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1-Allyl-5-(2,5-dimethyl-1*H*-pyrrol-1-yl)-1*H*-indazole

Mohamed El Ghozlani,[a]* Adiba Rais,[a] El Mostapha Rakib,[a] Souad Mojahidi,[a] Mohamed Saadi[b] and Lahcen El Ammari[b]

[a]Laboratoire de Chimie Organique et Analytique, Université Sultan Moulay Slimane, Faculté des Sciences et Techniques, Béni-Mellal, BP 523, Morocco, and [b]Laboratoire de Chimie du Solide Appliquée, Faculty of Sciences, Mohammed V University in Rabat, Avenue Ibn Battouta, BP 1014, Rabat, Morocco. *Correspondence e-mail: m_elghozlani@yahoo.fr

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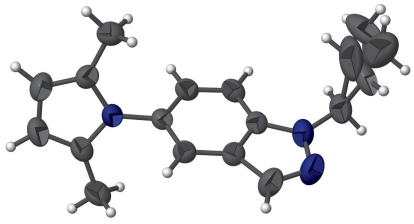
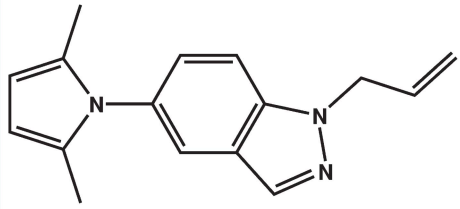
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{16}H_{17}N_3$, the indazole ring system makes a dihedral angle of 64.73 (12)° with the pyrrole ring. The atoms of the allyl group are disordered over two sets of sites, with a refined occupancy ratio of 0.70 (3):0.30 (3). The mean plane through the major component of the allyl group is nearly perpendicular to the indazole ring, as indicated by the N—C—C=C torsion angle of −114 (1)°. In the crystal, molecules are linked by C—H···π interactions, forming undulating sheets parallel to the *ab* plane.

3D view



Chemical scheme



Structure description

Indazole is recognized to be a highly effective pharmacophore in medicinal chemistry as well as being the core of important nitrogen-containing heterocycles that show a broad range of biological activities (Cerecetto *et al.*, 2005; Gaikwad *et al.*, 2015; Jennings & Tennant, 2007). Previously, our group has researched indazole derivatives with potential anticancer activity. Some of them exert pharmacologically interesting antiproliferative/apoptotic activity against human and murine cell lines (Bouissane *et al.*, 2006; Abbassi *et al.*, 2012, 2014).

The molecule of the title compound is built up from an indazole ring system (C1–C7/N2/N3) linked to an allyl group and to a 2,5-dimethyl-pyrrol-1-yl moiety, as shown in Fig. 1. The mean plane of the major component of the allyl group, which is disordered over two positions, is almost perpendicular to the indazole ring as indicated by the N3—C14—C15*A*=C16*A* torsion angle of −114 (1)°. The pyrrole ring makes a dihedral angle of 64.73 (12)° with the indazole ring system.

In the crystal, molecules are linked by C14—H14*B*···π and C16*A*—H16*B*···π interactions (Table 1), forming undulating sheets parallel to (001), as shown in Fig. 2.

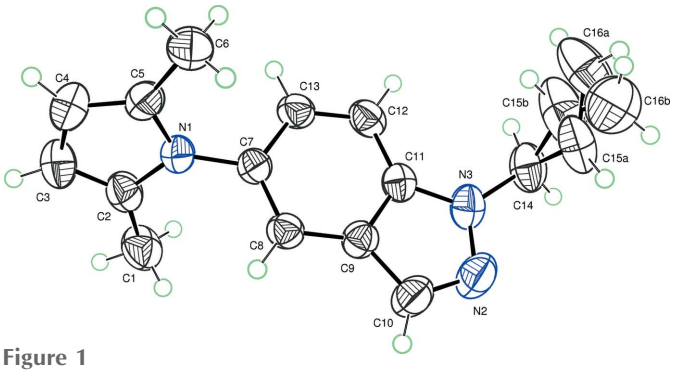


Figure 1
A view of the molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The atoms of the allyl group (C15*A*/16*A* and C15*B*/16*B*) are disordered over two sets of sites.

