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1-(1-Benzyl-2,5-dimethyl-4-phenyl-1*H*-pyrrol-3-yl)-ethanone

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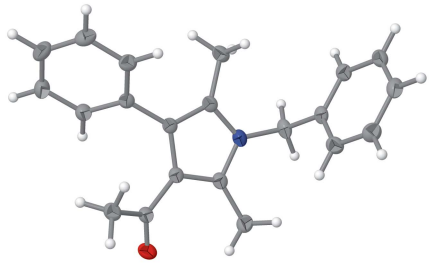
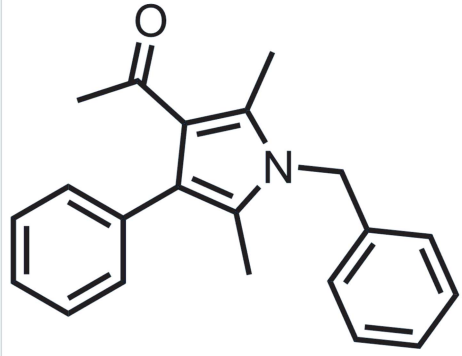
In the title compound, $C_{21}H_{21}NO$, the dihedral angles between the planes of the phenyl and pyrrole rings are 47.04 (5) and 79.27 (3)°. In the crystal, centrosymmetrically related molecules are linked into dimers by pairs of C—H···O hydrogen bonds, forming rings of graph-set motif $R_2^2(16)$.

3D view



Chemical scheme



Structure description

Pyrrole derivatives, which are widespread in nature (Iwao *et al.*, 2003), are of interest with respect to their versatility in organic synthetic procedures (Loudet & Burgess, 2007) and their biological and medicinal activities (Fan *et al.*, 2008).

The molecular structure of 1-(1-benzyl-2,5-dimethyl-4-phenyl-1*H*-pyrrol-3-yl)ethanone is shown in Fig. 1. The dihedral angles between the planes of the pyrrole ring and the C8–C13 and C16–C21 phenyl rings are 47.04 (5) and 79.27 (3)°, respectively. The molecular conformation is enforced by an intramolecular hydrogen bond involving a methyl H atom and the carbonyl O atom (Table 1). In the crystal, molecules are linked through pairs of C—H···O hydrogen bonds to form $R_2^2(16)$ centrosymmetric dimers.

Synthesis and crystallization

The synthesis of the title compound was carried out by mixing acetylacetone (1.1 mmol), benzylamine (1.0 mmol), benzaldehyde (1.0 mmol) and nitroethane (1.3 mmol) in the presence of $Ca_5(PO_4)_3OH$ (0.05 mmol) as a catalyst. The mixture was stirred at 333 K for 24 h. After extraction with ethyl acetate (3 × 25 ml), the organic layer was dried with Na_2SO_4 and the solvent was removed under reduced pressure. The product was obtained in 74% yield after silica-gel column chromatography using a mixture of *n*-hexane and

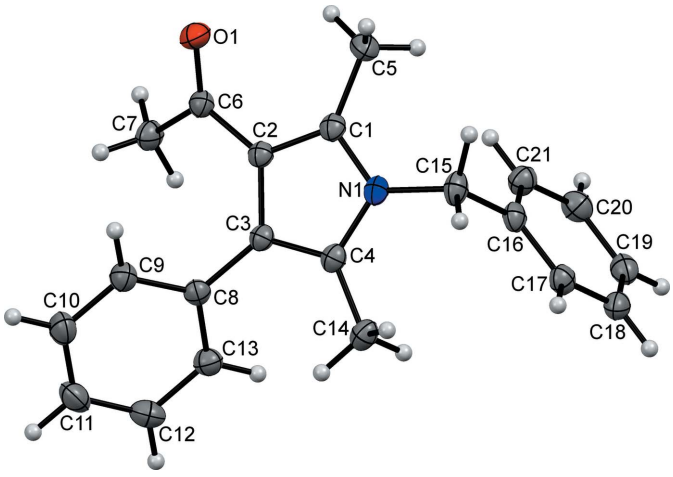


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

ethyl acetate (94:6 *v*/*v*) as eluent. White crystals were obtained by slow evaporation of the solvent at room temperature (m.p. 364–365 K). ^{1}H NMR (DMSO): δ 1.77 (*s*, 3H), 1.94 (*s*, 3H), 2.36 (*s*, 3H), 5.19 (*s*, 2H), 6.98–7.38 (*m*, 10H). ^{13}C NMR (DMSO): δ 9.99, 11.47, 30.64, 46.25, 120.79, 121.42, 125.73, 126.41, 127.16, 128.12, 128.77, 130.29, 133.52, 136.72, 137.33, 195.04.

