—C9	119.2 (2)
C1—C2—C6	129.4 (2)	C13—C8—C7	121.7 (2)
C3—C2—C6	124.2 (2)	C9—C8—C7	118.95 (19)
C4—C3—C2	109.4 (2)	C10—C9—C8	120.3 (2)
C4—C3—H3	125.3	C10—C9—H9	119.9
C2—C3—H3	125.3	C8—C9—H9	119.9
N1—C4—C3	106.8 (2)	C11—C10—C9	120.3 (3)
N1—C4—C5	121.5 (2)	C11—C10—H10	119.9
C3—C4—C5	131.7 (2)	C9—C10—H10	119.9
C4—C5—H5A	109.5	C10—C11—C12	120.2 (2)
C4—C5—H5B	109.5	C10—C11—H11	119.9
H5A—C5—H5B	109.5	C12—C11—H11	119.9
C4—C5—H5C	109.5	C11—C12—C13	120.3 (2)
H5A—C5—H5C	109.5	C11—C12—H12	119.9
H5B—C5—H5C	109.5	C13—C12—H12	119.9
C2—C6—H6A	109.5	C8—C13—C12	119.8 (2)

| C2—C6—H6B | 109.5 | C8—C13—H13 | 120.1 |
| H6A—C6—H6B | 109.5 | C12—C13—H13 | 120.1 |

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1[i]	0.86	2.08	2.898 (2)	160

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