Synthesis and crystallization

1-Allyl-5-nitroindazole (1.0 mmol) was added to a mixture of anhydrous $SnCl_2$ powder (460 mg, 4.0 mmol), and acetic acid (0.572 ml, 10 mmol) in tetrahydrofuran (10 ml), followed by the addition of 2,5-hexadione (1.0 mmol) in THF (15 ml). The reaction mixture was stirred at 353 K for 4 h. After the reaction was completed, the mixture was diluted with ethyl acetate (30 ml), poured into 10% $NaHCO_3$ (30 ml), and then extracted with ethyl acetate (3 × 50 ml). The combined organic extracts were dried over $MgSO_4$, filtered and concentrated. The residue was purified by column chromatography on silica gel using ethyl acetate/hexane (3:7) to afford the title compound indazole in good yield. The title compound was recrystallized from ethyl ether at room temperature giving colourless crystals (m.p. 360 K, yield 68%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The atoms of the allyl group are disordered over two sets of sites, with a refined occupancy ratio of 0.70 (3):0.30 (3).

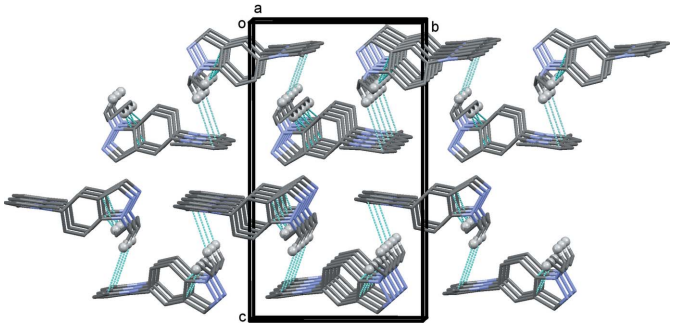


Figure 2
A view along the *a* axis of the crystal packing of the title compound, with the C—H···π interactions represented by dashed lines (see Table 1). For clarity, only the H atoms involved in these interactions (grey balls), and the atoms of the major component of the disordered allyl group, have been included.

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

*Cg*1 and *Cg*3 are the centroids of the N1/C2–C5 and C7–C13 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*B*···*Cg*1^{i}	0.97	2.92	3.860 (3)	162
C16*A*—H16*B*···*Cg*3^{ii}	0.93	2.98	3.783 (14)	145

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{17}N_3$
M_r	251.32
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	296
a, *b*, *c* (Å)	7.5140 (2), 10.3737 (3), 17.8484 (5)
V (Å^3)	1391.25 (7)
Z	4
Radiation type	Mo *K*α
μ (mm^{-1})	0.07
Crystal size (mm)	0.37 × 0.32 × 0.27
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.626, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23639, 3598, 2453
R_{int}	0.050
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.045, 0.113, 1.03
No. of reflections	3598
No. of parameters	193
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.12, −0.16

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *publCIF* (Westrip, 2010).

Acknowledgements

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full crystallographic data

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1-Allyl-5-(2,5-dimethyl-1*H*-pyrrol-1-yl)-1*H*-indazole