Refinement

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

References

Bruker (2013). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2014). *APEX2* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fan, H., Peng, J., Hamann, M. T. & Hu, J. F. (2008). *Chem. Rev.* **108**, 264–287.
Iwao, M., Takeuchi, T., Fujikawa, N., Fukuda, T. & Ishibashi, F. (2003). *Tetrahedron Lett.* **44**, 4443–4446.
Loudet, A. & Burgess, K. (2007). *Chem. Rev.* **107**, 4891–4932.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5B···O1	0.98	2.38	3.0007 (17)	121
C10—H10···O1^{i}	0.95	2.50	3.4114 (17)	161

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{21}NO$
M_r	303.39
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	150
a, b, c (Å)	10.7210 (2), 13.7039 (2), 11.5761 (2)
β (°)	107.0322 (6)
V (Å^3)	1626.16 (5)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.42 × 0.33 × 0.27
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.93, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	31011, 3926, 3484
R_{int}	0.021
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.661
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.104, 1.04
No. of reflections	3926
No. of parameters	211
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.22

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2006) and *publCIF* (Westrip, 2010).

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

full crystallographic data

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1-(1-Benzyl-2,5-dimethyl-4-phenyl-1*H*-pyrrol-3-yl)ethanone

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1-(1-Benzyl-2,5-dimethyl-4-phenyl-1*H*-pyrrol-3-yl)ethanone

Crystal data

$C_{21}H_{21}NO$
M_r = 303.39
Monoclinic, $P2_1/c$
a = 10.7210 (2) Å
b = 13.7039 (2) Å
c = 11.5761 (2) Å
β = 107.0322 (6)°
V = 1626.16 (5) Å^3
Z = 4
$F(000)$ = 648
D_x = 1.239 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9842 reflections
θ = 2.5–30.5°
μ = 0.08 mm^{-1}
T = 150 K
Prism, colourless
0.42 × 0.33 × 0.27 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 8.3333 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.93, T_{max} = 0.98
31011 measured reflections
3926 independent reflections
3484 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 28.0°, θ_{min} = 2.0°
h = −14→14
k = −18→18
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.104
S = 1.04
3926 reflections
211 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.4997P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.69940 (10)	0.00933 (7)	0.28342 (9)	0.0226 (2)
C2	0.77764 (9)	0.04143 (7)	0.21445 (9)	0.0207 (2)
C3	0.88508 (9)	0.09425 (7)	0.29356 (9)	0.0203 (2)
C4	0.86945 (10)	0.09142 (7)	0.40726 (9)	0.0221 (2)
C5	0.57153 (11)	−0.04261 (8)	0.24566 (11)	0.0301 (2)
H5A	0.5842	−0.1110	0.2711	0.045*
H5B	0.5354	−0.0394	0.1576	0.045*
H5C	0.5110	−0.0115	0.2835	0.045*
C6	0.74045 (9)	0.03075 (7)	0.08261 (9)	0.0223 (2)
C7	0.77732 (11)	0.10944 (8)	0.00737 (9)	0.0271 (2)
H7A	0.8575	0.0906	−0.0112	0.041*
H7B	0.7916	0.1710	0.0525	0.041*
H7C	0.7068	0.1178	−0.0680	0.041*
C8	0.99949 (9)	0.13556 (7)	0.26504 (8)	0.0214 (2)
C9	1.06614 (10)	0.08215 (8)	0.19876 (9)	0.0265 (2)
H9	1.0369	0.0184	0.1715	0.032*
C10	1.17418 (11)	0.12067 (9)	0.17224 (11)	0.0333 (3)
H10	1.2177	0.0838	0.1263	0.040*
C11	1.21862 (11)	0.21310 (10)	0.21292 (11)	0.0353 (3)
H11	1.2924	0.2397	0.1945	0.042*
C12	1.15563 (11)	0.26646 (9)	0.28026 (11)	0.0322 (2)
H12	1.1871	0.3293	0.3094	0.039*
C13	1.04641 (10)	0.22844 (8)	0.30547 (10)	0.0260 (2)
H13	1.0029	0.2661	0.3508	0.031*
C14	0.95716 (11)	0.12583 (8)	0.52522 (9)	0.0271 (2)
H14A	1.0417	0.1445	0.5154	0.041*
H14B	0.9697	0.0733	0.5849	0.041*
H14C	0.9178	0.1823	0.5529	0.041*
C15	0.70566 (11)	0.02234 (8)	0.50245 (9)	0.0268 (2)
H15A	0.7778	−0.0018	0.5712	0.032*
H15B	0.6384	−0.0294	0.4805	0.032*
C16	0.64695 (10)	0.11232 (7)	0.54209 (9)	0.0231 (2)
C17	0.69601 (10)	0.14761 (8)	0.65922 (9)	0.0252 (2)
H17	0.7676	0.1155	0.7143	0.030*
C18	0.64164 (11)	0.22944 (8)	0.69680 (10)	0.0274 (2)
H18	0.6760	0.2528	0.7771	0.033*
C19	0.53749 (11)	0.27678 (8)	0.61709 (10)	0.0284 (2)
H19	0.5005	0.3329	0.6424	0.034*
C20	0.48743 (11)	0.24209 (8)	0.50021 (10)	0.0300 (2)
H20	0.4156	0.2742	0.4455	0.036*
C21	0.54191 (10)	0.16052 (8)	0.46281 (9)	0.0277 (2)
H21	0.5073	0.1373	0.3824	0.033*
N1	0.75568 (8)	0.04036 (6)	0.39956 (8)	0.02273 (19)
O1	0.67584 (8)	−0.03911 (6)	0.03179 (7)	0.03149 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0272 (5)	0.0185 (4)	0.0237 (5)	0.0001 (4)	0.0102 (4)	−0.0007 (4)
C2	0.0232 (5)	0.0183 (4)	0.0213 (5)	0.0004 (3)	0.0078 (4)	−0.0003 (3)
C3	0.0220 (4)	0.0186 (4)	0.0207 (4)	0.0017 (3)	0.0068 (4)	−0.0003 (3)
C4	0.0244 (5)	0.0208 (4)	0.0213 (5)	0.0031 (4)	0.0071 (4)	0.0004 (4)
C5	0.0317 (5)	0.0280 (5)	0.0343 (6)	−0.0083 (4)	0.0154 (4)	−0.0046 (4)
C6	0.0228 (4)	0.0230 (5)	0.0218 (5)	0.0008 (4)	0.0078 (4)	−0.0018 (4)
C7	0.0318 (5)	0.0291 (5)	0.0211 (5)	−0.0023 (4)	0.0088 (4)	0.0014 (4)
C8	0.0199 (4)	0.0237 (5)	0.0193 (4)	0.0011 (3)	0.0041 (3)	0.0015 (4)
C9	0.0257 (5)	0.0281 (5)	0.0264 (5)	0.0007 (4)	0.0086 (4)	−0.0028 (4)
C10	0.0268 (5)	0.0435 (6)	0.0326 (6)	0.0018 (5)	0.0136 (4)	−0.0035 (5)
C11	0.0236 (5)	0.0456 (7)	0.0378 (6)	−0.0066 (5)	0.0109 (5)	0.0019 (5)
C12	0.0271 (5)	0.0307 (6)	0.0360 (6)	−0.0067 (4)	0.0049 (4)	−0.0005 (4)
C13	0.0249 (5)	0.0252 (5)	0.0270 (5)	−0.0001 (4)	0.0062 (4)	−0.0023 (4)
C14	0.0294 (5)	0.0299 (5)	0.0207 (5)	0.0041 (4)	0.0052 (4)	−0.0015 (4)
C15	0.0365 (6)	0.0245 (5)	0.0240 (5)	0.0030 (4)	0.0162 (4)	0.0041 (4)
C16	0.0269 (5)	0.0232 (5)	0.0228 (5)	−0.0008 (4)	0.0129 (4)	0.0030 (4)
C17	0.0263 (5)	0.0282 (5)	0.0221 (5)	−0.0001 (4)	0.0089 (4)	0.0035 (4)
C18	0.0310 (5)	0.0290 (5)	0.0249 (5)	−0.0054 (4)	0.0124 (4)	−0.0035 (4)
C19	0.0296 (5)	0.0241 (5)	0.0358 (6)	−0.0012 (4)	0.0163 (4)	−0.0019 (4)
C20	0.0264 (5)	0.0305 (5)	0.0324 (5)	0.0026 (4)	0.0073 (4)	0.0031 (4)
C21	0.0300 (5)	0.0297 (5)	0.0227 (5)	−0.0008 (4)	0.0071 (4)	0.0001 (4)
N1	0.0277 (4)	0.0216 (4)	0.0212 (4)	0.0010 (3)	0.0109 (3)	0.0008 (3)
O1	0.0374 (4)	0.0303 (4)	0.0272 (4)	−0.0095 (3)	0.0100 (3)	−0.0083 (3)