Crystal data

$C_{16}H_{17}N_3$
M_r = 251.32
Orthorhombic, $P2_12_12_1$
a = 7.5140 (2) Å
b = 10.3737 (3) Å
c = 17.8484 (5) Å
V = 1391.25 (7) Å^3
Z = 4
F(000) = 536
D_x = 1.200 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3598 reflections
θ = 2.3–28.7°
μ = 0.07 mm^{-1}
T = 296 K
Block, colourless
0.37 × 0.32 × 0.27 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.626, T_{max} = 0.746
23639 measured reflections
3598 independent reflections
2453 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 28.7°, θ_{min} = 2.3°
h = −10→10
k = −13→14
l = −22→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.113
S = 1.03
3598 reflections
193 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.1215P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 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}	Occ. (<1)
C1	0.0292 (4)	0.5717 (3)	0.39588 (19)	0.0704 (8)	
H1A	−0.0924	0.6002	0.3952	0.106*	
H1B	0.0483	0.5173	0.4386	0.106*	
H1C	0.0542	0.5242	0.3509	0.106*	
C2	0.1492 (3)	0.6855 (2)	0.40034 (14)	0.0526 (6)	
C3	0.1113 (4)	0.8125 (3)	0.41117 (16)	0.0642 (7)	
H3	−0.0015	0.8476	0.4177	0.077*	
C4	0.2731 (4)	0.8816 (3)	0.41073 (16)	0.0637 (7)	
H4	0.2849	0.9701	0.4173	0.076*	
C5	0.4079 (3)	0.7978 (2)	0.39917 (14)	0.0508 (6)	
C6	0.6036 (4)	0.8194 (3)	0.39599 (18)	0.0669 (8)	
H6A	0.6458	0.8019	0.3463	0.100*	
H6B	0.6618	0.7629	0.4309	0.100*	
H6C	0.6294	0.9073	0.4088	0.100*	
C7	0.4314 (3)	0.5599 (2)	0.38047 (12)	0.0425 (5)	
C8	0.4341 (3)	0.4660 (2)	0.43402 (12)	0.0450 (5)	
H8	0.3724	0.4760	0.4788	0.054*	
C9	0.5322 (3)	0.3540 (2)	0.41963 (12)	0.0462 (5)	
C10	0.5682 (5)	0.2378 (3)	0.45860 (15)	0.0653 (8)	
H10	0.5227	0.2180	0.5057	0.078*	
C11	0.6264 (3)	0.3416 (2)	0.35235 (12)	0.0438 (5)	
C12	0.6233 (3)	0.4369 (2)	0.29737 (13)	0.0503 (6)	
H12	0.6855	0.4278	0.2526	0.060*	
C13	0.5245 (3)	0.5446 (2)	0.31236 (12)	0.0487 (6)	
H13	0.5184	0.6096	0.2766	0.058*	
C14	0.8354 (4)	0.1698 (3)	0.30186 (16)	0.0643 (7)	
H14A	0.8083	0.0794	0.2938	0.077*	
H14B	0.8243	0.2139	0.2542	0.077*	
C15A	1.0165 (13)	0.1818 (11)	0.3286 (4)	0.070 (3)	0.70 (3)
H15A	1.0529	0.1368	0.3710	0.085*	0.70 (3)
C16A	1.1372 (15)	0.2616 (18)	0.2904 (7)	0.105 (4)	0.70 (3)
H16A	1.1006	0.3065	0.2480	0.126*	0.70 (3)
H16B	1.2536	0.2695	0.3074	0.126*	0.70 (3)
C15B	1.030 (6)	0.243 (5)	0.309 (3)	0.138 (19)	0.30 (3)
H15B	1.0336	0.3328	0.3071	0.166*	0.30 (3)
C16B	1.132 (8)	0.199 (3)	0.316 (2)	0.151 (17)	0.30 (3)
H16C	1.1299	0.1099	0.3183	0.181*	0.30 (3)
H16D	1.2390	0.2436	0.3199	0.181*	0.30 (3)
N1	0.3325 (3)	0.67630 (18)	0.39266 (10)	0.0462 (5)	
N2	0.6734 (4)	0.1615 (2)	0.42016 (13)	0.0692 (7)	
N3	0.7080 (3)	0.2236 (2)	0.35444 (12)	0.0563 (6)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0457 (16)	0.0734 (19)	0.092 (2)	−0.0031 (14)	0.0043 (15)	−0.0055 (16)
C2	0.0426 (13)	0.0607 (16)	0.0546 (14)	0.0065 (12)	0.0006 (11)	−0.0010 (12)
C3	0.0513 (16)	0.0661 (18)	0.0752 (17)	0.0171 (14)	−0.0004 (14)	−0.0065 (14)
C4	0.0691 (19)	0.0468 (14)	0.0752 (17)	0.0099 (14)	−0.0047 (16)	−0.0010 (12)
C5	0.0530 (15)	0.0449 (13)	0.0546 (14)	0.0015 (11)	−0.0004 (12)	0.0056 (11)
C6	0.0565 (16)	0.0588 (17)	0.0855 (19)	−0.0083 (14)	0.0088 (14)	0.0007 (15)
C7	0.0370 (12)	0.0438 (12)	0.0467 (12)	0.0006 (10)	0.0001 (10)	0.0013 (10)
C8	0.0476 (13)	0.0479 (13)	0.0396 (11)	−0.0006 (11)	0.0035 (10)	0.0000 (10)
C9	0.0496 (14)	0.0454 (13)	0.0437 (11)	0.0016 (11)	−0.0016 (10)	0.0002 (10)
C10	0.092 (2)	0.0473 (15)	0.0570 (14)	0.0094 (15)	0.0060 (15)	0.0055 (12)
C11	0.0381 (12)	0.0457 (13)	0.0475 (12)	0.0000 (11)	−0.0049 (9)	−0.0054 (10)
C12	0.0460 (14)	0.0629 (16)	0.0419 (12)	0.0034 (12)	0.0054 (10)	−0.0006 (11)
C13	0.0463 (14)	0.0561 (14)	0.0436 (11)	0.0024 (12)	0.0024 (10)	0.0095 (11)
C14	0.0605 (17)	0.0608 (16)	0.0715 (17)	0.0099 (14)	−0.0018 (14)	−0.0227 (14)
C15A	0.049 (4)	0.096 (6)	0.067 (3)	0.026 (4)	−0.006 (2)	−0.025 (3)
C16A	0.054 (5)	0.147 (11)	0.114 (6)	−0.022 (5)	0.020 (4)	−0.063 (6)
C15B	0.11 (3)	0.10 (2)	0.20 (4)	0.04 (2)	0.00 (3)	−0.07 (3)
C16B	0.20 (5)	0.078 (15)	0.17 (2)	−0.007 (19)	0.07 (3)	−0.008 (15)
N1	0.0425 (11)	0.0444 (11)	0.0516 (10)	0.0053 (9)	0.0010 (9)	0.0027 (9)
N2	0.0916 (18)	0.0497 (12)	0.0662 (13)	0.0146 (13)	−0.0013 (13)	0.0042 (11)
N3	0.0586 (14)	0.0523 (12)	0.0582 (13)	0.0122 (11)	−0.0016 (10)	−0.0087 (10)