Geometric parameters (Å, º)

C1—N1	1.3701 (13)	C11—C12	1.3808 (17)
C1—C2	1.3881 (13)	C11—H11	0.9500
C1—C5	1.4920 (14)	C12—C13	1.3885 (15)
C2—C3	1.4393 (13)	C12—H12	0.9500
C2—C6	1.4674 (13)	C13—H13	0.9500
C3—C4	1.3752 (13)	C14—H14A	0.9800
C3—C8	1.4735 (13)	C14—H14B	0.9800
C4—N1	1.3861 (13)	C14—H14C	0.9800
C4—C14	1.4896 (14)	C15—N1	1.4634 (12)
C5—H5A	0.9800	C15—C16	1.5154 (14)
C5—H5B	0.9800	C15—H15A	0.9900
C5—H5C	0.9800	C15—H15B	0.9900
C6—O1	1.2262 (12)	C16—C17	1.3897 (14)
C6—C7	1.5098 (14)	C16—C21	1.3934 (15)
C7—H7A	0.9800	C17—C18	1.3912 (15)
C7—H7B	0.9800	C17—H17	0.9500
C7—H7C	0.9800	C18—C19	1.3840 (16)
C8—C13	1.3980 (14)	C18—H18	0.9500
C8—C9	1.3987 (14)	C19—C20	1.3854 (16)

C9—C10	1.3866 (15)	C19—H19	0.9500
C9—H9	0.9500	C20—C21	1.3878 (16)
C10—C11	1.3864 (18)	C20—H20	0.9500
C10—H10	0.9500	C21—H21	0.9500
N1—C1—C2	107.42 (9)	C11—C12—C13	120.13 (10)
N1—C1—C5	122.57 (9)	C11—C12—H12	119.9
C2—C1—C5	129.85 (9)	C13—C12—H12	119.9
C1—C2—C3	107.41 (8)	C12—C13—C8	120.94 (10)
C1—C2—C6	122.73 (9)	C12—C13—H13	119.5
C3—C2—C6	129.42 (9)	C8—C13—H13	119.5
C4—C3—C2	107.19 (9)	C4—C14—H14A	109.5
C4—C3—C8	124.62 (9)	C4—C14—H14B	109.5
C2—C3—C8	127.90 (9)	H14A—C14—H14B	109.5
C3—C4—N1	107.71 (9)	C4—C14—H14C	109.5
C3—C4—C14	130.33 (10)	H14A—C14—H14C	109.5
N1—C4—C14	121.74 (9)	H14B—C14—H14C	109.5
C1—C5—H5A	109.5	N1—C15—C16	113.13 (8)
C1—C5—H5B	109.5	N1—C15—H15A	109.0
H5A—C5—H5B	109.5	C16—C15—H15A	109.0
C1—C5—H5C	109.5	N1—C15—H15B	109.0
H5A—C5—H5C	109.5	C16—C15—H15B	109.0
H5B—C5—H5C	109.5	H15A—C15—H15B	107.8
O1—C6—C2	121.34 (9)	C17—C16—C21	118.66 (10)
O1—C6—C7	119.17 (9)	C17—C16—C15	120.43 (9)
C2—C6—C7	119.44 (9)	C21—C16—C15	120.90 (9)
C6—C7—H7A	109.5	C16—C17—C18	120.80 (10)
C6—C7—H7B	109.5	C16—C17—H17	119.6
H7A—C7—H7B	109.5	C18—C17—H17	119.6
C6—C7—H7C	109.5	C19—C18—C17	119.97 (10)
H7A—C7—H7C	109.5	C19—C18—H18	120.0
H7B—C7—H7C	109.5	C17—C18—H18	120.0
C13—C8—C9	117.94 (9)	C18—C19—C20	119.76 (10)
C13—C8—C3	121.08 (9)	C18—C19—H19	120.1
C9—C8—C3	120.96 (9)	C20—C19—H19	120.1
C10—C9—C8	121.08 (10)	C19—C20—C21	120.20 (10)
C10—C9—H9	119.5	C19—C20—H20	119.9
C8—C9—H9	119.5	C21—C20—H20	119.9
C11—C10—C9	119.93 (11)	C20—C21—C16	120.61 (10)
C11—C10—H10	120.0	C20—C21—H21	119.7
C9—C10—H10	120.0	C16—C21—H21	119.7
C12—C11—C10	119.95 (10)	C1—N1—C4	110.26 (8)
C12—C11—H11	120.0	C1—N1—C15	125.82 (9)
C10—C11—H11	120.0	C4—N1—C15	123.93 (9)
N1—C1—C2—C3	−0.20 (11)	C10—C11—C12—C13	−1.16 (18)
C5—C1—C2—C3	175.30 (10)	C11—C12—C13—C8	0.93 (17)
N1—C1—C2—C6	−173.26 (9)	C9—C8—C13—C12	0.15 (15)

C5—C1—C2—C6	2.24 (17)	C3—C8—C13—C12	178.85 (10)
C1—C2—C3—C4	0.73 (11)	N1—C15—C16—C17	−121.33 (10)
C6—C2—C3—C4	173.16 (10)	N1—C15—C16—C21	59.56 (13)
C1—C2—C3—C8	174.70 (9)	C21—C16—C17—C18	−0.06 (15)
C6—C2—C3—C8	−12.87 (17)	C15—C16—C17—C18	−179.19 (9)
C2—C3—C4—N1	−0.96 (11)	C16—C17—C18—C19	−0.07 (16)
C8—C3—C4—N1	−175.18 (9)	C17—C18—C19—C20	0.30 (16)
C2—C3—C4—C14	173.69 (10)	C18—C19—C20—C21	−0.39 (16)
C8—C3—C4—C14	−0.53 (17)	C19—C20—C21—C16	0.26 (17)
C1—C2—C6—O1	−32.75 (15)	C17—C16—C21—C20	−0.03 (15)
C3—C2—C6—O1	155.84 (10)	C15—C16—C21—C20	179.09 (10)
C1—C2—C6—C7	144.56 (10)	C2—C1—N1—C4	−0.41 (11)
C3—C2—C6—C7	−26.86 (15)	C5—C1—N1—C4	−176.30 (9)
C4—C3—C8—C13	−49.76 (14)	C2—C1—N1—C15	−179.82 (9)
C2—C3—C8—C13	137.25 (10)	C5—C1—N1—C15	4.28 (15)
C4—C3—C8—C9	128.90 (11)	C3—C4—N1—C1	0.87 (11)
C2—C3—C8—C9	−44.10 (15)	C14—C4—N1—C1	−174.33 (9)
C13—C8—C9—C10	−1.01 (15)	C3—C4—N1—C15	−179.70 (9)
C3—C8—C9—C10	−179.71 (10)	C14—C4—N1—C15	5.09 (14)
C8—C9—C10—C11	0.79 (17)	C16—C15—N1—C1	−106.93 (11)
C9—C10—C11—C12	0.31 (18)	C16—C15—N1—C4	73.73 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5*B*···O1	0.98	2.38	3.0007 (17)	121
C10—H10···O1^{i}	0.95	2.50	3.4114 (17)	161

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