Geometric parameters (Å, º)

C1—C2	1.488 (4)	C9—C10	1.418 (3)
C2—C3	1.361 (4)	C10—N2	1.312 (4)
C2—N1	1.387 (3)	C11—N3	1.370 (3)
C3—C4	1.411 (4)	C11—C12	1.393 (3)
C4—C5	1.350 (4)	C12—C13	1.368 (3)
C5—N1	1.387 (3)	C14—C15A	1.448 (9)
C5—C6	1.489 (4)	C14—N3	1.452 (3)
C7—C8	1.366 (3)	C14—C15B	1.65 (5)
C7—C13	1.412 (3)	C15A—C16A	1.40 (2)
C7—N1	1.434 (3)	C15B—C16B	0.90 (7)
C8—C9	1.399 (3)	N2—N3	1.364 (3)
C9—C11	1.400 (3)		
C3—C2—N1	106.7 (2)	N3—C11—C12	131.4 (2)
C3—C2—C1	130.4 (2)	N3—C11—C9	106.6 (2)
N1—C2—C1	122.8 (2)	C12—C11—C9	122.0 (2)
C2—C3—C4	108.1 (2)	C13—C12—C11	116.8 (2)
C5—C4—C3	108.7 (2)	C12—C13—C7	122.0 (2)
C4—C5—N1	106.9 (2)	C15A—C14—N3	111.9 (4)
C4—C5—C6	130.5 (2)	N3—C14—C15B	110.6 (12)
N1—C5—C6	122.5 (2)	C16A—C15A—C14	119.8 (10)

C8—C7—C13	121.0 (2)	C16B—C15B—C14	122 (7)
C8—C7—N1	120.16 (19)	C5—N1—C2	109.6 (2)
C13—C7—N1	118.8 (2)	C5—N1—C7	124.47 (19)
C7—C8—C9	118.2 (2)	C2—N1—C7	126.0 (2)
C8—C9—C11	120.0 (2)	C10—N2—N3	106.2 (2)
C8—C9—C10	135.7 (2)	N2—N3—C11	111.1 (2)
C11—C9—C10	104.2 (2)	N2—N3—C14	120.0 (2)
N2—C10—C9	111.8 (2)	C11—N3—C14	128.4 (2)

Hydrogen-bond geometry (Å, º)

Cg1 and Cg3 are the centroids of the N1/C2–C5 and C7–C13 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H14*B*···*Cg*1^{i}	0.97	2.92	3.860 (3)	162
C16*A*—H16*B*···*Cg*3^{ii}	0.93	2.98	3.783 (14)	145

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